Sorption potential of impregnated charcoal for removal of heavy metals from phosphoric acid

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A B S T R A C T

The viability of some heavy ions removal from phosphoric acid solution by means of impregnated charcoal with triphenylphosphine sulphide extractant has been demonstrated in this work. Factors affecting removal of Cu^{2+}, Cd^{2+}, Zn^{2+} and Pb^{2+} include initial concentration of ions, volume of the aqueous solution to weight of adsorbent ratio (V/m), concentration of the extractant loaded onto charcoal and temperature. The removal percent of these ions is increased by decreasing values of V/m ratio and increasing temperature for all ions. The sorption isotherms data fit Langmuir, Freundlich and Dubinin–Radushkviech (D–R) models. The values of the mean free energy, E_a, of sorption is in all cases in the range 9.1–25.6 kJ/mol, which are within the ranges of chemical sorption reaction. The sorption reaction was found to obey a pseudo second-order rate model.

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1. Introduction

Phosphoric acid is generally produced in Egypt by the wet process method, where the hydroxyl appetite mineral is treated mainly with sulfuric acid. Wet process phosphoric acid (WPA) contains a number of organic and inorganic impurities that affect on the grade of the acid. Some of these inorganic impurities are heavy ions such as copper, cadmium, zinc and lead which are considered as hazardous substances. The presence of these impurities is the reason for about 95% of the acid produced by the wet process is directly used as fertilizers and not in other applications as foodstuff, pharmaceutics and sugar industry [1].

Removal of heavy ions from phosphoric acid has been studied using many techniques, such as precipitation [2], liquid membranes [3], liquid–liquid extraction [4,5] and solid–liquid extraction [6].

This technique provides the best use of advantages of both liquid–liquid extraction and solid–liquid technologies. The solvent impregnated materials are characterized by their high binding capacity, selectivity and enhanced mobility of the extractant in the solid surface [7]. Numerous extractants [8–12] were used by this technique such as organophosphorus extractants like di(2-ethyl hexyl phosphoric acid (HDEHP), CYANEX-272, PC-88A; neutral extractants like tri-*n*-butyl phosphate (TBP) and also basic extractants like tri-*n*-octylamine (TOA). These extractants were impregnated onto different supports like XAD series Amberlite resins, silica gel, kiesselguhr and activated carbons.

In this contribution, the removal of Cu(II), Cd(II), Zn(II) and Pb(II) ions form phosphoric acid solutions by triphenylphosphine sulphide impregnated onto charcoal has been investigated by batch kinetics and equilibrium studies.

2. Experimental

2.1. Chemical and reagents

All chemicals and reagents used were of AR grade. Copper, cadmium, zinc and lead were prepared using analytical grade $CuSO_4$, $CdSO_4$, $ZnSO_4$ and $PbNO_3$ obtained from Winlab, England. Analytical grade phosphoric acid solution (Adwic) was used. Triphenylphosphine sulphide (TPPS) obtained from Aldrich and chloroform was purchased from Adwic. Activated charcoal supplied by LOBA Chemie. The concentrations of the heavy ions studied were determined using atomic absorption spectrometry (AAS) model Z-8100, Hitachi, Japan.

2.2. Preparation of the solid phase

TPPS in (chloroform) solution was impregnated onto charcoal by dry method technique which is widely used [13]. In this concern, 50 mL of the impregnated solutions were mixed with 10 g of

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charcoal. The resulting slurry is gently stirred for 2 h and chloroform was then evaporated at ambient temperature ($25 \pm 1\,^\circ$C). The solid materials were dried at 70 $^\circ$C before use.

2.3. Procedure

Cu, Cd, Zn and Pb solutions were prepared by dissolution of certain weights of their salts in 3.5 M phosphoric acid to obtain solutions containing 1.0 g/L for each ion. Ions concentrations were determined by AAS.

Batch sorption experiments were performed by shaking 0.05 g of the impregnated charcoal with 5.0 mL of the ion solution in a thermostated shaker bath at ($25 \pm 1\,^\circ$C). All the experiments were carried out in triplicate and the mean values were presented.

The amount of the ion uptake was calculated by the difference between the equilibrium concentration and the initial concentration. The amount of ion retained in the solid phase q_e (mg/g) was calculated using the relation:

$$q_e = (C_o - C_e) \left[\frac{V}{m} \right] \tag{1}$$

where C_o and C_e are the initial and equilibrium concentrations (mg/L) of ions solution, respectively, V is the volume of solution (L) and m is the weight (g) of the adsorbent.

The removal percent of ions from the aqueous phase is calculated from the relation:

$$R\% = \frac{C_o - C_e}{C_o} \times 100 \tag{2}$$

3. Results and discussion

3.1. Infrared spectroscopy

The impregnation process of TPPS on charcoal is mainly due to a combination of pore filling as well as surface adsorption i.e. extractants fill almost all porous system for charcoal. This suggestion is confirmed using detailed investigation of quantitative IR spectroscopic characterization of charcoal before and after impregnation step.

The spectrum of untreated charcoal is shown in Fig. 1a, and the spectrum of charcoal loaded with triphenylphosphine sulphide is shown in Fig. 1b.

Fig. 1a shows the characteristic conjugated polynuclear aromatic compounds band at 1567 cm^{-1}. This figure also shows C—H and O—H bending bands at 1111 cm^{-1}, also, bands in the range of 622–450 cm^{-1} arising from out of plan C—H bending modes of aromatic structures.

As shown in Fig. 1b and comparing with Fig. 1a, It could be observed that addition of triphenylphosphine sulphide shows a new P=S stretching band at 870 and 550 cm^{-1}. Also P=Ph bands at 1527 and 1105 cm^{-1} and a stretching band of benzene cycle C—H at 3764 cm^{-1}. All of these bands confirmed that TPPS is typically impregnated on charcoal.

3.2. Batch investigation

The removal of Cu, Cd, Zn and Pb ions were tested experimentally to investigate the adsorption capacities of the impregnated charcoal. The factors affecting removal percent of the studied ions are, V/m, ion concentrations, extractants concentration and temperature.

3.2.1. Removal kinetics

The removal kinetics of Cu, Cd, Zn and Pb ions at a concentration of 50 mg/L for each on the impregnated charcoal are studied as
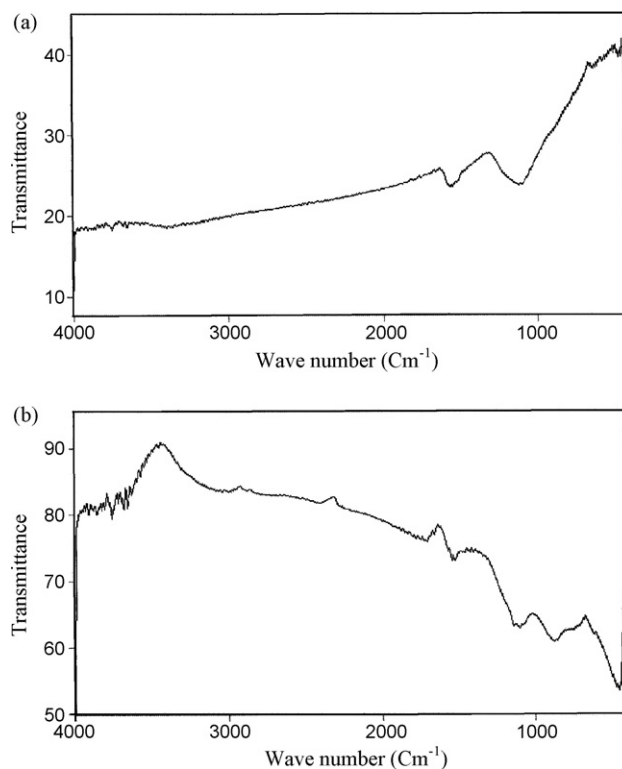

Fig. 1. (a) Infra-red spectrum of charcoal. (b) Infra-red spectrum of charcoal impregnated with TPPS.

a function of shaking time at different time intervals ranged from 15 min to 2 h. As shown in Fig. 2a and b the equilibrium of the four elements is reached after 60 min for the impregnated charcoal system at different concentration of extractant. From the figures, it was found that the removal of the ions is increased with increasing shaking time until a steady state is attained after 60 min. The kinetic data indicates that no significant change in the removal percent after the 2 h.

3.2.2. Volume to weight ratio

The effect of V/m on the removal percent of Cu, Cd, Zn and Pb ions from 3.5 M phosphoric acid is studied in the range from 25 to 200 to evaluate the optimum resin weight to achieve a high removal capability. As shown in Fig. 3a and b the removal percent increases as V/m ratio decrease from 200 to 25. The V/m ratio was kept at 100 during all the experiments whereas there is no increase in the removal percent less than this ratio.

3.2.3. Extractant concentration

The effect of TPPS concentrations on the removal percent of the studied ions is shown in Table 1. It was noticed that as the concentration of TPPS increased from 0.05 to 0.1 M a slight increase in the removal percent was observed. The order of removal is as follow

Table 1
Effect of extractant concentration on the removal percent of Zn^{2+}, Cd^{2+}, Cu^{2+} and Pb^{2+} ions onto impregnated charcoal.

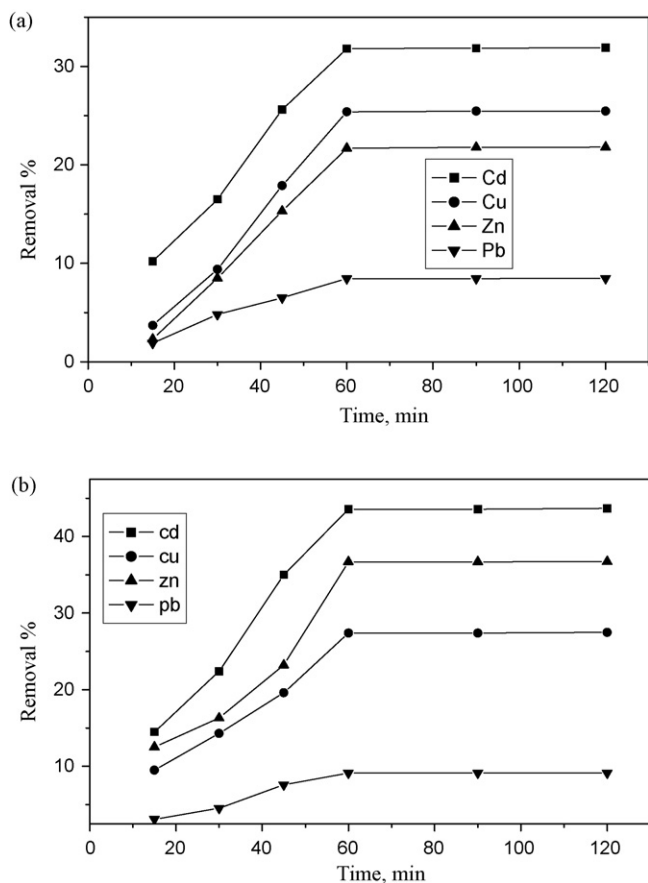| Metal ion	Removal%, 0.05 M TPPS	Removal%, 0.1 M TPPS
Cu^{2+}	25.46	27.45
Cd^{2+}	31.89	43.7
Zn^{2+}	21.8	36.74
Pb^{2+}	8.47	9.12

Fig. 2. (a) Effect of shaking time on the removal of Cd, Cu, Zn and Pb by 0.05 M TPPS impregnated onto charcoal. (b) Effect of shaking time on the removal of Cd, Cu, Zn and Pb by 0.1 M TPPS impregnated onto charcoal.

Cd > Zn > Cu > Pb. The removal of lead is too low by this impregnated system compared with the other ions.

3.2.4. Removal capacity

The removal capacity of the impregnated charcoal was determined using the batch process by equilibrating 0.05 g of charcoal with 5 mL of 3.5 M phosphoric acid solution containing various concentrations of the ions ranged from 50 to 500 ppm. As shown in Fig. 4a and b, as the concentration increase the removal percent is decreased. The maximum removal of the four ions was obtained at concentration 50 mg/L and a gradual decrease was observed with the increase of the concentration of the studied ions. The removal capacity of the impregnated charcoal was found to be 1.59, 1.27, 1.08 and 0.42 mg/g for Cd, Cu, Zn and Pb, respectively in case of 0.05 M TPPS impregnated onto charcoal. In case of 0.1 M TPPS the removal capacity was found to be 2.18, 1.37, 1.84 and 0.46 mg/g for Cd, Cu, Zn and Pb, respectively.

3.2.5. Effect of temperature

The effect of temperature on the removal of Cu, Cd, Zn and Pb ions from 3.5 M phosphoric acid by impregnated charcoal was studied in the range 25–65 °C. From the results shown in Fig. 5a and b, it is clear that increasing the temperature leads to increase in the removal percent of the four ions. This behavior indicates that the removal of the studied ions from 3.5 M phosphoric acid by impregnated charcoal with the TPPS is an endothermic process.
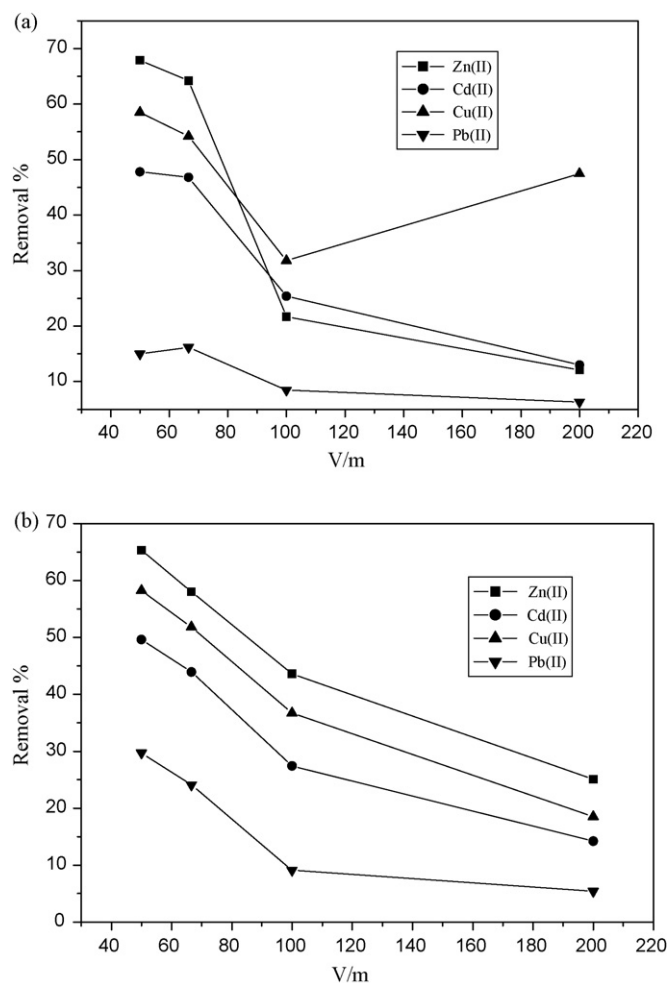

Fig. 3. (a) Effect of V/m on the removal of Zn, Cd, Cu and Pb by 0.05 M TPPS impregnated onto charcoal from 3.5 M phosphoric acid. (b) Effect of V/m on the removal of Zn, Cd, Cu and Pb by 0.1 M TPPS impregnated onto charcoal from 3.5 M phosphoric acid.

3.3. Sorption isotherms

Several common sorption isotherm models such as Freundlich, Langmuir and Dubinin–Radushkviech (D–R) isotherm models were considered to fit the obtained isotherm data.

3.3.1. Freundlich isotherm model

Freundlich equation is derived to model the multilayer sorption and for the sorption on heterogeneous surfaces. The logarithmic form of Freundlich equation is written as follow:

$$\log q_e = \log K_f + \frac{1}{n} \log C_e \tag{3}$$

where K_f is the constant indicative of the relative adsorption capacity of the adsorbent (mg/kg) and $1/n$ is the constant indication of the intensity of the adsorption process. The illustration of $\log q_e$ versus $\log C_e$ is shown in Fig. 6a and b which suggest that the sorption of the four studied ions obeys Freundlich isotherm over the entire range of sorption concentration studied. The numerical values of the constants $1/n$ and K_f are computed from the slope and intercepts by means of a linear least square fitting method and are given in Table 2. The Freundlich intensity constant $(1/n)$ for all cases is less than unity, indicating a concentration dependant sorption for all ions onto all impregnated charcoal.

Fig. 5. (a) Effect of temperature on the removal of Zn, Cd, Cu and Pb by 0.05 M TPPS impregnated onto charcoal from 3.5 M phosphoric acid. (b) Effect of temperature on the removal of Zn, Cd, Cu and Pb by 0.1 M TPPS impregnated onto charcoal from 3.5 M phosphoric acid.
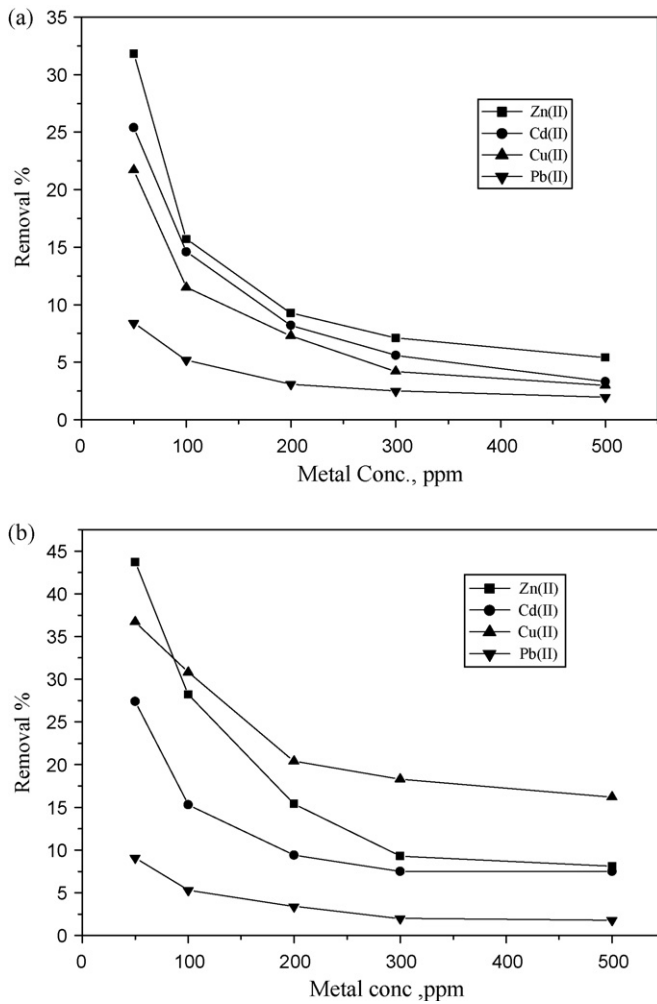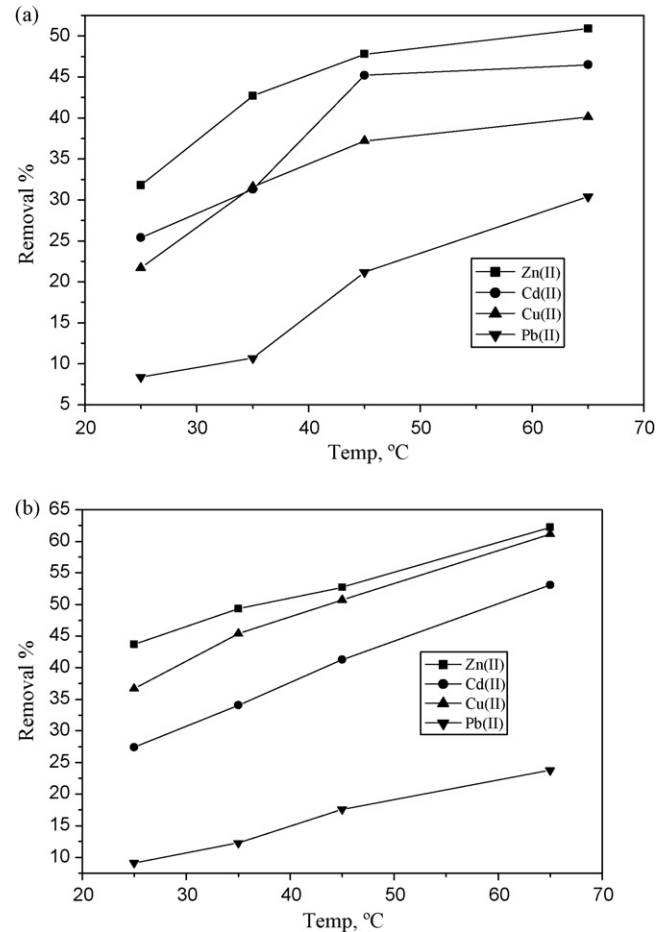
Fig. 4. (a) Effect of metal concentration on the removal of Zn, Cd, Cu and Pb by 0.05 M TPPS impregnated onto charcoal from 3.5 M phosphoric acid. (b) Effect of metal concentration on the removal of Zn, Cd, Cu and Pb by 0.1 M TPPS impregnated onto charcoal from 3.5 M phosphoric acid.

3.3.2. Langmuir isotherm model

Langmuir sorption isotherm models the monolayer coverage of the sorption surfaces and assumes that sorption occurs on a structurally homogeneous adsorbent and all the sorption sites are energetically identical. The linearized form of the Langmuir equation is given by

$$\frac{C_e}{q_e} = \left[\frac{1}{Q^o b}\right] + \left[\frac{1}{Q^o}\right] C_e \qquad (4)$$

where q_e is the amount of solute sorbed per unit weight of adsorbent (mg/kg), C_e is the equilibrium concentration of the solute in the bulk solution (mg/L), Q^o is the monolayer adsorption capacity (mg/kg) and b is the constant related to the free energy of adsorption ($b = a\,e^{-\Delta G/RT}$).

The graphic representation of (C_e/q_e) versus C_e gives straight lines for all ions sorbed onto impregnated charcoal as represented in Fig. 7a and b confirming that this expression is indeed a reasonable representation of chemisorptions isotherm. The numerical value of constants Q^o and b evaluated from the slope and intercept of each plot are given in Table 2. The value of saturation capacity Q^o corresponds to the monolayer

Table 2
Freundlich and Langmuir parameters for the sorption of Zn, Cd, Cu and Pb onto impregnated charcoal.

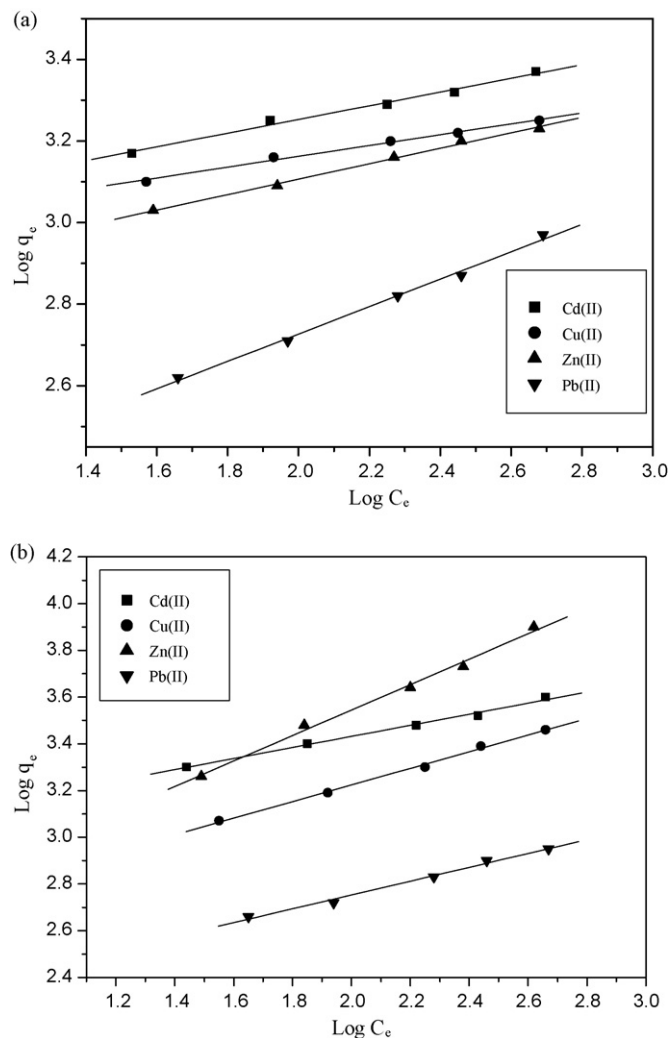| Metals	Adsorbent	Freundlich model parameters			Langmuir model parameters		
		$1/n$	K_f (mg/kg)	R^2	Q^o (mg/kg)	$b \times 10^{-3}$ (L/mg)	R^2
Zn²⁺	0.05 M TPPS	0.18	501.4	0.99	1831.5	2.5	0.99
	0.1 M TPPS	0.54	281.8	0.99	9433.9	7.07	0.99
Cd²⁺	0.05 M TPPS	0.16	812.8	0.99	2885	2.9	0.996
	0.1 M TPPS	0.23	891.2	0.99	4184	2.3	0.996
Cu²⁺	0.05 M TPPS	0.13	630	0.99	17421	8.2	0.99
	0.1 M TPPS	0.35	323	0.996	3703.7	7.29	0.99
Pb²⁺	0.05 M TPPS	0.33	112.2	0.99	1169.5	9.3	0.99
	0.1 M TPPS	0.29	144.5	0.99	1030.9	1.28	0.99

Fig. 6. (a) Frundlich isotherm plots for removal of Zn^{2+}, Cd^{2+}, Cu^{2+}, Pb^{2+} by 0.05 M TPPS impregnated onto charcoal. (b) Frundlich isotherm plots for removal of Zn^{2+}, Cd^{2+}, Cu^{2+}, Pb^{2+} by 0.1 M TPPS impregnated onto charcoal.

coverage and defines the total capacity of the adsorbent for a specific metal ion.

3.3.3. Dubinin–Radushkviech isotherm (D–R isotherm)

To study the nature of the sorption processes, the D–R isotherm was also verified in the form [14]:

$$\ln q_e = \ln q_m - \beta \varepsilon^2 \tag{5}$$

where q_m is the maximum amount of ion that can be sorbed onto unit weight impregnated charcoal, i.e. sorption capacity (mg/kg), β the constant related to the sorption energy (mol²/kJ²); and ε is the polanyi potential $= RT \ln(1 + 1/C_e)$, where R is the gas constant (kJ/mol K), and T is the solute temperature (K).

The mean free energy of sorption is the free energy change when one mole of ion is transferred to the surface of impregnated charcoal from infinity in the solution and it is calculated from:

$$E = (2\beta)^{-1/2} \tag{6}$$

The D–R plots of $\ln q_e$ versus ε^2 for the sorption of Zn, Cd, Cu and Pb ions onto impregnated charcoal are given in Fig. 8a and b. These linear plots indicate that the D–R isotherm expression is followed for each metal ion. Linear regression analysis using paired of $\ln q_e$ and ε^2 resulted in the derivation of q_m, β, E and the correlation factor
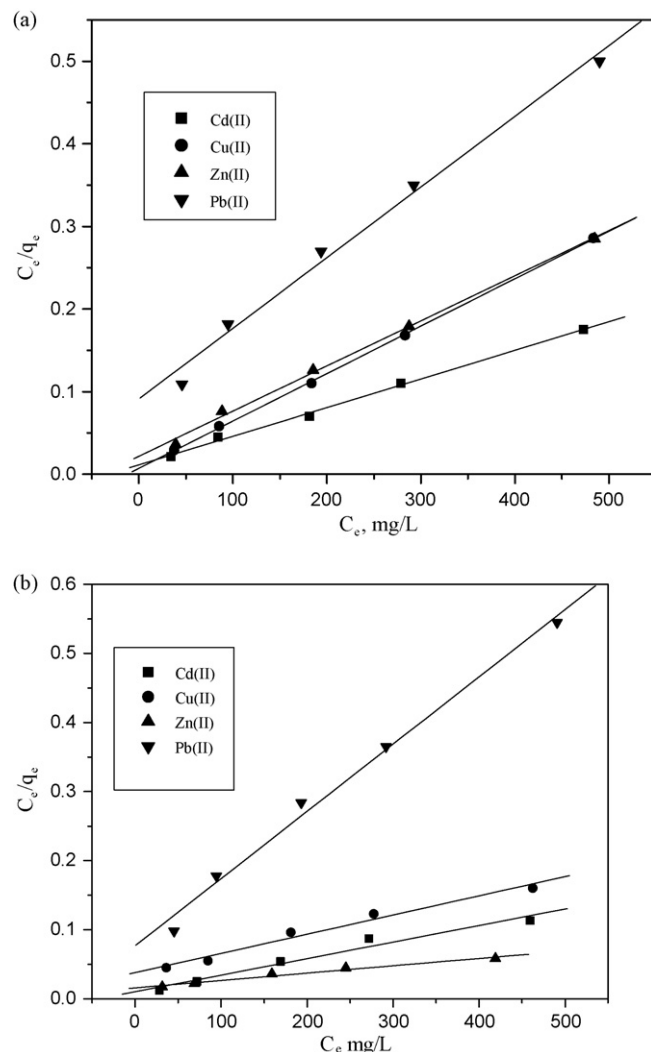

Fig. 7. (a) Langmuir isotherm plots for removal of Zn, Cd, Cu and Lead from phosphoric acid by 0.05 M TPPS impregnated onto charcoal. (b) Langmuir isotherm plots for removal of Zn, Cd, Cu and Pb from phosphoric acid by 0.1 M TPPS impregnated onto charcoal.

(R^2). These parameters are listed in Table 3. The values of the mean free energy, E_a, of sorption is in all cases in the range 9.1–25.6 kJ/mol, which are within the ranges of ion exchange reaction.

3.4. Sorption kinetic modeling

It is well recognized that the characteristic of the sorbent surface is a critical factor that affect the sorption rate parameters

Table 3
D–R isotherm parameters of the sorption of Zn, Cd, Cu and Pb ions onto impregnated charcoal.

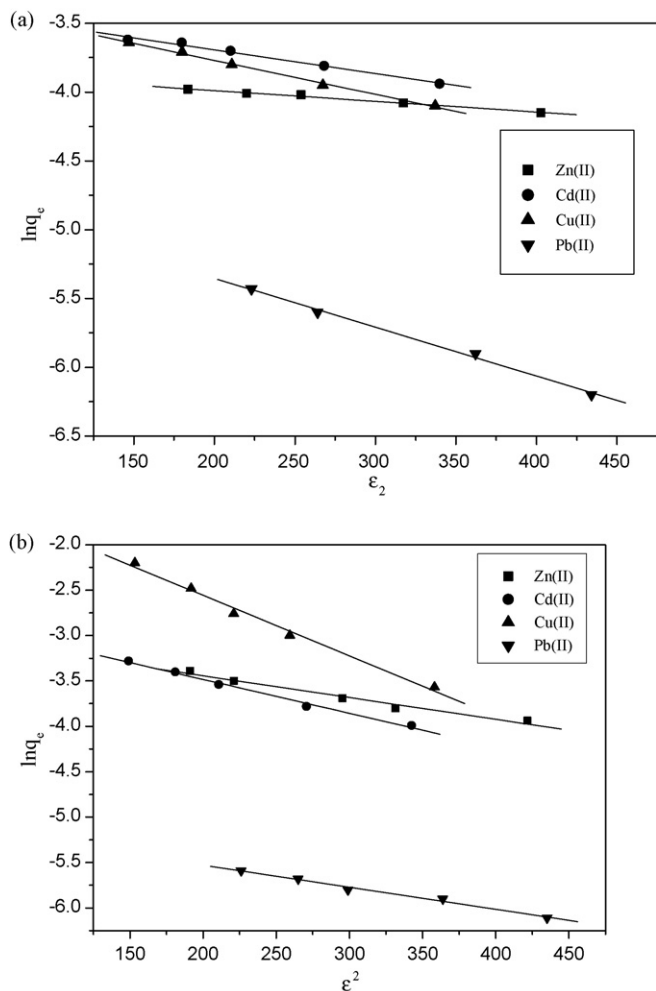| Metal ion	Adsorbent	β (mol² kJ²)	q_m (mmol/g)	R^2	E (kJ/mol)
Zn^{2+}	0.05 M TPPS	−0.00077	0.0216	0.99	25.6
	0.1 M TPPS	−0.006	0.179	0.99	9.1
Cd^{2+}	0.05 M TPPS	−0.0017	0.035	0.99	17.2
	0.1 M TPPS	−0.002	0.05	0.99	15.8
Cu^{2+}	0.05 M TPPS	−0.002	0.038	0.99	15.8
	0.1 M TPPS	−0.0029	0.056	0.99	13.1
Pb^{2+}	0.05 M TPPS	−0.0035	0.0096	0.99	12.0
	0.1 M TPPS	−0.002	0.0063	0.99	15.8

Fig. 8. (a) D–R isotherm for removal of Zn^{2+}, Cu^{2+}, Cd^{2+} and Pb^{2+} from phosphoric acid by 0.05 M TPPS impregnated charcoal. (b) D–R plots for removal of Cd^{2+}, Cu^{2+}, Zn^{2+} and Pb^{2+} from phosphoric acid by 0.1 M TPPS impregnated onto charcoal.

Fig. 9. (a) Lagergreen plots for removal of Zn^{2+}, Cd^{2+}, Cu^{2+} and Pb^{2+} from phosphoric acid by 0.05 M TPPS impregnated onto charcoal. (b) Legergreen plots for removal of Zn^{2+}, Cd^{2+}, Cu^{2+} and Pb^{2+} from phosphoric acid by 0.1 M TPPS impregnated onto charcoal.

and that diffusion resistance plays an important role in the over-all transport of the ions. To describe the changes in the sorption of metal ions with time, two simple kinetic models were tested. The rate constant of each metal ion removal from the solution by impregnated charcoal was determined using pseudo first-order and pseudo second-order rate models. Lagergreen pseudo first-order expression is written as [15]:

$$\log(q_e - q_t) = \log q_e = \frac{K_1}{2.303}t \tag{7}$$

where q_e is the concentration of the ion sorbed at equilibrium (mg/kg), q_t is the concentration of the ion sorbed at time t (mg/kg) and K_1 is the over all rate constant.

It was observed that the sorption of all ions followed the Lager-green equation. From the slopes of the plots, as shown in Fig. 9a and b the values of the first-order rate constants (K_1) and the theoret-ical sorption capacities (q_e) were evaluated. The calculated values of K_1 and q_e with the linear correlation coefficients (R^2) of each plot are listed in Table 4. Straight lines obtained from the pseudo first-order kinetic plots suggest the applicability of this model to fit the experimental data over the initial stage of the sorption process (15–45 min). But it is also required that the theoretically calculated equilibrium sorption capacities, q_e should be in accordance with the experimental sorption capacity values. As can be shown from Table 4, although the linear correlation coefficients of the plots are
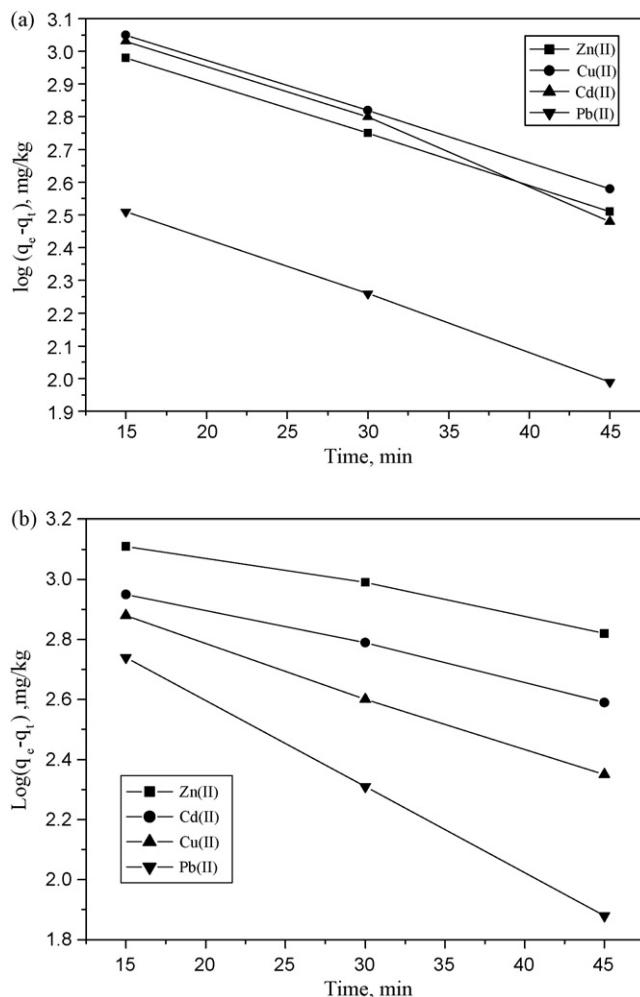
so good, the q_e (calculated) values are not in agreement with q_e (experimental) for all studied sorption process. So, it could suggest that the sorption of the four studied ions onto impregnated charcoal is not a first-order rate reaction.

The pseudo second-order rate model is expressed as [16,17]:

$$\frac{t}{q_t} = \frac{1}{K_2 q_e^2} + \frac{1}{q_e}t \tag{8}$$

where K_2 is the rate constant of pseudo second-order equation (kg/mg·min)).

The kinetic plots of t/q_t versus t for zinc, cadmium, copper and lead ions sorption onto impregnated charcoal with different extrac-tants are represented in Fig. 10a and b. The relations are linear and the values of the correlation coefficients (R^2) suggest strong relationships between the parameters and also explain that the process of sorption of ions follows the pseudo second-order kinetic model. The products $K_2 q_e^2$ is the initial sorption rate represented as $h = K_2 q_e^2$. The kinetic parameters of this model are calculated from the slope and intercept of the linear plots and are given in Table 4. The correlation coefficients (R^2) have an extremely high values and its calculated equilibrium sorption capacity (q_e) is con-sistent with the experimental data. So, it is possible to suggest that the sorption of all ions onto impregnated charcoal followed the pseudo second-order kinetic model and that the overall rate

Table 4
The calculated parameters of the pseudo first-order and pseudo second-order kinetic models for Zn^{2+}, Cd^{2+}, Cu^{2+} and Pb^{2+} ions sorbed onto impregnated charcoal.

Metal ion	Adsorbent	First-order kinetic parameters				Second-order kinetic parameters			
		K_1 (min^{-1})	$q_{e\,calc}$ mg/kg	q_{exp} (mg/kg)	R^2	K_2 (kg/(mg min))	$q_{e\,calc}$ (mg/kg)	h (mg/kg min)	R^2
Zn^{2+}	0.05 M TPPS	0.0359	1621.8	1085	0.999	9.6×10^{-5}	1000	9.6	0.99
	1 M TPPS	0.0221	1832.3	1840	0.99	1.04×10^{-5}	1832.3	34.9	0.99
Cd^{2+}	0.05 M TPPS	0.0359	1931.9	1590	0.998	1.4×10^{-5}	1555.5	33.87	0.99
	0.1 M TPPS	0.0276	1348.9	2185	0.995	1.02×10^{-5}	2182.5	48.5	0.99
Cu^{2+}	0.05 M TPPS	0.042	1995.2	1270	0.99	7.4×10^{-6}	1204.9	10.7	0.99
	1 M TPPS	0.039	1380.3	1350	0.998	1.9×10^{-5}	1305	32.3	0.99
Pb^{2+}	0.05 M TPPS	0.039	588.8	420	0.999	3.3×10^{-5}	423.4	5.9	0.99
	1 M TPPS	0.064	147.9	460	1.00	5.9×10^{-4}	433.9	11.0	0.99
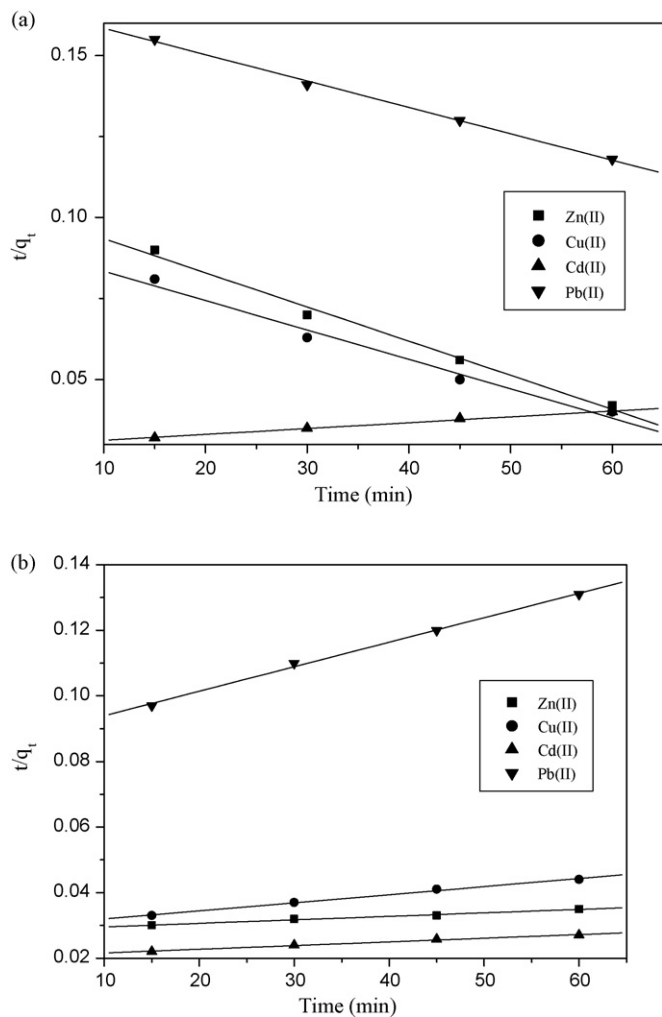
Fig. 10. (a) Pseudo second-order plots for removal of Zn^{2+}, Cu^{2+}, Cd^{2+} and Pb^{2+} from phosphoric acid by 0.05 M TPPS impregnated charcoal. (b) Pseudo second-order plots for removal of Zn^{2+}, Cu^{2+}, Cd^{2+} and Pb^{2+} from phosphoric acid by 0.1 M TPPS impregnated charcoal.

constant of each sorption process appears to be controlled by the chemical sorption process [17].

4. Conclusion

The results of Zn^{2+}, Cd^{2+}, Cu^{2+} and Pb^{2+} ions sorption reported, showed that impregnated charcoal with triphenyl phosphine sulphide is an efficient sorbent media for the removal of these ions from aqueous phosphoric acid solutions. The removal of these four ions by the sorbent material takes place via a particle diffusion mechanism, Langmuir, Freundlich and D–R isotherm models are the best choice to describe the observed equilibrium data. The sorption of all ions is an endothermic process and the results show that impregnated charcoal can be fruitfully employed for the removal of these ions in a wide range of concentrations.

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