

# D2EHPA-impregnated alumina for adsorption of strontium ions in environmental samples

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**Abstract** The alumina impregnated by the di-2-ethyl hexyl phosphoric acid was introduced to make more adsorption of strontium as well as to determine the optimal conditions. The influence of various parameters such as pH, equilibrium time, adsorbent mass, interfering ions, and various eluant agents for the desorption of the strontium ions, initial concentration, and temperature was investigated to find out the adsorption behavior of the adsorbent under different conditions. The adsorbent was characterized by the Fourier transform infrared spectroscopy. The experimental data were fitted on the two-parameter and three-parameter adsorption isotherm models. The Freundlich and Redlich–Peterson models have suitable fitting on the experimental data ( $R^2 = 0.9307$ ). The kinetic models of adsorption were analyzed by the pseudo-first and pseudo-second-order models. The results have been indicated that the pseudo-second-order kinetic model is more appropriate than the others. Advantages of our method were simple operation, less time for preparation of adsorbent, rapid phase separation, and capability to combine with various detection techniques. The method has been utilized to extract and the recovery of strontium ions in environmental aqueous samples.

**Keywords** Alumina · Strontium · Adsorption · Isotherms · D2EHPA

## Introduction

Strontium may be harmful on the health and environment due to strontium<sup>90</sup> being beta emission. One of the important compounds is strontium chromate, which is toxic with high capability in generation lung cancer and various diseases even at low amounts. It may create anemia, hypoxia, and cancer at too high uptake (MacMillan et al. 2002). Therefore, its removal has a key role to improve environment. Various methods such as precipitation, liquid–liquid extraction, ion-exchange chromatography, and adsorption were proposed to remove strontium from aqueous solutions. The performance of adsorption technique is better than the others due to simplicity, selectivity, and high efficiency (Hafizi et al. 2011).

As shown, the adsorption of Sr(II) ions from aqueous solutions may be carried out by various organic and inorganic adsorbents such as goethite, hematite, manganese dioxide, bentonite, kaolinite, montmorillonite, silica colloid, clay minerals, pecan shells, activated charcoal, zirconia-modified vermiculite, and activated carbon, which their specifications have been fully understood (Sahai et al. 2000; Karasyova et al. 1999; Hasany and Chandhary 1981; Liang et al. 1993; Jeong 2001; Papachristodoulou et al. 2002; Lu and Mason 2001; Cole et al. 2000; Shawabkeh et al. 2002; Qadeer et al. 1992; Siviah et al. 2004; Che-grouche et al. 2009).

In this study, a new adsorbent, namely alumina ( $\text{Al}_2\text{O}_3$ ) impregnated by the di-2-ethyl hexyl phosphoric acid (D2EHPA), was introduced to remove strontium ions from aqueous solutions, and the effects of adsorption parameters

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such as pH, initial concentration of Sr (II), temperature, and interfering metals were investigated. Furthermore, the isotherm models at adsorption process such as two-parameter (Langmuir, Freundlich) and three-parameter modeling (Redlich–Peterson, Toth) were evaluated to find out the best adsorption process (Wang et al. 2008; Taffarel and Rubio 2009).

## Materials and methods

Activated alumina has high adsorption capacity because of having porous structure, and high chemical activity on the surfaces. Thus, it may be widely employed to product adsorbent and catalyst with high porosity. The aluminum oxide, D2EHPA, and  $\text{Sr}(\text{NO}_3)_2$  were used as the adsorbent, impregnated solution, and salt, respectively. Also, the hydrochloric acid, nitric acid, and ammonium nitrate were used as the washing solutions in the experiments. They are the Merck's products (Darmstadt, Germany). The methods of Fourier transform infrared spectroscopy (FTIR) and inductively coupled plasma atomic emission spectroscopy (ICP) have been employed to analyze the samples. The amounts of pH and metallic ions were measured by the Metrohm model 744 pH meter (Zofingen, Switzerland) and the 150AX ICP-OES-Varian Turbo instrument. Infrared spectra were obtained by the Vector 22-FTIR spectrometer, (Bruker, Germany) via the potassium bromide pellet method.

### Modification of adsorbent

To impregnate adsorbent, the 1-g powdered alumina was combined in the D2EHPA and ethanol solutions by the shaker at rotational speed of 180 rpm for 24 h. To test, the ratio of D2EHPA to ethanol was considered as 1:1, 2:1, and 1:2. The optimum ratio was found to be 1:1. Finally, the solution was filtered, and the adsorbent was dried in an oven at 50 °C for 72 h.

### Adsorption experiment

The adsorption experiments were carried out in polyethylene tubes with 20 ml Sr ion solution in a thermostatic shaker bath, GFL-1083 model at rotational speed of 180 rpm for 5 h, and then the solutions were separated from the solids by filtration. The concentration of residual Sr ion solutions was measured by the ICP instrument. One may calculate the absorption yields (%) as follows,

$$\text{Ads yield (\%)} = \frac{(C_i - C_e)}{C_i} \times 100 \quad (1)$$

where  $C_i$  and  $C_e$  are the initial and equilibrium concentrations of the solution, respectively.

## Results and discussion

### Characterization of impregnated resin

Figure 1 shows the FTIR spectra of the  $\text{Al}_2\text{O}_3$ , D2EHPA, and  $\text{Al}_2\text{O}_3$ -D2EHPA. The intensity of  $\text{Al}_2\text{O}_3$ -D2EHPA is higher than the others. The characteristic bands of P=O, P–O–C, or P–O–H and O–H were 1247, 1054, 1480, and 3500  $\text{cm}^{-1}$ , respectively (Khaldun et al. 2008; Vel-laichamy and Palanivelu 2011).

### Effect of adsorbent' amount

The various values of adsorbent (0.02, 0.05, 0.1, 0.2, 0.5, 0.75, 1 g) in filled batch of 20 mL Sr ion solution with the concentration of 5 mg/L at 25 °C for 5 h were used. The results have been indicated that at low values of adsorbent, the percentage of adsorption will be increased with increasing adsorbent, and then it will be invariant at high values, as shown in Fig. 2. The optimum amount of adsorbent was 0.2 g, leading to adsorption of 96 %. All subsequent experiments were carried out at this optimum value.

### Effect of initial concentration

The various initial concentrations were investigated to find out the best adsorption. The six concentrations at values of 0 through 3 mg/L in 20 mL Sr ion solution with 0.2-g adsorbent were used. The experimental results have been demonstrated that the strontium adsorption is dependent to the initial concentrations as a function of parabolic in which the maximum value of 87.5 % is at 2 mg/L, as shown in Fig. 3.

### Effect of pH

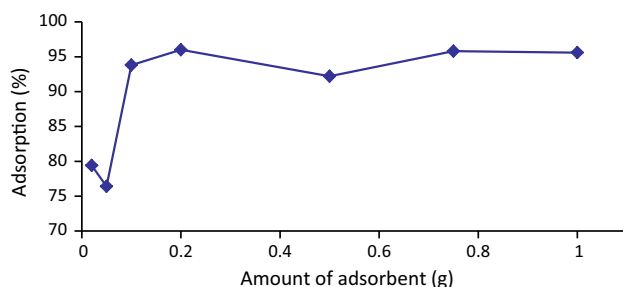
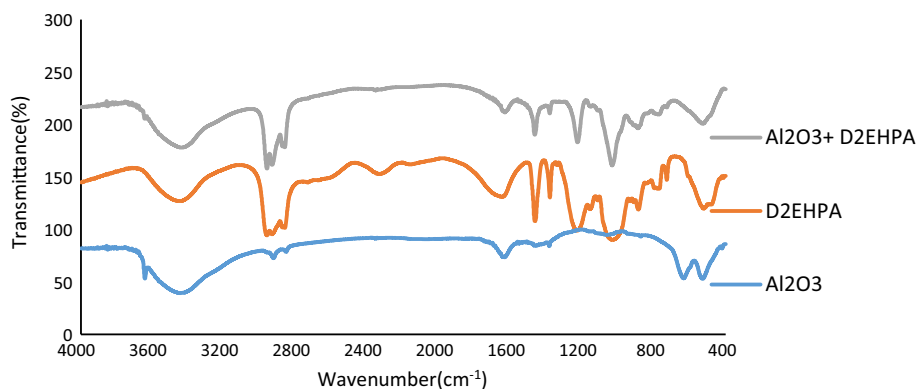
The pH values of 3 through 12 were considered at initial concentration of 5 mg/L. The acid or base was used to adjust the pH. The results show that the best pH is 5.8 with adsorption of 98 %, as shown in Fig. 4. The percentage amount of adsorption is decreased by increasing the pH value because the Sr ion has the positive charge. All of subsequent experiments were carried out at the pH 5.8.

### Desorption study

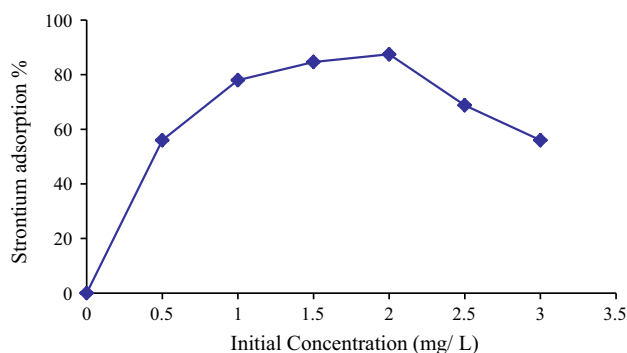
The Sr(II) desorption with 10 ml solutions of 5M-HCl,  $\text{HNO}_3$ , and  $\text{NH}_4\text{NO}_3$  at the pH 5.8 was investigated. The 5M-HCL solution is better than the others because of having the desorption efficiency value of 79.5 %. The efficiency values for the solutions of  $\text{NH}_4\text{NO}_3$  and  $\text{HNO}_3$  were 69 and 38 %, respectively.



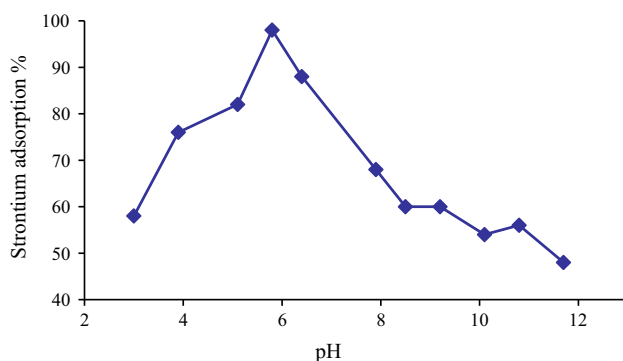
**Fig. 1** FTIR spectra of pure  $\text{Al}_2\text{O}_3$ , D2EHPA, and  $\text{Al}_2\text{O}_3$  impregnated with D2EHPA



**Fig. 2** Effect of adsorbent dose on strontium removal onto alumina impregnated by D2EHPA



**Fig. 3** Effect of initial concentration on adsorption



**Fig. 4** Effect of pH on adsorption

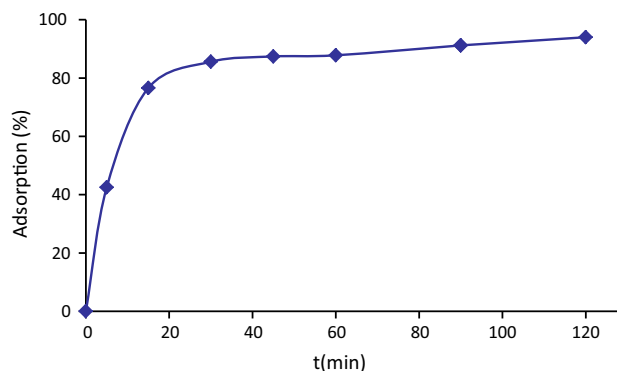
### Effect of the adsorption time

The adsorption time of strontium ions at different times (5, 15, 30, 45, 60, 90, and 120 min) was carried out. The 0.2-g  $\text{Al}_2\text{O}_3$ -D2EHPA adsorbent was shaken at 20 mL solution containing strontium 5 mg/L. After filtering the adsorbent, the concentration of strontium ions was determined by the ICP-AES using the recommended batch method. The adsorption as a function of contact time is as exponential at the times less than 30 min, and it will approach to the distinct value with increasing time. The adsorption value was 42.5 % at the time of 5 min, as shown in Fig. 5.

### Effect of temperature

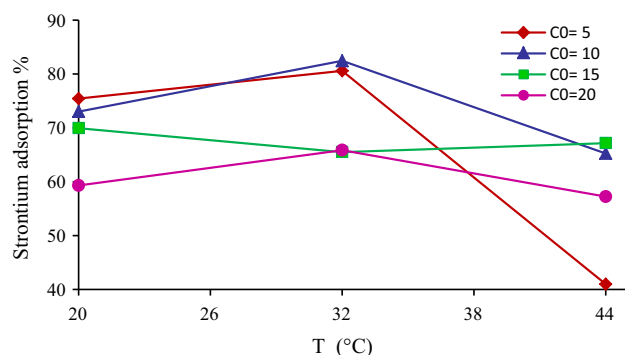
The adsorption capacity was investigated at various temperatures (20, 32, and 44 °C) and initial concentrations of 5, 10, 15, 20 mg/L, as shown in Fig. 6 and Tables 1, 2. In all experiments, volume of solution and amount of adsorbent were 20 mL and 0.2 g, respectively.

The thermodynamic parameters are considered to determine the effect of temperature on the adsorption capacity at initial concentrations of 5, 10, 15, 20 mg/L as follows (Cortés-Martínez et al. 2010),



**Fig. 5** Kinetics adsorption of Sr(II) ions on alumina impregnated by D2EHPA





**Fig. 6** Effect of temperature on adsorption percent

$$\ln K_D = \frac{\Delta S^\circ}{R} - \frac{\Delta H^\circ}{RT} \quad (2)$$

where  $K_d$ ,  $\Delta S^\circ$ ,  $\Delta H^\circ$ ,  $T$ , and  $R$  are the distribution coefficient of strontium, standard entropy, standard enthalpy, temperature in Kelvin, and the gas constant (8.314 J/mol K), respectively. The distribution coefficient ( $K_d$ ) of adsorbed strontium on the adsorbent is calculated as follows,

$$K_d = \frac{C_0 - C_e}{C_e} \times \frac{V}{m} \quad (3)$$

where  $C_0$  and  $C_e$  are the initial and equilibrium concentrations of the aqueous solution, respectively. The  $V$ (mL) and  $m$ (g) are the volume of the solution and the mass of adsorbent. The standard Gibbs free energy,  $\Delta G^\circ$ , is calculated by the following well-known equation,

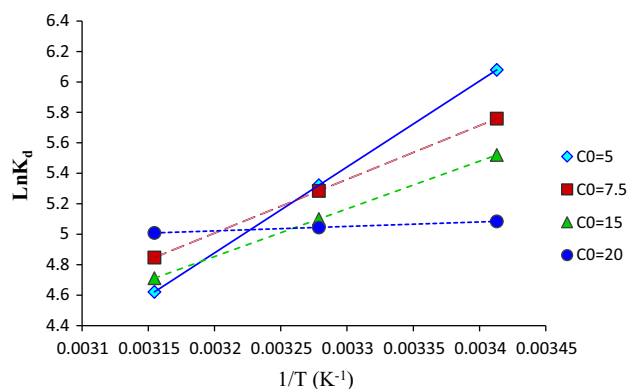
$$\Delta G^\circ = \Delta H^\circ - T\Delta S^\circ \quad (4)$$

**Table 1** The adsorption results in various temperatures

$C_0$ (mg L <sup>-1</sup> )	20 (°C)		32 (°C)		44 (°C)	
	$C_E$	Ads (%)	$C_E$	Ads (%)	$C_E$	Ads (%)
5	1.228	75.44	0.971	80.58	2.951	40.98
7.5	1.961	73.85	2.137	71.50	3.531	52.92
10	2.698	73.02	1.755	82.45	3.475	65.25
15	4.507	69.95	5.174	65.50	4.925	67.17
17.5	4.602	73.70	5.343	69.47	6.396	63.45
20	8.139	59.30	6.828	65.86	8.554	57.23

**Table 2** Thermodynamic parameters for strontium adsorption on alumina impregnated by D2EHPA

$C_0$ (mgL <sup>-1</sup> )	$\Delta H$ (kJ mol <sup>-1</sup> )	$\Delta S$ (kJ mol <sup>-1</sup> K <sup>-1</sup> )	$\Delta G$ (kJ mol <sup>-1</sup> )		
			293 K	305 K	317 K
5	-46.9408	-0.10966	-14.8104	-13.4945	-12.1786
7.5	-29.3401	-0.05226	-14.0279	-13.4008	-12.7737
15	-26.0394	-0.04297	-13.4492	-12.9336	-12.4179
20	-2.4717	0.03384	-12.3868	-12.7929	-13.1990



**Fig. 7** Effect of temperature on  $\ln K_d$

The effect of temperature on distribution coefficient ( $K_d$ ) of strontium is shown in Fig. 7 and Table 2. This coefficient is increased with increasing the temperature at various concentrations.

The  $\Delta S^\circ$ ,  $\Delta H^\circ$ , and  $\Delta G^\circ$  were measured at the different concentrations, as indicated in Table 2. The  $\Delta G^\circ$  was negative for all initial concentrations because of being the spontaneous nature of the adsorption process. In contrast, this value will be increased with increasing initial concentration at the temperatures of 293 and 305, but at 317 K this value will be decreased.

### Adsorption isotherms

Four different adsorption isotherm models containing two parameter (Langmuir–Freundlich) and three parameter (Toth–Redlich–Peterson) were considered to explain the equilibrium data. These models are commonly used to investigate the adsorption isotherm before.

### Langmuir isotherm

The most common model is the Langmuir model. This is used to quantify the amount of adsorbate adsorbed on an adsorbent as a function of partial pressure of gas or concentration of liquid at a distinct temperature. This empirical model assuming monolayer adsorption (the adsorbed layer is one molecule in thickness) may occur at a distinct number of the similar localized sites, without

lateral interaction and steric hindrance between the adsorbed molecules, even on adjacent sites (Vijayaraghavan et al. 2006). One may obtain the equilibrium concentration as follows,

$$q_e = \frac{q_m K_L C_e}{(1 + K_L C_e)} \quad (5)$$

where  $K_L$  and  $q_m$  are the Langmuir constant and the maximum amount of adsorption capacity (mg/g), respectively. The  $q_e$  and  $C_e$  are equilibrium concentrations in solid and liquid phases, respectively.

### Freundlich isotherm

The Freundlich isotherm is a curve related to the concentration of a solute on the surface of an adsorbent to the concentration of the solute in the liquid phase (Freundlich 1906). This model may be applied to multilayer adsorption, with non-uniform distribution of adsorption heat and affinities over the heterogeneous surface (Adamson and Gast 1997). It is stated as follows,

$$q_e = K_F C_e^{\frac{1}{n}} \quad (6)$$

The  $K_F$  and  $n$  refer to the Freundlich constants, and the  $q_e$  and  $C_e$  are equilibrium concentration in solid and liquid phases, respectively.

### Toth isotherm

The Toth isotherm was developed to yield an improved fit versus traditional Langmuir isotherm modeling (Tóth 2000). This model is useful to describe heterogeneous systems and approaches to the Henry region at infinite dilution. It may be rearranged to give a linear transform as follows,

$$q_e = \frac{K_{T1} C_e}{(K_{T2} + C_e)^{1/n}} \quad (7)$$

where  $q_e$ ,  $C_e$ , and  $n$  are the amount of adsorbed material (mg/g), the concentration of the adsorbate (mg/L) at equilibrium, and the Toth model exponent, respectively. The  $K_{T1}$  and  $K_{T2}$  are the Toth maximum adsorption capacity (mg/g) and the Toth equilibrium constant, respectively.

### Redlich–Peterson isotherm

The Redlich–Peterson isotherm is a hybrid of the Langmuir and Freundlich isotherms (Redlich and Peterson 1959). The numerator is from the Langmuir isotherm and has the benefit of approaching to the Henry region at infinite dilution. The denominator has the hybrid Langmuir–Freundlich form as follows,

**Table 3** Equilibrium data of impregnated alumina for Sr ions

Sr	
$Q_e$ (mg g <sup>-1</sup> )	$C_e$ (mg L <sup>-1</sup> )
0	0
0.4029	0.971
0.5363	2.137
0.8245	1.755
0.9826	5.174
1.2157	5.343
1.3172	6.828

**Table 4** The parameters of isotherm models for Sr ions

Name of isotherm	Known parameters
Langmuir	$q_m = 1.926$ $k_L = 0.2731$ $R^2 = 0.9241$
Freundlich	$k_F = 0.455$ $n = 1.845$ $R^2 = 0.9307$
Toth	$k_{T1} = 72.69$ $k_{T2} = 1.328$ $n = 0.1668$ $R^2 = 0.9297$
Redlich–Peterson	$k_{RP1} = 3253$ $k_{RP2} = 7147$ $n = 0.4581$ $R^2 = 0.9307$

$k_L$  Langmuir constant,  $q_m$  Maximum amount of adsorption capacity,  $k_F$  and  $n$  Freundlich constants,  $q_e$  and  $C_e$  Equilibrium concentration in solid and liquid phases,  $q_e$ ,  $C_e$  and  $n$  Amount of adsorbed material, the concentration of the adsorbate at equilibrium, and the Toth model exponent,  $k_{T1}$  and  $k_{T2}$  Toth maximum adsorption capacity and the Toth equilibrium constant,  $k_{RP1}$ ,  $k_{RP2}$  and  $n$  Redlich–Peterson isotherm constant, a constant, and an exponent that lies within 0 and 1,  $C_e$  and  $q_e$  Equilibrium liquid-phase concentration of the adsorbate and the equilibrium adsorbate loading onto the adsorbent

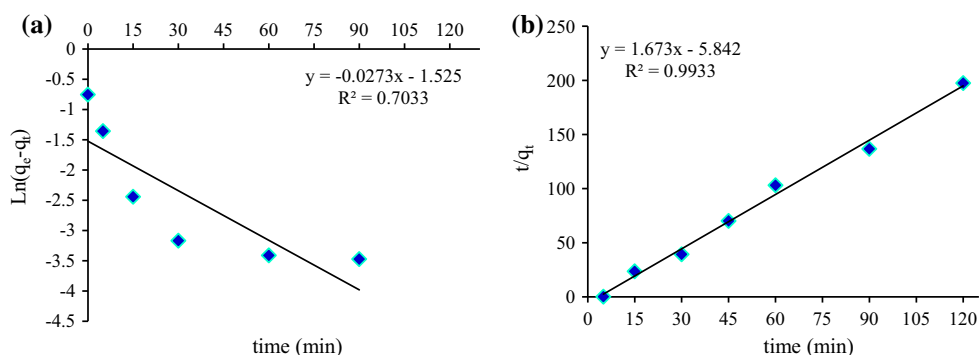
$$q_e = \frac{K_{RP1} C_e}{1 + K_{RP2} C_e^n} \quad (8)$$

where  $K_{RP1}$ ,  $K_{RP2}$ , and  $n$  are the Redlich–Peterson isotherm constant (L/g), a constant (L/mg), and an exponent that lies within 0 and 1, respectively. The  $C_e$  and  $q_e$  are the equilibrium liquid-phase concentration of the adsorbate (mg/L) and the equilibrium adsorbate loading onto the adsorbent (mg/g), respectively.

The equilibrium data of impregnated alumina are indicated in Table 3 at 32 °C, and the various parameters of isotherm models using the equilibrium experimental data are listed in Table 4. The Freundlich and Redlich–Peterson



**Fig. 8** **a** Pseudo-first-order and **b** pseudo-second-order kinetics on alumina impregnated by D2EHPA at different concentrations



models have suitable fitting on the experimental data ( $R^2 = 0.9307$ ).

### Study of adsorption kinetics

One of the most important characteristics is the solute uptake rate that represents the adsorption efficiency of the adsorbent. The adsorption process was initially fast (0–15 min) and then was more slowly with increasing time, and the equilibrium state was at the times of 15 till 120 min. To analyze the adsorption rate of Sr ion on impregnated alumina, Eqs. (10) and (11) were considered based on the experimental data as follows (Ho and McKay 1999; Alihosseini et al. 2010),

$$\text{Log}(q_e - q_t) = \text{Log}q_e - k_1 t \quad (9)$$

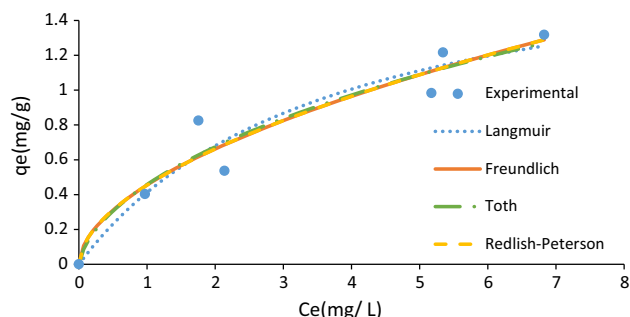
$$\frac{t}{q_t} = \frac{1}{2k_2 q_e^2} + \frac{t}{q_e} \quad (10)$$

where  $k_1$  and  $k_2$  are the Lager green adsorption rate constant ( $\text{h}^{-1}$ ) and the pseudo-second-order adsorption rate constant ( $\text{g/mg h}$ ), and the  $q_e$  and  $q_t$  are the amounts of metal ions absorbed ( $\text{mg/g}$ ) at equilibrium and at time of  $t$ , respectively. The plots of  $\log(q_e - q_t)$  and  $t/q_t$  versus  $t$  are shown in Fig. 8. The pseudo-second-order kinetic model provides a correlation for the adsorption of Sr ions on the impregnated alumina better than the pseudo-first-order model. In addition, the correlation coefficient of the pseudo-second-order kinetic model is more than that of the pseudo-first-order kinetic model (Table 4). The best fit was carried out at temperature of 32 °C, as shown in Fig. 9.

### Effect of other ions

The effect of other ions (5 mg/L) such as  $\text{Ca}^{+2}$ ,  $\text{Ni}^{+2}$ ,  $\text{Fe}^{+2}$ ,  $\text{Mg}^{+2}$ ,  $\text{Ba}^{+2}$ ,  $\text{Cd}^{+2}$ ,  $\text{Al}^{+3}$ , and  $\text{Na}^{+}$  on the uptake of Sr(II) ions (concentration 5 mg/L) was investigated here. One may calculate the extraction percentage (E %) by the following equations,

$$Q = (C_0 - C_e)V/W \quad (11)$$



**Fig. 9** Best nonlinear isotherms for the adsorption of strontium (II) ions at 32 °C

**Table 5** Effect of interfering ions on the adsorption of Sr (II) ions

Interfering ion	A ( $\text{mg L}^{-1}$ )	%L	%E	D
–	4.58	0	91.6	9.16
$\text{Ca}^{+2}$	3.0	34.49	60	6.0
$\text{Ni}^{+2}$	3.82	16.59	76.4	7.64
$\text{Fe}^{+2}$	3.69	19.43	73.8	7.38
$\text{Mg}^{+2}$	2.24	51.09	44.8	4.48
$\text{Ba}^{+2}$	3.52	23.14	70.4	7.04
$\text{Cd}^{+2}$	3.92	14.41	78.4	7.84
$\text{Al}^{+3}$	3.17	30.78	63.4	6.34
$\text{Na}^{+}$	3.03	33.84	60.6	6.06
Mixed above	1.74	62.01	34.8	3.48

A amount of adsorbed Sr (II) ( $\text{mg L}^{-1}$ ), L loss adsorption (%), E extraction percentage (%), D distribution ratio

$$E = C_e/C_0 \quad (12)$$

$$D = EV/W \quad (13)$$

$$L = C_e^{\text{No-ion}} - C_e/C_e^{\text{No-ion}} \quad (14)$$

where  $Q$  is the adsorbent capacity ( $\text{mg/g}$ ), the  $C_0$  and  $C_e$  stand for the initial and equilibrium concentrations ( $\mu\text{g/mL}$ ), and the  $W$ ,  $V$ , and  $E$  % are the mass of adsorbent  $\text{Al}_2\text{O}_3/\text{D2EHPA}$  (0.2 g), the volume of solution (0.02 L), and the extraction percentage, respectively. The interfering





**Table 6** Results of Sr(II) recovery after spiking in various water samples

Sample	Spiked (mg L <sup>-1</sup> )	STD	Found (mg L <sup>-1</sup> )	<sup>a</sup> Recovery % + RSD %
Tap	0.0	0.006	0.153	–
Water	0.2	0.015	0.356	100.8 ± 4.28
	0.4	0.010	0.560	101.2 ± 1.79
Well	0.0	0.058	3.567	–
Water	0.2	0.252	3.630	96 ± 6.92
	0.4	0.058	4.130	104 ± 1.4
River	0.0	0.100	8.700	–
Water	0.2	0.100	8.900	100 ± 1.12
	0.4	0.100	9.100	100 ± 1.10

SD standard deviation

RSD relative standard deviation

<sup>a</sup> For three determinations

ions have negligible effect on Sr (II) uptake, as listed in Table 5.

### Analysis of the real samples

To examine the ability of the impregnated adsorbent under optimal conditions, we have estimated the adsorption of the Sr (II) ions in various environmental aqueous samples. Table 6 shows the results of recovery of three different samples under the similar conditions with high recovery in which is defined as follows,

$$\text{Recovery}_{(N+1)}\% = \frac{C_{\text{Found}(N+1)}}{C_{\text{Spiked}(N+1)} + C_{\text{Found}(N)}} \times 100 \quad (15)$$

### Conclusion

In this study, the adsorption of Sr<sup>2+</sup> was investigated by the D2EHPA-impregnated alumina at batch process. The effect of pH, contact time, mass of adsorbent, initial concentration, and temperature was evaluated to determine the optimum conditions of strontium adsorption. The results have been indicated that the optimum conditions of adsorption were at pH 5.8, mass of adsorbent 0.2 g, initial concentration 2 mg/L, and temperature 32 °C. Also, the experimental data were fitted into various adsorption isotherm models. The Freundlich and Redlich–Peterson models have suitable fitting on the experimental data with  $R^2 = 0.9307$ . The kinetic models of adsorption were analyzed by pseudo-first- and pseudo-second-order models. The results have been illustrated that the pseudo-second-order kinetic model was more appropriate than the others. Advantages of our method were simple operation, less time for preparation of adsorbent, rapid phase separation, and capability to combine with

various detection techniques. We have investigated the effect of other ions such as Ca<sup>2+</sup>, Ni<sup>2+</sup>, Fe<sup>2+</sup>, Mg<sup>2+</sup>, Ba<sup>2+</sup>, Cd<sup>2+</sup>, Al<sup>3+</sup>, and Na<sup>+</sup> on the uptake of Sr(II) ions. The interfering ions have negligible effect on Sr (II) uptake. Moreover, the ability of the impregnated adsorbent under optimal conditions at the real samples was investigated, and we have estimated the adsorption of Sr(II) ions in various environmental aqueous samples.

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