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A kinetic study on cadmium adsorption from aqueous solutions by pre-conditioned phosphogypsum

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ABSTRACT

In the present study, the effect of contact time, adsorbent dosage, and initial cadmium concentration on the adsorption of Cd(II) ions from aqueous solutions by pre-conditioned phosphogypsum was investigated. The kinetics of adsorption was studied and, pseudo-first-order, pseudo-second-order, and Elovich models were used to fit the experimental data, which was obtained from batch experiments. The kinetic adsorption data fitted well into the second-order kinetic model. Adsorption mechanism was explained with the intraparticle diffusion and Boyd kinetic models.

Keywords: Adsorption; Cadmium; Kinetics; Phosphogypsum; Reuse

1. Introduction

Phosphogypsum (PG) is a by-product of the phosphate fertilizer industry. Million tons of PG are stacked worldwide every year [1]. Only in Turkey, ~3 million tons of PG are produced each year. As stated by Türkel and Aksın [2], the utilization of this industrial byproduct is important in environmental and economical point of view. However, this by-product has no apparent commercial value. Furthermore, since it is accumulated at industrial sites, it poses a disposal problem.

Nowadays, new ways are still investigated for the beneficial reuse of the accumulated PG. Our previous study showed that PG can be used as an adsorbent for lead removal from aqueous solution [3]. PG was also

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used as an adsorbent for heavy metal removal from aqueous solution after conditioning with lime, as in other studies. The lime-conditioned phosphogypsum (LCP) was examined as an adsorbent in the removal of Zn(II) and Cd(II) ions from aqueous solutions [4,5], respectively. It was reported that LCP can also be used successfully for adsorbing Cd(II) ions as well as Zn(II) ions from aqueous solution. However, the study on the kinetics of LCP for Cd(II) removal has not been reported. As known, it is important to be able to predict the rate at which contamination is removed from aqueous solutions for the design of an adsorption treatment plant [6]. In other words, in order to design industrial adsorption columns, the prediction of batch adsorption kinetics is necessary [7].

The present paper primarily focused on the kinetics of Cd(II) adsorption by LCP. The adsorption

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kinetics were analyzed using pseudo-first-order, pseudo-second-order, Elovich, intraparticle diffusion. and Boyd models. The adsorption feature of the LCP was also investigated as a function of adsorbent dosage and initial cadmium concentration as well as contact time.

2. Materials and methods

2.1. Adsorbent

PG was obtained from TÜGSAŞ Fertiliser Plant in Samsun, Turkey. It was sieved, and then the PG fraction retained on the 65 mesh was used as the adsorbent in our study. Physical and chemical characteristics of the PG have already been reported in detail in our previous papers [3,5]. PG was subjected to precondition with milk of lime, the preparation of which is described elsewhere [8], before using in the experimental studies.

2.2. Chemicals

All products used in the present study were purchased among the products of high purity (analytical grade). Simulated cadmium bearing synthetic samples were prepared by dissolving known quantity of cadmium nitrate in deionized water and used as a stock solution. Deionized water was produced by ELGA Maxima Ultra Pure Water Systems. The working solutions were prepared by a suitable dilution of standard cadmium nitrate solution (concentration 1,000 mg/L) with deionized water. The solutions in the concentration range of 5.0–100.0 mg/L were used in the adsorption experiments.

2.3. Adsorption studies

Batch adsorption experiments were conducted in a series of Erlenmeyer flasks covered with Parafilm® to prevent contamination. One hundred milliliter of an aqueous solution of known concentration and pH were put in contact with LCP, and agitated intermittently by mechanical shaking at 200 rpm and room temperature (Clifton NE5 28D Model shaking water bath). After contact time, the aqueous phase was separated from LCP by centrifugation and the concentrations of Cd(II) ions before and after adsorption were determined using an atomic absorption spectrophotometer (ATI UNICAM 929 Model Flame AAS). The pH of the solutions was adjusted to the optimum pH value, which was found as 9.5 in our previous study [5], using HNO₃ and NaOH solutions.

All the experiments were performed in duplicate, and average values are reported. The maximum error determined with 95% confidence level was 5%.

The amount of adsorbed per unit mass of the adsorbent, q (mg/g) was calculated by the following equation:

$$q (\mathrm{mg/g}) = (C_0 - C) \times \frac{V}{M}$$
⁽¹⁾

where C_0 and C (mg/L) are the initial and equilibrium concentrations of Cd(II) in solution, respectively. V (L) is the total volume of the Cd(II) solution, and M (g) is the mass of LCP used.

3. Results and discussion

3.1. Effect of contact time

The effect of contact time on the adsorption of Cd (II) by LCP is shown in Fig. 1. As can be seen from Fig. 1, the rapid Cd(II) uptake occurred within the first 10 min and thereafter the adsorption proceeds at a slower rate until it finally attains equilibrium. Time required for attaining equilibrium was about 30–40 min.

An initial rapid adsorption of Cd(II) for the first 10 min is probably due to an increased number of vacant adsorption sites available at the beginning of the adsorption process [9–11]. Also, it can be said that an increased number of vacant adsorption sites available at initial stage of the adsorption process results in an increased concentration gradient between the solute in solution and the solute on the adsorbent surface. Depending on this increased concentration gradient, the adsorption rate tends to increase at initial stage of the adsorption process [9].



Fig. 1. Effect of initial contact time (initial concentration of Cd(II): 50 mg/L; adsorbent dosage: 10 g/L: pH 9.5).

3.2. Adsorption kinetics

The kinetics of metal ion uptake are important for determining the time necessary to attain equilibrium and the adsorption mechanism type; i.e. chemisorption or physical adsorption [12]. In this work, the adsorption kinetics data was first analyzed using pseudofirst-order [13] and pseudo-second-order [14] kinetic models as shown below:

$$\log (q_{\rm e} - q_{\rm t}) = \log (q_{\rm e}) - \frac{k_1}{2.303}t$$
(2)

$$\frac{t}{q_{\rm t}} = \frac{1}{k_2 q_{\rm e}^2} + \frac{1}{q_{\rm e}} t$$
(3)

where k_1 (1/min) and k_2 (mg/g min) are the pseudo-first-order and second-order rate constants of adsorption, q_e (mg/g) is the equilibrium adsorption capacity, and q_t (mg/g) is the adsorption at any time *t* (h).

The constant k_2 is used to calculate the initial sorption rate $h \pmod{g \min}$, at $t \rightarrow 0$ as follows:

$$h = k_2 q_e^2 \tag{4}$$

The pseudo-first-order rate constant k_1 was obtained from the slope of plot between log (q_e-q_t) and time, t (Fig. 2). The pseudo-second-order rate constant, k_2 , and the equilibrium adsorption capacity, q_e , were calculated from the intercept and slope of the plots of t/q_t vs. t (Fig. 3).

The pseudo-first-order and pseudo-second-order reaction rate constants are presented in Table 1. Since the correlation coefficient value, r was found as 0.9982, it can be said that pseudo-second-order expression better predicts the sorption kinetics than the pseudo-first-order model. Higher correlation coefficient for the



Fig. 2. Pseudo-first-order reaction kinetics for the adsorption of Cd(II) onto LCP (initial concentration of Cd(II): 50 mg/L; adsorbent dosage: 10 g/L; pH 9.5).



Fig. 3. Pseudo-second-order reaction kinetics for the adsorption of Cd(II) onto LCP (initial concentration of Cd(II): 50 mg/L; adsorbent dosage: 10 g/L; pH 9.5).

pseudo-second-order model suggests that chemical reaction is probable dominant mechanism in process of Cd(II) adsorption on LCP [15].

It is well-known that the pseudo-second-order model assumes that the rate-limiting step may be chemical sorption or chemisorption involving valency forces through sharing or exchange of electrons between adsorbate and adsorbent [14,16–18]. Therefore, it can be said that the rate-limiting step may be the chemical sorption for Cd(II) adsorption by LCP. The adsorption of Cd(II) on raw and purified clay [19], dried activated sludge [20], modified Baker's yeast [21], and palm oil boiler mill fly ash [22] also follows pseudo-second-order kinetic model.

The Elovich model is presented by the following equation [23]:

$$q_{t} = \frac{1}{\beta} \ln \alpha \beta + \frac{1}{\beta} \ln t$$
(5)

where α is the initial adsorption rate (mg/g min) and β is the desorption constant (g/mg).

Plot of q_t vs. ln t was given in Fig. 4. The kinetic constants (α and β) were obtained from the slope and intercept of the plot of q_t vs. ln t are given in Table 1. As can be seen from Fig. 4, plot of q_t vs. ln t do not give a linear relationship. Elovich model was commonly suitable to describe the adsorption kinetics of ion exchange process. Therefore, it can be said that the adsorption behaviors of Cd(II) ions on LCP was not ion exchange reactions [24].

3.3. Sorption mechanisms

As also stated by Febrianto et al. [25], adsorption equilibria studies are important to determine the

Pseudo-first-order				Pseudo-second-order				Elovich model		
<i>k</i> ₁ (1/min)	q _{e, exp} (mg∕g)	q _{e, cal} (mg∕g)	r	$\frac{k_2}{(g/mg min)}$	q _{e, cal} (mg∕g)	$h = k_2 q_e$ (mg/g min)	r	α (mg/g min)	β (g/mg)	r
0.1743	4.9435	5.9525	0.9217	0.0682	5.1706	0.499	0.9982	1.1426	4.6451	0.8965

Parameters of pseudo-first-order, pseudo-second-order, and Elovich models

Note: r: Correlation coefficient.



Fig. 4. Elovich kinetics for the adsorption of Cd(II) onto LCP (initial concentration of Cd(II): 50 mg/L; adsorbent dosage: 10 g/L; pH 9.5).

efficacy of adsorption. In spite of this, it is also necessary to identify the adsorption mechanism type in a given system.

In order to estimate the mechanism of the adsorption process, intraparticle diffusion, and Boyd models were also employed to the experimental data. It is known that at an intensive stirring of the sorptive system, the intraparticle diffusion of the solute sorbed from the solution into the sorbent pores could be a rate-limiting step [26]. The intraparticle diffusion equation is defined as [27]:

$$q_{\rm t} = K_{\rm i} t^{1/2} \tag{6}$$

where $q_t \text{ (mg/g)}$ is the amount of sorption at time *t* (min) and K_i is the intraparticle diffusion rate constant (mg/g min^{1/2}).

As can be seen from Fig. 5, the plot of q against $t^{1/2}$ is multilinear containing two linear parts. Such a double nature or a non-linear relationship suggests that the adsorption process proceeds by surface sorption and intraparticle diffusion. The initial curved portion is attributed to the boundary layer effect, and the final linear portion to the intraparticle or

pore diffusion [28–30]. The calculated intraparticle diffusion rate constant values, k_{i1} and k_{i2} were 0.9905 and 0.6298 mg/g min^{1/2}, respectively. The slope of the second linear portion of the plot has been defined as the intraparticle diffusion parameter k_{i2} . It can be said that the intraparticle diffusion is the rate limiting step for Cd(II) adsorption process because of $k_{i1} > k_{i2}$ [29,30].

In order to investigate the contribution of film resistance to the kinetics of Cd(II) adsorption, the adsorption kinetic data was further analyzed by Boyd's film diffusion model.

Boyd kinetic expression is given as follow [31],

$$F = 1 - (6/\pi^2) \exp(-Bt)$$
(7)

and

$$F = q/q_0 \tag{8}$$

where q_0 is the amount of Cd(II) adsorbed at infinite time *t*, mg/g and *q* represents the amount of Cd(II) adsorbed at any time *t*, min, *F* represents the fraction of solute adsorbed at any time *t*, and *Bt* is a mathematicel function of *F*.



Fig. 5. Intraparticle diffusion plot for adsorption of Cd(II) onto LCP (initial concentration of Cd(II): 50 mg/L; adsorbent dosage: 10 g/L; pH 9.5).

Table 1

Substituting Eq. (8) in (7), Eq. (7) simplifies to:

$$1 - F = (6/\pi^2) \exp(-Bt)$$
(9)

or

$$Bt = -0.4977 - \ln (1 - F) \tag{10}$$

The Bt values at different contact times can be calculated using Eq. (10). The calculated Bt value was plotted against time t as shown in Fig. 6. This figure is used to identify whether external transport or intraparticle transport control the rate of sorption.

The linear plot of Bt against t differentiates film diffusion and particle diffusion. If a plot of Bt vs. t is linear passing through the origin, then the rate-limiting step is a particle diffusion, otherwise, it is governed by film diffusion [26,32]. It is clearly seen from Fig. 6 that the plot does not pass through the origin. Thus, it can be said that the adsorption dominates by film diffusion.

Cadmium species can be present in deionized water in the forms of Cd^{2+} , $Cd(OH)^+$, $Cd(OH)_2^0$, and $Cd(OH)_{2(s)}$, etc. [33]. Cadmium ions have little tendency to hydrolyze at pH values <8 [34], and the dominant Cd(II) species at pH < 8.0 is Cd^{2+} and $Cd(OH)^+$. Above 8, the dominant Cd(II) species is Cd (OH)_2 [35,36]. Therefore, calcium hydroxide may be formed at high pH on surface of LCP. Adsorption of Cd(II) by LCP can be represented as:

$$\equiv Ca - OH + Cd^{2+} \rightarrow \equiv Ca - OCd^{+} + H^{+} \quad (11)$$

$$\equiv Ca - OH + Cd(OH)^{+} \rightarrow \\ \equiv Ca - OCd(OH) + H^{+}$$
(12)



Fig. 6. Plot of Bt vs. t (initial concentration of Cd(II): 50 mg/L; adsorbent dosage: 10 g/L; pH 9.5).

where " \equiv " symbol indicates that particle surface sites.

However, it can be said that as pointed out in our previous paper [5], Cd(II) removal by LCP was dominantly controlled by adsorption at pH values of about 9.0, but it could be slightly enhanced by cadmium hydroxide precipitation at pH > 9.0. It is known that precipitation plays a major role in removing of Cd(II) ions in alkaline range. On the other hand, the precipitation of metal hydroxides into pores or spaces around the particles is hardly possible since the sorption process is kinetically faster than the precipitation [37,38].

3.4. Effect of adsorbent dosage

The effect of adsorbent dosage on the adsorption of Cd(II) by LCP is shown in Fig. 7. As seen from Fig. 7, the percent removal of Cd(II) increases as the adsorbent dosage increases. However, the amount of adsorbed per unit mass of the adsorbent, uptake, decreases as the adsorbent dosage for a given initial Cd(II) concentration increases. The decrease in metal uptake with increase in the adsorbent dose is due to the result of the increase in the total specific surface area of LCP [39,40]. Besides, it can be said that this



Fig. 7. Effect of LCP dosage: (a) Cd(II) removal (%) and (b) Cd(II) uptake (mg/g) (initial concentration of Cd(II): 50 mg/L; contact time: 45 min; pH 9.5).



Fig. 8. Effect of Cd(II) concentration (adsorbent dosage: 10 g/L; contact time: 45 min; pH 9.5).

effect is mainly due to unsaturation of adsorption sites through the adsorption reaction. Another reason may be due to the particle interactions, such as aggregation, resulting from high sorbent concentration. Such aggregation would lead to a decrease in the total surface area of the sorbent [16,22,40,41].

3.5. Effect of initial Cd(II) concentration

Fig. 8 shows the effect of the initial Cd(II) concentration on the adsorption of Cd(II) by LCP. The amount of adsorbed per unit mass of the adsorbent, uptake, increases with the increase in initial Cd(II) concentration. It may be a result from the enhanced driving force that allows the ionic transfer between the solid-aqueous phases [22]. This trend may also be due to an increase in electrostatic interactions, involving sites of progressively lower affinity for Cd(II) ions at low initial Cd(II) concentrations [42,43]. Similar phenomena have been reported in the adsorption of Zn(II) on PG [4], Cd(II) on palm oil boiler mill fly ash [22], Pb(II), and Zn(II) on activated carbon from *Ceiba pentandra* hulls [40], Cu(II), and Cr(III) on *Aspergillus carbonarius* biomass [42], and Zn(II) on scoria [43].

4. Conclusion

From the kinetic studies, it is observed that adsorption of Cd(II) is very rapid in the initial stage and decreases while approaching equilibrium. The kinetics followed a pseudo-second-order process, suggesting a chemisorption mechanism. It was concluded that intraparticle diffusion is not a fully operative mechanism for this system. Cd(II) removal from aqueous solution by LCP is influenced by the adsorbent dosage and initial Cd(II) concentration. A higher dosage of adsorbent led to an increase of the percentage removal of Cd(II), but decrease in the Cd(II) uptake. The Cd(II) uptake increased as the initial Cd(II) concentration increased.

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