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ORIGINAL PAPER

Kinetics and thermodynamics of zinc removal from a metal-plating wastewater by adsorption onto an Iranian sepiolite

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Abstract The study of the kinetics and thermodynamics of the adsorption process can provide valuable information for the proper design and control of adsorption systems. The influence of temperature on the kinetics of Zn^{2+} sorption on an Iranian sepiolite sample was investigated through a series of batch experiments. Consistent with an exothermic reaction, an increase in the temperature resulted in decreasing Zn^{2+} adsorption rate. The sorption kinetics was tested for pseudo-first-order, pseudo-second-order, and intraparticle diffusion models, and the rate constants were calculated. Results showed that the adsorption kinetics followed the pseudo-second-order kinetic model. The intraparticle diffusion model described that sorption of Zn onto the sepiolite particles is a two-step process, and intraparticle diffusion is not the only rate-limiting step. The results obtained from the study of thermodynamic data indicated that sorption of Zn onto sepiolite is non-spontaneous and less favored at higher temperatures.

Keywords Sepiolite · Adsorption kinetics · Exothermic · Temperature · Enthalpy

Introduction

Activated carbon has undoubtedly been the most popular and widely used adsorbent in wastewater treatment applications throughout the world. In spite of its extensive use, it is an expensive material (Babel and Kurniawan 2003). Activated carbon also requires complexing agents to improve its removal performance for inorganic materials. Therefore, this situation makes it no longer attractive to be widely used in small-scale industries because of cost inefficiency. Due to the above-mentioned problems, research interest into the production of alternative adsorbents to replace the costly activated carbon has intensified in recent years (Fu and Wang 2011). Attentions have been focused on the adsorbents which have metal-binding capacities and are able to remove excessive amounts of heavy metals from contaminated water at low cost (Babel and Kurniawan 2003; Fu and Wang 2011; Gupta and Saleh 2013). Agricultural wastes (e.g., orange peel powder), fishery wastes (e.g., chitosan), industrial byproducts and wastes (e.g., waste rubber tire), natural substances (e.g., zeolite, bentonite, etc.), and polymeric materials (e.g., resins) have been studied as adsorbents for the removal of heavy metals from wastewater samples (Babel and Kurniawan 2004; Gupta et al. 2010, 2012; Gupta and Nayak 2012).

Among all the different adsorptive materials, clays have been used extensively to capture heavy metals from aqueous solutions. This ability is mainly due to their unique properties such as high surface area associated with small particle size, swelling properties, and presence of exchangeable surface cations which facilitate ion-exchange mechanisms, low cost, and their abundance in most soil and sediment environments (Bradl 2006; Liang et al. 2013).

Sepiolite is a fibrous clay mineral which is widely distributed in arid and semiarid regions of the world (Singer 1989). Large deposits of sepiolite have been recently found in several locations of Iran, and mining of them has already been started (Hojati and Khademi 2013). The mineral is

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formed as a result of stowage of two inversed silica tetrahedral sheets and a magnesium octahedral layer between them making alternate hollow channels allow penetration of solutes into the structure (Shirvani et al. 2006). Due to its peculiar structure, sepiolite has considerable sorption ability and can adsorb up to 200–250 times water of its own weight (Ugurlu 2009). Although there are several reports about the adsorption characteristics of sepiolites from different regions of the world (Shirvani et al. 2006; Kocaoba 2009; Lazarevic et al. 2012), little information is available about the Iranian counterparts. Besides, most of the previous studies were carried out using synthetic solutions rather than a real wastewater (Babel and Kurniawan 2004; Shirvani et al. 2006; Kocaoba 2009; Lazarevic et al. 2012; Liang et al. 2013).

Zinc is a heavy metal that is present in effluents from various industries such as galvanization and metal-plating facilities, manufacture of batteries, and other metallurgical industries (Fu and Wang 2011). Although zinc is essential for the physiological functions of living tissues and regulates many biochemical processes in body, however, too much zinc can cause eminent health problems, such as stomach cramps, skin irritations, tooth-amalgam, vomiting, nausea, and anemia (Lindh et al. 1997; Oyaro et al. 2007).

Sorption of heavy metals (e.g., Zn^{2+}) onto the natural particles such as sepiolite and palygorskite determines the mobility of these metals in the environment (Shirvani et al. 2006). It has been suggested that during the sorption of pollutants from wastewater samples, different parameters such as contact time, pH and particle size of adsorbent play important roles (Echeverria et al. 2005; Sharma 2008). However, for the process design and control of adsorption systems, it is important to understand the underlying mechanism that results in the dynamic behavior of the system. As a result, the study of the kinetics and thermodynamics of the adsorption process can provide such valuable information (Adebowale et al. 2008; Boparai et al. 2011). The objectives of this study are twofold. The first is to investigate the adsorption kinetics of zinc removal by an Iranian sepiolite at different temperatures. The second is to develop a better understanding of the mechanisms of sorption reactions and to predict the rate at which the target contaminant is removed from the wastewater samples.

Materials and methods

Wastewater sample

The wastewater sample used in this study was taken from a zinc metal-plating facility in Tehran, northern Iran. The sample was analyzed for pH, electrical conductivity (EC) and total concentration of dissolved solids (TDS), turbidity, and the total concentration of Zn, Fe, Mg, Pb, and Cd ions (Table 1). The pH, EC and TDS, and turbidity of the wastewater sample were measured by using pH meter (Metrohm 780), EC meter (TPS Aqua-CP/A model), and turbidity meter (Lutron TU-2016), respectively. The concentration of heavy metals was determined using a Savant GBC Atomic Absorption Spectrophotometer (AAS) with the detection limits of 8, 50, 3, 60, 8, and 9 ppb for Zn, Fe, Mg, Pb, and Cd, respectively.

Materials and reagents

The sepiolite sample used in this study was collected from a mine in Fariman region, northeastern Iran. The chemical composition and physicochemical characteristics of the mineral used in this study are given in Table 2.

The mineralogical composition of the samples (Fig. 1) was determined by a Philips PW 1840 X-ray diffractometer, and the semi-quantitative analysis was based on the XRD pattern of disoriented powder sample using the method described by Schultz (1965). The total elemental analysis of the sepiolite sample was determined using X-ray Fluorescence Spectrometer, Bruker S4 Pioneer. The specific surface area and cation exchange capacity of the samples were measured by N_2-BET sorption analysis (using a Belsorb Mini-II machine) and NH_4-acetate method, respectively.

Kinetic experiments

All sorption studies were performed using batch technique because of simplicity and reliability (Potgieter et al. 2006). The experiments were conducted at pH 5, sorbent concentration of 20 g L^{-1}, and at the temperature of 20 ± 1 °C.

Batch adsorption experiments were carried out by mixing a constant mass of adsorbent (sepiolite of particle

Table 1 Some physicochemical characteristics of the wastewater studied

Property	Value
Color	Yellow
Electrical conductivity (dS m^{-1})	5.05
Total dissolved solids (mg L^{-1})	3230
pH	6.06
Zn (mg L^{-1})	285.53
Fe (mg L^{-1})	5.20
Mg (mg L^{-1})	3.19
Pb (mg L^{-1})	7.79
Turbidity (NTU)	37.5

Table 2 Physicochemical properties and chemical and mineralogical composition of sepiolite sample (20–53 µm) employed in the study

Property	Value
Specific surface area (m^2 g^{-1})	179.9
Specific total volume pore (cm^3 g^{-1})	0.378
Cation exchange capacity (cmol (+) kg^{-1})	11.8
Chemical composition	
SiO_2 (%)	55.32
Al_2O_3 (%)	0.30
MgO (%)	15.73
Na_2O (%)	0.02
K_2O (%)	0.01
TiO_2 (%)	0.02
MnO (%)	0.00
CaO (%)	2.74
P_2O_5 (%)	0.00
Fe_2O_3 (%)	0.59
SO_3 (%)	0.00
LOI (%)	25.11
Total (%)	99.84
Mineralogical composition	
Sepiolite (%)	70
Quartz (%)	15
Dolomite (%)	15

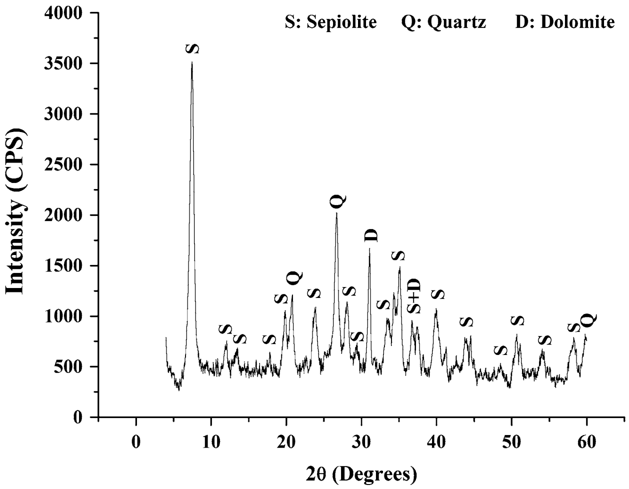

Fig. 1 XRD pattern analysis of sepiolite samples

size 20–50 µm; 1 g) and 50 mL of known concentration of wastewater in different polypropylene centrifuge tubes using a Jintan SHZ-82 thermostatic shaker bath at agitating speed of 175 rpm. The investigating temperatures were 20, 30, and 40 °C. Blank treatments were also run without the adsorbent. All measurements were carried out with three replications.

The amount of Zn adsorbed onto the sepiolite (Q_e) was calculated from the difference between initial and the equilibrium concentration in solution as below:

$$Q_e = \frac{[(C_0 - C_e)] \cdot V}{M} \quad (1)$$

where C_0 and C_e (mg L^{-1}) are the initial and equilibrium concentrations of Zn, respectively, V (mL) is the volume of filtrates, and M is the mass of sorbent (mg).

Satisfactory conformity between experimental data and the model predicted values was assessed using correlation coefficient (r^2) and non-linear chi-square (χ^2). The χ values were calculated from Eq. (2):

$$\chi^2 = \sum \frac{(Q_e - Q_e')^2}{Q_e'} \quad (2)$$

where Q_e and Q_e' are observed and model-estimated amounts of Zn which sorbed at equilibrium, respectively, and n is the number of measurements. If data from the model are similar to those of the experimental data, χ^2 would be small, and if they differ, it would be large (Boparai et al. 2011).

Results and discussion

Kinetic studies

Contact time and temperature are important factors governing the sorption process (Ugurlu 2009; Boparai et al. 2011; Ugurlu and Karaoglu 2011). Figure 2 shows the effect of contact time and temperature on the adsorption capacity of sepiolite for Zn^{2+} ions.

As can be seen, by increasing temperature from 20 to 40 °C, the adsorption capacity of sepiolite for Zn^{2+} ions decreased from 13.1 to 11.8 mg g^{-1}. The decrease in the removal capacity of Zn^{2+} ion with the rise in temperature was probably due to an increase in desorption of Zn^{2+} ion from the minerals interface to the solution and confirms that at high temperatures; adsorption of Zn^{2+} ions onto sepiolite could be considered as an exothermic phenomenon (Saltali et al. 2007; Eloussaief and Benzina 2010). This will be further discussed in relation to the thermodynamic parameters in "Thermodynamic of sorption" section.

From Fig. 2, it can also be concluded that the removals of Zn^{2+} ions by adsorption on sepiolite were initially rapid but then slowed down and remained constant with increase in contact time, and the time required to reach equilibrium was 720 min at all temperatures studied.

In order to evaluate the kinetics of Zn^{2+} adsorption on sepiolite, two different kinetic models, pseudo-first-order and pseudo-second-order, were fitted to the experimental

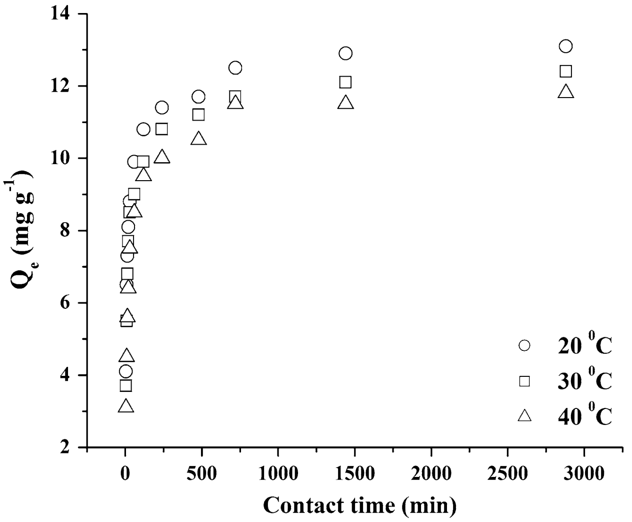

Fig. 2 Effects of contact time and suspension temperature on the adsorption capacity of sepiolite particles for Zn^{2+} ions

data (Fig. 3a, b). The linear forms of pseudo-first-order and pseudo-second-order kinetic models are given in Eqs. (3) and (4):

$$\text{Log}\,(Q_e - Q_t) = \text{Log}\,Q_e - \frac{K_1 \cdot t}{2.303} \qquad (3)$$

$$\frac{t}{Q_t} = \frac{1}{K_2 \cdot Q_e^2} + \frac{t}{Q_e} \qquad (4)$$

where Q_t (mg g^{-1}) is the adsorption capacity at time t, K_1 (min^{-1}) is the rate constant of the pseudo-first-order model (Lagergren 1898), and K_2 (g mg^{-1} min^{-1}) is the rate constant of the pseudo-second-order adsorption model (Ho and McKay 1999).

Compared with the pseudo-second-order kinetic model, the pseudo-first-order model predicts a much lower value of the equilibrium adsorption capacity (Q_e) than the experimental value (Table 3). When χ^2 values of the pseudo-first-order kinetic model and that of the pseudo-second-order kinetic model are compared, it can be concluded that the adsorption of Zn^{2+} ions onto the sepiolite particles followed the pseudo-second-order kinetic model. This suggests that the sorption capacity of Zn^{2+} ions is proportional to the number of active sites occupied on sepiolite, and the overall rate of Zn^{2+} sorption processes appears to be controlled by sharing of electrons between the adsorbent and the adsorbate, or covalent forces, through the exchange of electrons between the particles involved (Zou et al. 2011). Similar results were also reported by Sen and Gomez (2011) and Bektas et al. (2004) in their studies on sorption of zinc and lead ions on bentonite and sepiolite particles, respectively.

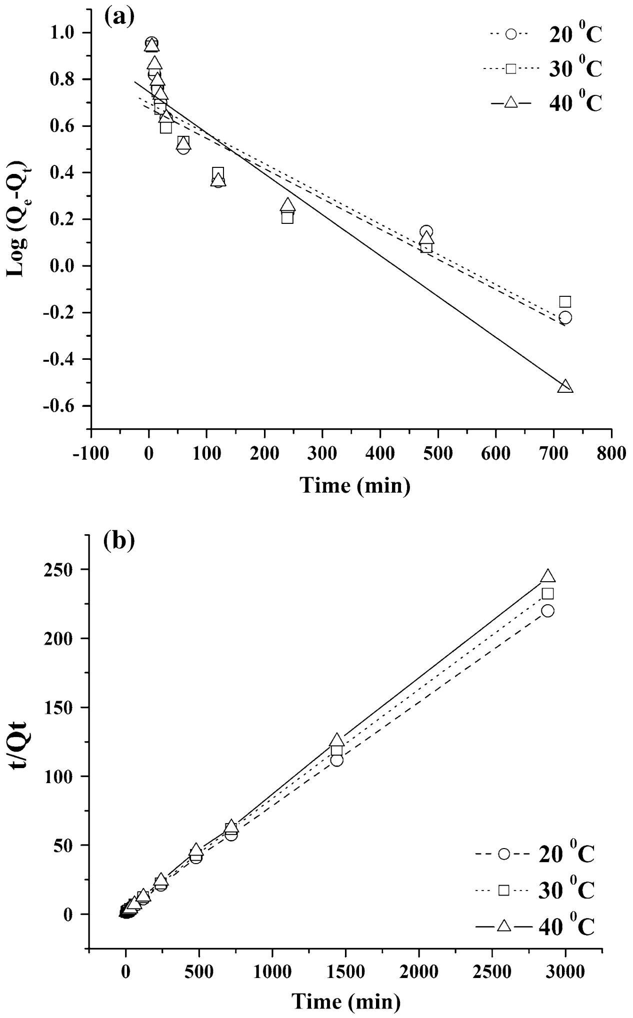

Fig. 3 **a** Pseudo-first-order, and **b** pseudo-second-order plots for the removal of Zn^{2+} ions from the wastewater using sepiolite at different temperatures

Mechanism of sorption

The sorption of metal ions from an aqueous phase onto a solid phase is a multi-step process, involving transport of metal ions from the aqueous phase to the surface of the solid particles (bulk diffusion), diffusion of metal ions through the boundary layer to the surface of the sorbent (film diffusion), transport of the metal ions from the surface to interior pores of the particle (intraparticle diffusion or pore diffusion), and sorption of metal ions at active sites on the surface of the material by a chemical reaction, such as ion exchange, complexation, or chelation (Lazarevic et al. 2012). Owing to the fact that the pseudo-first-order and second-order kinetic models could not identify the diffusion mechanism, the kinetic data were further analyzed by the intraparticle diffusion model, which can be described as (Weber and Morris 1963):

Table 3 Percentage removal and rate constants of Zn^{2+} adsorption on sepiolite particles as estimated from the pseudo-first- and second-order kinetic models at different temperatures

Temperature (°C)	Q_e (mg g^{-1})	Removal efficiency (%)	Pseudo-first order				Pseudo-second order				
			K_1 (min^{-1})	Q'_e (mg g^{-1})	χ^2	R^2	K_2 (g mg^{-1} min^{-1})	Q'_e (mg g^{-1})	H (mg g^{-1} min^{-1})	χ^2	R^2
20	13.1	91.7	0.0023	5.36	11.17	0.857	0.0024	13.15	0.41	0.0002	0.999
30	12.4	86.9	0.0023	5.33	9.37	0.853	0.0020	12.5	0.31	0.0008	1.000
40	11.8	82.7	0.0023	5.29	8.01	0.831	0.0016	11.90	0.23	0.0008	0.999

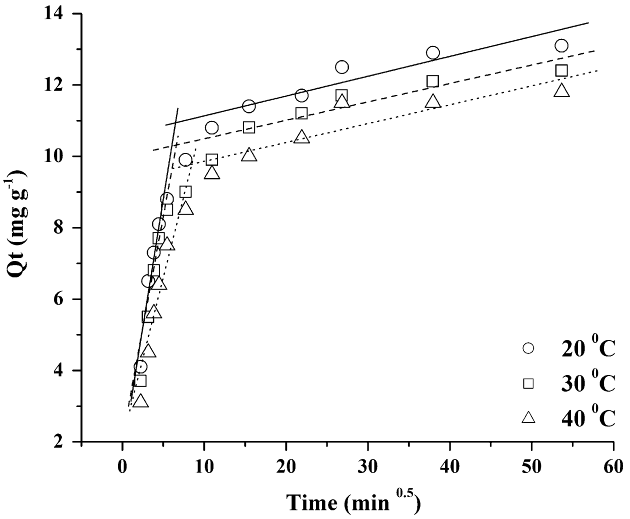

Fig. 4 Intraparticle diffusion plot for the sorption Zn onto sepiolite particles

Table 4 Intraparticle diffusion coefficients (k_i) and intercept values (C) for Zn^{2+} adsorption on sepiolite at different temperatures

Temperature	k_i (mg g^{-1} min$^{0.5}$)	C	χ^2	r_i^2
20	0.065	10.12	0.07	0.830
30	0.053	9.86	0.06	0.844
40	0.051	9.33	0.09	0.791

$$Q_t = k_i t^{0.5} + C \qquad (5)$$

where k_i is the intraparticle diffusion rate constant (mg g^{-1} min$^{-0.5}$) and C is the intercept. If the plot of Q_t versus $t^{0.5}$ gives a straight line that passes through the origin, then the adsorption process is controlled by intraparticle diffusion only. However, if the plot deviates from linearity, then the boundary layer diffusion (film diffusion) controls the sorption process. In addition, if the data show multi-linear plots, then a combination of diffusion and adsorption controls the process of sorption (Boparai et al. 2011; Liang et al. 2013). As shown in Fig. 4, given the multi-linearity of the plot for sorption of Zn^{2+} ions on sepiolite, it indicates that two steps take place. The first steeper section could be attributed to the diffusion of Zn^{2+} ions through the solution to the external surface of sepiolite (film diffusion), and the second linear section represents a gradual adsorption stage, where the intraparticle diffusion is the rate-limiting step. Similar results were also reported by Ugurlu and Karaoglu (2011) in the study of ammonium adsorption from an aqueous solution by fly ash and sepiolite.

The intraparticle diffusion rate constant k_i was calculated from the slope of the second linear section (Fig. 4; Table 4). The value of the intercept in this section provides information related to the thickness of the boundary layer

(Boparai et al. 2011; Kavita and Namasivayam 2007). Larger intercepts suggest that surface diffusion has a larger role as the rate-limiting step. The intercept values decreased with the increase in temperature. This implies that the surface diffusion became more important at lower temperatures because of the less random motion associated with the increased thermal energy (Boparai et al. 2011).

Adsorption activation energy

The activation energy of Zn adsorption on sepiolite was calculated using Arrhenius equation which is expressed as below:

$$k_2 = k \exp(-E_a/RT) \qquad (6)$$

where k is the temperature-independent factor (g mg^{-1} min^{-1}), E_a the activation energy of sorption (kJ mol^{-1}), R the universal gas constant (8.314 J mol^{-1} K^{-1}), and T is the solution temperature (K). Rate constants of Zn^{2+} adsorption were determined at 20, 30, and 40 °C using the pseudo-second-order kinetic model. Then using Arrhenius equation parameters, adsorption type and the temperature-independent rate parameters were determined. A plot of ln k_2 versus $1/T$ yields a straight line, with slope $-E_a/R$ (Fig. 5). The magnitude of the activation energy is commonly used as the basis for differentiating between physical and chemical adsorption. Physical adsorption reactions are readily reversible, equilibrium is attained rapidly, and thus energy requirements are small, ranging from 5 to 40 kJ mol^{-1}. Chemical adsorption is specific, involves stronger forces, and thus requires larger activation energies (e.g., 40–800 kJ mol^{-1}). The activation energy for Zn^{2+} adsorption onto sepiolite was -15.99 kJ mol^{-1}, suggesting that the Zn^{2+} ions were physically adsorbed onto the sepiolite surface. The negative value of Ea suggests the exothermic nature of Zn^{2+} adsorption onto the sepiolite.

Thermodynamic of sorption

Thermodynamic parameters of sorption including Gibbs free energy (ΔG^0), change in enthalpy (ΔH^0), and change in entropy (ΔS^0) were also calculated using the following equations (Sari et al. 2007):

$$\Delta G^0 = -RT \ln K_0 \qquad (7)$$

where R is the universal gas constant (8.314 J mol^{-1} K^{-1}) and T is temperature (K).

$$\text{Ln } K_0 = \Delta S^0/R - \Delta H^0/RT \qquad (8)$$

K_0 can be defined as:

$$K_0 = C_{\text{solid}}/C_{\text{liquid}} \qquad (9)$$

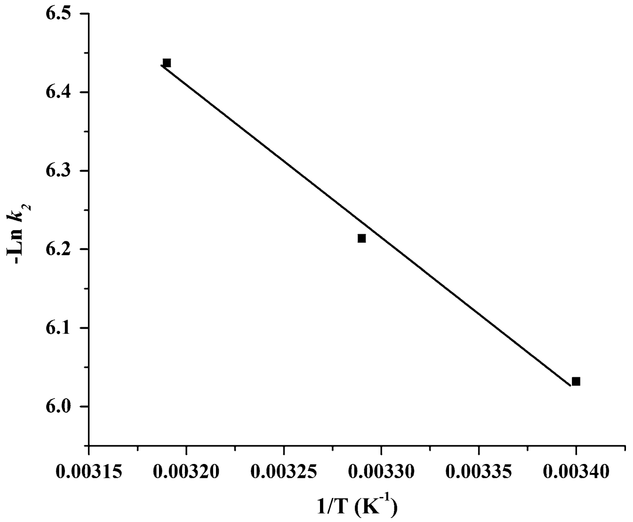

Fig. 5 Estimation of activation energy for the sorption of Zn^{2+} ions onto sepiolite particles ($Y = 1924X - 12.56$) with $R^2 = 0.992$

Table 5 Thermodynamic parameters for adsorption of Zn^{2+} ions on sepiolite particles

ΔH^0 (kJ mol^{-1})	ΔS^0 (J mol^{-1} K^{-1})	ΔG^0 (kJ mol^{-1})		
		293.15 K	303.15 K	313.15 K
−4.357	−40.422	7.49	7.89	8.30

where C_{solid} is the amount of Zn^{2+} adsorbed by sepiolite at equilibrium and C_{liquid} is the equilibrium concentration of Zn^{2+} in solution.

The values of ΔH^0, ΔS^0 (from the slope and intercept of the plot of Ln K_0 vs. $1/T$, respectively), and ΔG^0 calculated in this study are given in Table 5. As revealed in the table, with the rise in temperature from 20 to 40 °C, the value of ΔG^0 becomes more positive showing the existence of an energy barrier and that the process of Zn^{2+} sorption by sepiolite is not favorable at higher temperatures (Ugurlu 2009; El-Kamash et al. 2005). The negative values of ΔH^0 and ΔS^0 reflect the exothermic nature of sorption reaction and the decrease of randomness at the solid/liquid interface during the sorption of Zn^{2+} onto sepiolite (Bradl 2006; Alkan et al. 2007; Huang et al. 2011).

Conclusion

Kinetic studies have shown that the sorption of Zn^{2+} ions onto sepiolite particles is a relatively rapid process and can be described well with the pseudo-second-order model. The intraparticle diffusion model shows that the sorption process comprises two different stages, namely film diffusion and intraparticle diffusion. Thermodynamic studies showed that the sorption of Zn^{2+} ions onto sepiolite particles is an exothermic process and decreases with the increase in temperature. To conclude, it seems that Iranian sepiolite, with low cost and abundant availability, has a good potential to be used as a sorbent for removal of Zn^{2+} ions from the wastewater studied.

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