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A comparative study on Pb^{2+}, Zn^{2+} and Cd^{2+} sorption onto zirconium phosphate supported by a cation exchanger

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Abstract

In the present study, a novel hybrid sorbent ZrP-001 was prepared by loading zirconium phosphate (ZrP) onto a strongly acidic cation exchanger D-001. Sorption behavior of Pb^{2+}, Zn^{2+}, and Cd^{2+} onto ZrP-001 was experimentally examined by comparing with the host exchanger D-001. ZrP-001 was characterized by scanning electron micrograph (SEM), X-ray diffraction (XRD), thermogravimetric analysis (TGA), pH-titration and pore size distribution analysis. Sorption of the heavy metals onto ZrP-001 was found to be pH-dependent due to the ion exchange mechanism. Compared to D-001, a smaller pore size of ZrP-001 due to the ZrP dispersion consequently resulted in a lower sorption rate. Competitive effect of Ca^{2+} on sorption of heavy metals onto ZrP-001 and D-001 was compared to elucidate sorption preference of the hybrid sorbent towards heavy metals. More favorable sorption of ZrP-001 than D-001 was observed for all the three metals and their sorption preference onto ZrP-001 followed the order $Pb^{2+} >> Zn^{2+} \approx Cd^{2+}$. Fixed-bed sorption results and its efficient regeneration property further demonstrated that ZrP-001 is a potential candidate for removing heavy metals from contaminated water.

Keywords: Heavy metals; Removal; Zirconium phosphate; Hybrid sorbent; Donnan membrane effect

1. Introduction

Water pollution by heavy metals remains an important environmental issue associated negatively with health and economy [1]. Several technologies have been currently proposed in industry in response to the problem such as chemical precipitation [2], solvent extraction [3], adsorption [4] and ion exchange [5–7]. Recently, ion exchange has been remarkably extended by the invention and application of new organic and inorganic ion exchangers [8–11].

As a family of inorganic cation exchangers prepared by combining group IV oxides with more acidic oxides of group V and VI [12], zirconium phosphate [$Zr(HPO_4)_2$, hereafter denoted ZrP] is generally taken as a representative one because it is extremely insoluble in water and offers high capacity, fast kinetics, and is superior to the organic exchangers in terms of thermal stability and resistance to radiation and abrasion [12]. In our previous study, we explored that heavy metals including Pb^{2+}, Zn^{2+}, and Cd^{2+} are loaded more selectively onto ZrP than a polystyrenesulfone exchanger D-001 in presence of Ca^{2+}, Mg^{2+}, and Na^+ at high levels [13]. However, due to its ultrafine particles ranging from 1 to 20 μm, ZrP cannot be used in fix-bed or any flow-through systems due to the excessive pressure drop and mechanical rigidity.

As a continuation of our previous study, one of the objectives of the current study is to prepare a hybrid sorbent by loading ZrP onto a polystyrenesulfone cation exchanger D-001 to overcome the above problem. D-001 was selected as a host material for sorbent preparation mainly because of the Donnan membrane effect resulting from the non-diffusible negatively charged sulfonic acid group on the exchanger surface, which would enhance permeation of the targeted metal ions [14]. Another objective is to examine sorption behavior of heavy metals onto the novel

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hybrid sorbent including effect of solution pH, sorption kinetics, competitive sorption and fixed-bed sorption test.

2. Materials and methods

2.1. Materials

All chemicals used in the present study are of analytical grade and were purchased from Shanghai Reagent Station. The stock solutions containing targeted heavy metals was prepared by dissolving their corresponding nitrates in water and filtering through a 0.22 μm membrane. D-001, a polystyrenesulfone cation exchanger in H^+-type, was kindly provided by Zhenguang Resin Co., China. It was obtained in spherical bead forms with sizes ranging from 0.6 to 1.0 mm. Prior to use, it was subjected to D.I. flushing to remove the residue impurities until neutral pH (6.8–7.0) and then vacuum-desiccated at 348 K for 24 h until reaching the constant weight.

2.2. Sorbent preparation

Procedures for preparation of ZrP-001 are given in Scheme 1.

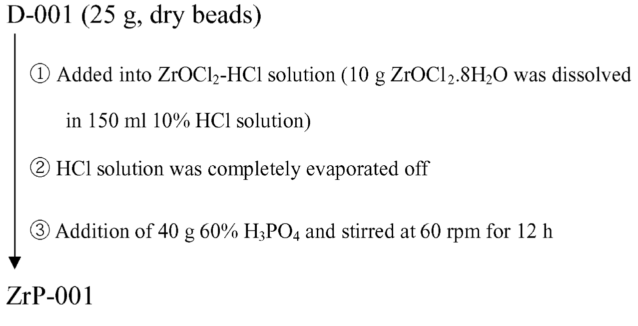
During the preparation process of ZrP-001, ZrP will be formed onto the inner surface of the D-001 beads as the following reaction

$$ZrClO_2 + 2H_3PO_4 \rightarrow Zr(HPO_4)_2\downarrow + HCl + H_2O$$

The hybrid sorbent ZrP-001 was then characterized by scanning electron micrograph (SEM), X-ray diffraction (XRD), thermogravimetric analysis (TGA) and pore size analysis.

2.3. pH titration

Portions (500 mg) of D-001 and ZrP-001 were mixed with 100 mL of 0.10 M NaCl. This mixture was kept for 2 h and titrated against 0.10 M NaOH solution. The solution pH was recorded after each addition of 1.0 mL of the titrant till the pH became constant. Based on the pH values before and after the exchange process, the milliequivalents (meq) of OH^- ion consumed were determined. Milliequivalents of OH^- ions consumed by the exchanger were then plotted against the corresponding pH values to obtain the pH–titration curves [12,15].



Scheme 1. A conceptual preparation procedure for ZrP-001.

2.4. Batch sorption experiments

Batch sorption tests were carried out in 250 ml glass bottles. To start the experiment, 50.0 mg of each sorbent was introduced to a 100 ml solution containing known concentration of heavy metals. The competing cation Ca^{2+} was introduced when necessary by dissolving $Ca(NO_3)_2$ into the solution. The flasks were then transferred to a G-25 model incubator shaker with thermostat (New Brunswick Scientific Co. Inc.) and shaken under 200 rpm for 24 h at the desired temperature to ensure the sorption equilibrium. One molar HNO_3 solution was used to adjust the solution pH throughout the experiment when necessary. One milliliter solution at various time intervals was sampled from the flasks to determine sorption kinetics. Uptakes of heavy metals onto sorbents are calculated by conducting a mass balance before and after the test.

2.5. Fixed-bed column sorption and regeneration

Column experiments were carried out with a glass column (12 mm diameter and 130 mm length) equipped with a water bath to maintain a constant temperature. Five milliliter of each sorbent was packed within the column before operation. A Lange-580 pump (Baoding, China) was used to ensure a constant flow rate. All the sorption column runs were performed under the identical hydrodynamic conditions: the superficial liquid velocity (SLV) and the empty bed contact time (EBCT) were identical and equal to 0.50 m/h and 6 min, respectively. After sorption 1 M HNO_3 or HCl solution was used as regenerant for desorption of the loaded heavy metals.

2.6. Analysis

Concentrations of all the heavy metals in solution were carried out by atomic absorption spectroscope (Z-8100, HITACHI) except when its content was less than 2 mg/l, which was determined by atom fluorescence spectrophotometer (AFS) with an online reducing unit (AF-610A, China) with $NaBH_4$ and HCl solution [16]. The loaded ZrP onto D-001 was observed with a scanning electron microscope (LEO 1530VP, Germany). It was also extracted by $HClO_4$ solution (70% in mass) [17] and determined by ICP (JA1100, USA) [18]. Speciation of ZrP in ZrP-001 beads was analyzed with an X-ray diffraction analysis instrument (XTRA, Switzerland). XPS analysis of the sorbent was performed with a spectrometer (ESCALAB-2, Great British) equipped with Mg Kα X-ray source (1253.6 eV protons). All binding energies were referenced to the C 1s peak at 288.75 eV to compensate for the surface charging effects. Thermogravimetric analysis was performed with a Pyris 1 TGA unit (Perkin-Elmer, USA) at a heating rate of 5 °C/min under a nitrogen atmosphere.

3. Results and discussion

3.1. Characterization of ZrP-D001

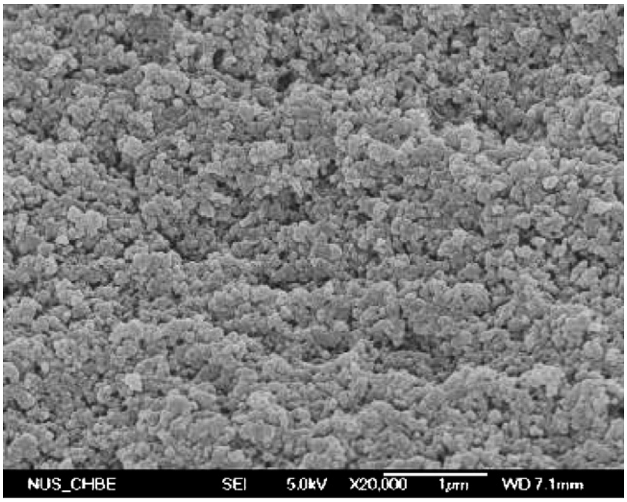
Some important properties of ZrP-001 are identified and shown in Table 1 by comparing with D-001, a strongly acidic

Table 1
Salient properties of a strong-acid cation exchanger D-001 and its ZrP-loaded derivative ZrP-001

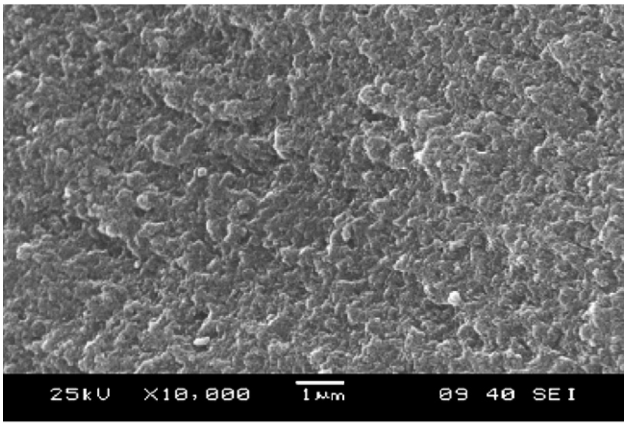
Designation	D-001	ZrP-001
Matrix structure	Polystyrene	
Surface group	$-SO_3H$	$-SO_3H$ and ZrP
BET surface area (m^2/g)	25.1	19.1
Pore volume (cm^3/g)	0.214	0.073
Average pore diameter (nm)	34.1	16.1
Apparent density (g/cm^3)	0.58	0.87
Ion-exchange capacity (meq/g)	4.17	3.2[a]
ZrP content (mass%)	0	32.8

[a] Determined at pH 7.0.

cation exchanger. It can be seen that ZrP has been successfully loaded on D-001 beads according to the ZrP content variation before and after loading. The loaded ZrP increased the apparent density of polymer beads, but it also blocked some fraction of pores and resulted in a decrease of BET surface area and pore volume. The micrographs of D-001 and ZrP-001 beads (Fig. 1) indicate that the loaded ZrP is uniformly dispersed in the pore region. The Zr/P ratio in the ZrP-001 beads was determined as 1:2 by XPS analysis, which further demonstrated the dispersion of ZrP within D-001. The X-ray diffraction pattern of the ZrP-001 beads (Fig. 2) implied that ZrP dispersed within D-001 beads is essentially in the amorphous state though some absorption peaks were observed in the pattern, as elucidated elsewhere [19,20]. TGA results showed that ZrP-001 exhibits excellent thermal stability under 300 °C and the weight loss before 300 °C was possibly attributed to the evaporation of the external water within the sorbent (Fig. 3).



(a) D-001



(b) ZrP-001

Fig. 1. SEM micrographs of (a) a strongly acidic cation exchanger D-001, and (b) its ZrP-loaded derivative ZrP-001.

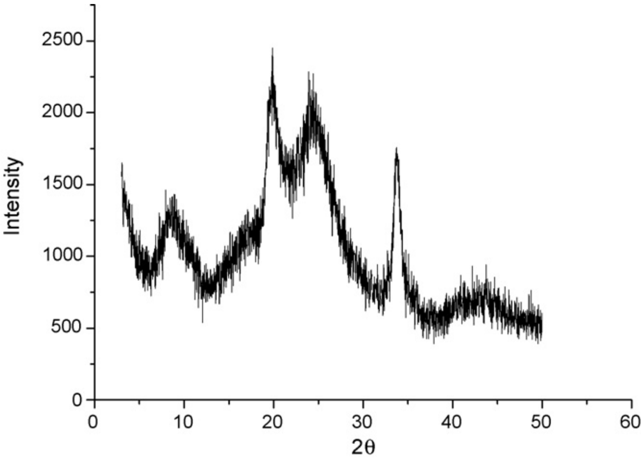


Fig. 2. XRD spectra of the hybrid sorbent ZrP-001.

It should be noteworthy that there exist two different exchangeable sites for uptake of heavy metals within ZrP-001: one is the sulfonic group on the host material D-001 and the other is ZrP loaded onto D-001. Our previous study revealed that only half of the exchangeable hydrogen ion in amorphous ZrP is accessible for ion exchange under neutral or acidic solution pH, and the rest is available only for the alkaline solution [13]. Therefore, uptake of heavy metals by ZrP-001 can be realized according to the following processes:

$$Zr(HPO_4)_2 + 1/2M^{2+} \rightleftharpoons ZrM_{0.5}H(PO_4)_2 + H^+ \quad (1)$$

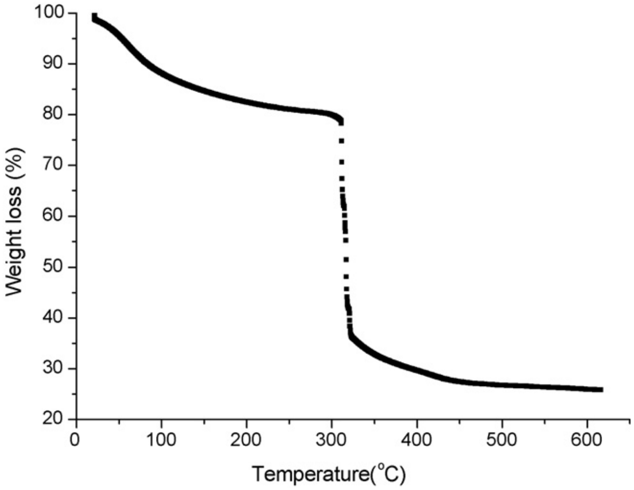


Fig. 3. Thermogravimetric curve of ZrP-001 prepared in the study.

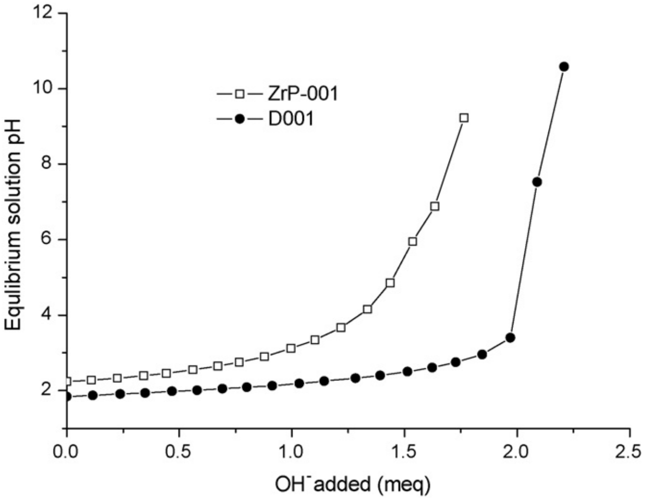


Fig. 4. Comparison of pH–titration curves of D-001 and ZrP-001 using 0.1 M NaOH solution at 303 K.

$$\text{R-SO}_3\text{H} + 1/2\text{M}^{2+} \rightleftharpoons \text{R-SO}_3\text{M}_{0.5} + \text{H}^+ \quad (2)$$

where M represents the heavy metals and R is the polymeric matrix of D-001.

A steeper pH–titration curve of D-001 than ZrP-001 (Fig. 4) was attributed to the specific ion-exchange property of amorphous ZrP with the sorbent. The ion-exchange capacity of ZrP-001 determined experimentally (3.20 meq/g) is less than the calculated value (4.07 meq/g) at neutral pH, implying that parts of the sulfonic acid groups or ZrP within ZrP-001 are inaccessible as a result of the ZrP dispersion.

3.2. *Effect of solution pH on sorption*

Effect of solution pH on uptake of heavy metals by ZrP-001 was examined and the results were presented in Fig. 5. Generally, higher solution pH (under acidic or neutral conditions) is more favorable for uptake of heavy metals onto ZrP-001. Note that further increase of solution pH from 3 to 6 displayed negligible variation of Pb^{2+} uptake. It was attributed the specific capacity of ZrP-001 towards Pb^{2+} and will be elucidated in the forthcoming section. The specific pH-dependent trend can be explained by the ion-exchange mechanism between solution and ZrP-001, and negligible uptake of the heavy metals at pH less than 0.5 suggested that the used ZrP-001 might be regenerated by strongly acidic solution, which was further demonstrated in the desorption experiments below.

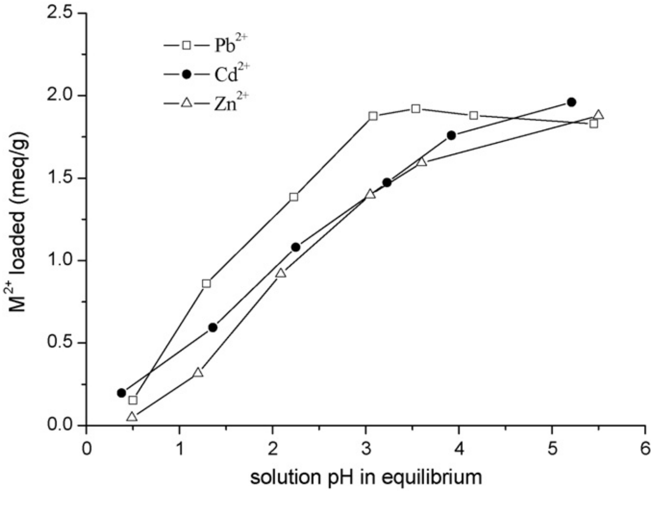


Fig. 5. Effect of solution pH on the uptake of heavy metals onto ZrP-001 at 303K. (0.050 g sorbent were added into 100-ml solution containing 1 mmol/l of each test metal).

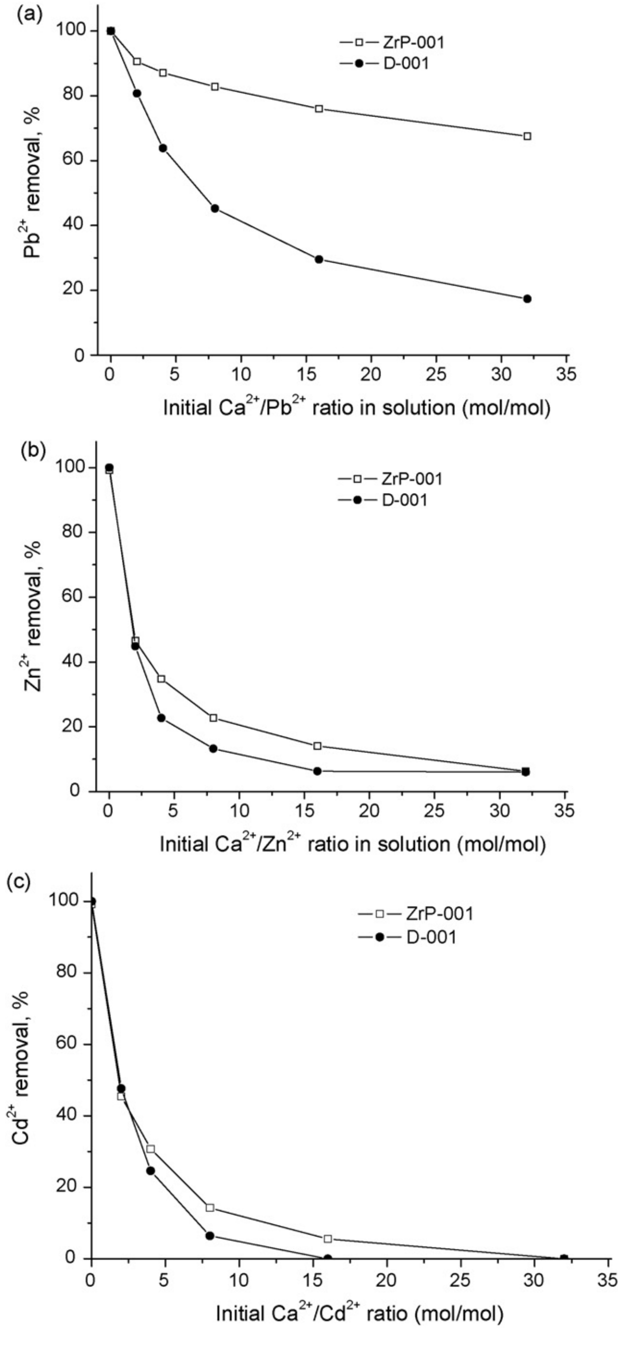


Fig. 6. Effect of Ca^{2+} on uptake of heavy metal ions onto ZrP-001 and D-001 at 303 K (a) Pb^{2+}, (b) Zn^{2+}, (c) Cd^{2+} (solution pH for Pb^{2+}, 4.3; Zn^{2+} and Cd^{2+}, 5.0).

Table 2
Distribution coefficients (K_d) of three metals for ZrP-001 and D-001 in presence of Ca^{2+} at different levels in solution (experimental data from Fig. 6)

Heavy metals (M^{2+})	Sorbent	K_d (l/g) at different initial Ca^{2+}/M^{2+} ratios in solution				
		2	4	8	16	32
Pb^{2+}	ZrP-001	38.5	27.1	19.3	12.7	8.31
	D-001	16.8	7.05	3.30	1.68	0.84
Zn^{2+}	ZrP-001	3.48	2.13	1.17	0.65	0.27
	D-001	3.25	1.17	0.609	0.27	0.21
Cd^{2+}	ZrP-001	3.96	1.77	0.67	0.23	0
	D-001	3.64	1.31	0.28	0	0

3.3. Effect of Ca^{2+} on sorption

Taken into account the fact that innocuous cations always coexist at high levels in waters and industrial wastewater, we next tested their competitive effect on uptake of heavy metals onto ZrP-001 and D-001 by selecting Ca^{2+} as a competitive cation. As shown in Fig. 6, the Pb^{2+} removal efficiency onto ZrP-001 was slightly influenced by Ca^{2+} addition. By contrast, removal of Zn^{2+} and Cd^{2+} was greatly influenced under the identical conditions, indicating that Pb^{2+} was more preferably sorbed onto ZrP-001 than other two metals. In general, divalent cations with low hydration energies are preferably sorbed by cation exchangers [21]. The Gibbs free energies of hydration are −1425 KJ/mol for Pb^{2+}, −1955 KJ/mol for Cd^{2+}, −1755 KJ/mol for Zn^{2+} and −1505 KJ/mol for Ca^{2+} [22]. This partly explains the preference sequence of these metals. On the other side, ZrP-001 was also found to exhibit more favorable sorption of all the three metals than D-001, as depicted in Fig. 6. We have previously shown that by comparison with D-001, amorphous ZrP offers selective uptake of heavy metals including Pb^{2+}, Zn^{2+}, and Cd^{2+} in presence of Ca^{2+} competition [13]. Moreover, the non-diffusible negatively charged sulfonic acid group within ZrP-001 would display the Donnan membrane effect, and thereby greatly enhance permeation of the targeted metal ions and favor their uptake. The Donnan membrane effect of the host material for hybrid sorbent preparation was explained by Cumbal and Sengupta [14].

To quantify the selectivity of both sorbents, the distribution ratio K_d (in l/g) was determined by the following equation [23]:

$$K_d = \frac{\text{mmol of heavy metals/g sorbent}}{\text{mmol of heavy metals/l solution}} \qquad (3)$$

The K_d values thus provide a measure of the sorptive ability for heavy metals per gram of sorbent. Table 2 listed the calculated K_d values of ZrP-001 and D-001 towards heavy metals in presence of Ca^{2+} at different levels. Despite the competing effect of Ca^{2+}, a substantially larger K_d value of ZrP-001 than D-001 promoted us to believe that ZrP-001 offers an enhanced removal of heavy metals from waters than D-001.

3.4. Sorption kinetics

Fig. 7 presents the plots of uptake of heavy metals versus contact time for ZrP-001 and D-001. As expected, a faster kinetics

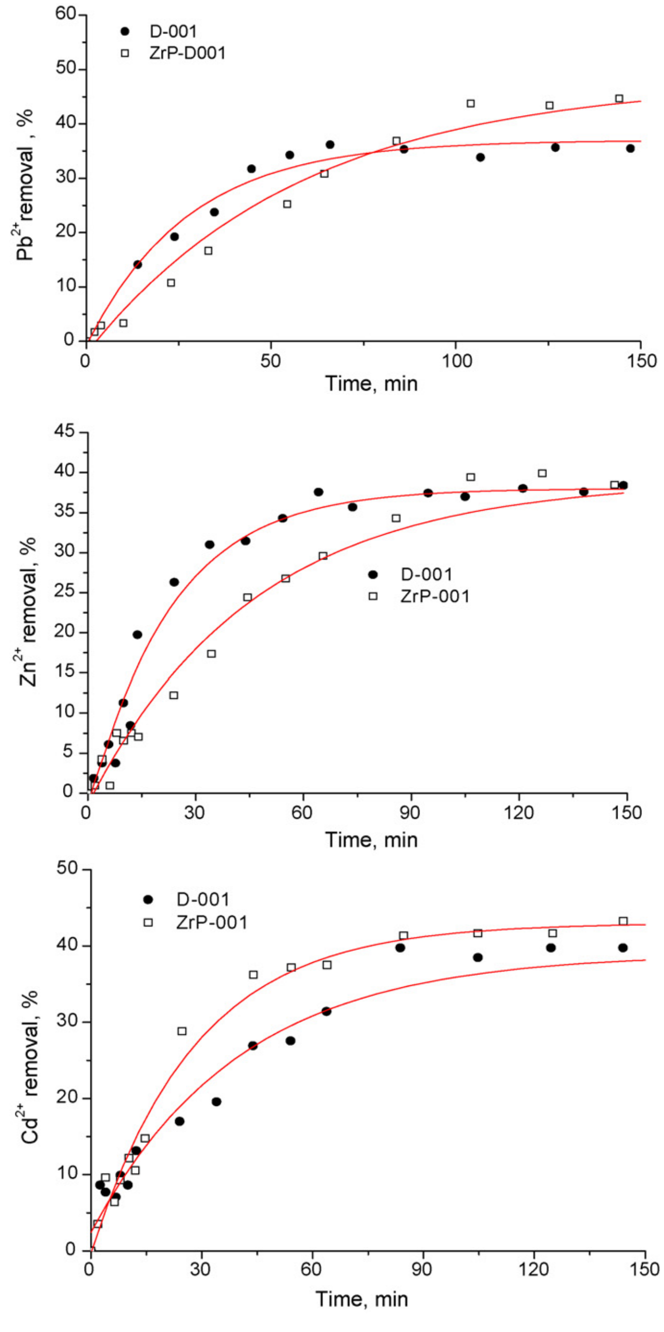


Fig. 7. Sorption kinetics of the heavy metals onto D-001 and ZrP-001 at 303 K. (0.150 g sorbent was added into 1000 ml solution containing 0.60 mM of each heavy metal ion; solution pH for Pb^{2+}, 4.3; Zn^{2+} and Cd^{2+}, 5.0).

of D-001 than ZrP-001 for all the metals was observed possibly because ZrP dispersion onto D-001 would partly block pore regions and thereafter lower the average pore diameter of the sorbent (Table 1). However, the contact time of about 150 min is long enough for ZrP-001 to achieve the sorption equilibrium. Kinetic data for both sorbents were then represented by the pseudo-first-order model [24].

$$\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303}t \tag{4}$$

Where q_e and q_t are the amounts extracted in equilibrium and at time t, respectively, k_1 is the first-order sorption constant. Higher correlation coefficients (Table 3) indicated that uptake of heavy metals onto both sorbents can be approximated favorably by the pseudo-first-order model. Higher k_1 value of D-001 than ZrP-001 is consistent with the pore structure variation before and after ZrP dispersion.

3.5. *Fixed-bed column adsorption and regeneration*

Fig. 8 illustrated an effluent history of separate fixed-bed columns packed with ZrP-001 for feeding solutions containing targeted heavy metal ions and competing cations (Na^+, Ca^{2+}, and Mg^{2+}) by comparison with D-001. Note that Pb^{2+} breaks through much earlier on D-001 than ZrP-001, though IEC of D-001 is higher than ZrP-001. Again, this proves sorption enhancement of Pb^{2+} on ZrP-001 than D-001. C/C_0 in excess of a unity on breakthrough curves for Zn^{2+} and Cd^{2+} sorption on D-001 (Fig. 7b and c) is caused by the elution effect of both metals by competing cations [23]. In other words, a fraction of Zn^{2+} and Cd^{2+} initially loaded on D-001 can be replaced by competing cations when insufficient sorption sites are available. This was, however, not observed for ZrP-001 due to its improved selectivity for both metals over innocuous cations.

The regeneration test of the spent ZrP-001 beads from the column runs was also conducted by 1 M HNO_3 at 303 K. As expected, all the loaded heavy metals were amenable to a complete desorption by about four-bed volumes (BV) of HNO_3 solution with the regeneration efficiencies larger than 99%. Additionally, HCl solution could also achieve a similar regeneration efficiency under otherwise identical conditions. Moreover, negligible loss of ZrP within ZrP-001 was demonstrated by the fact that less than 1% loss of ZrP in ZrP-001 beads was detected after five-column cycles.

Table 3
Sorption kinetic parameters of heavy metals onto ZrP-001 and D-001 at 303 K (experimental data from Fig. 7)

Heavy metals	Sorbents	K_1, 10^{-2} min^{-1}	R^2
Pb^{2+}	D-001	3.82	0.996
	ZrP-001	2.00	0.997
Zn^{2+}	D-001	4.41	0.976
	ZrP-001	3.26	0.971
Cd^{2+}	D-001	6.14	0.996
	ZrP-001	2.25	0.987

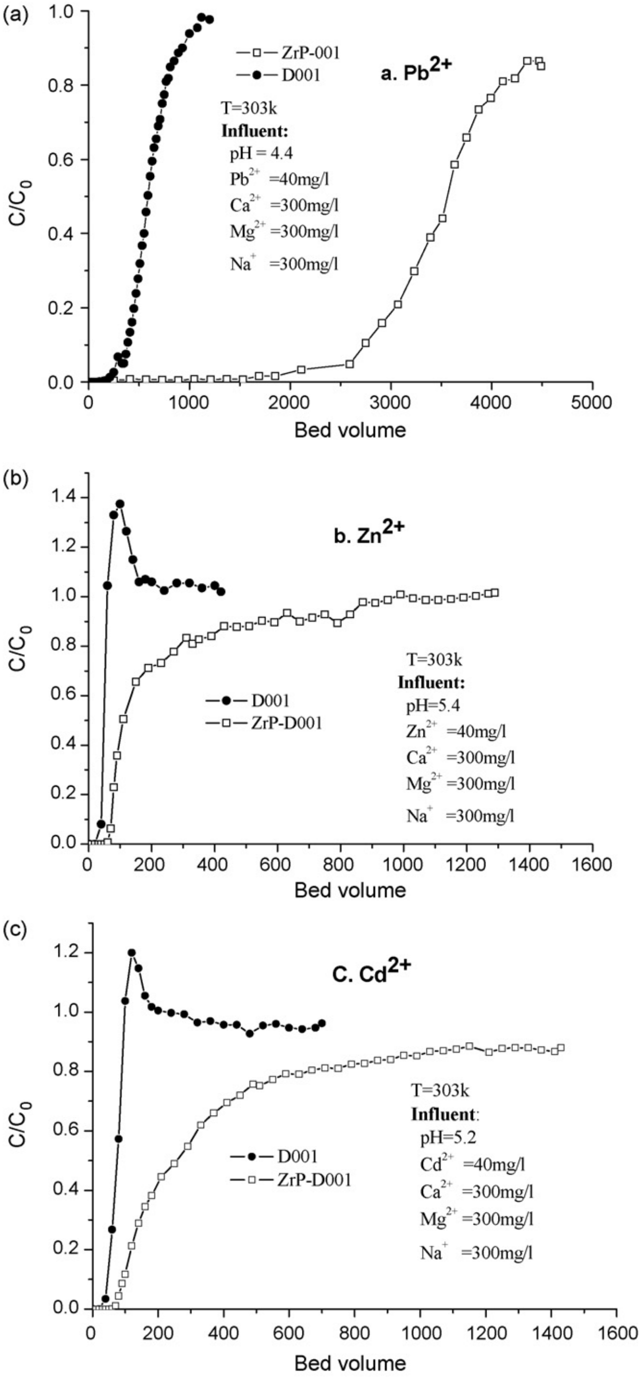


Fig. 8. Comparison of breakthrough curves of heavy metal sorption onto ZrP-001 and D-001 during three separate fixed-bed column runs at 303 K.(a)Pb^{2+}, (b)Zn^{2+}, (c)Cd^{2+}.

4. Conclusions

A novel hybrid sorbent ZrP-001 was prepared by loading zirconium phosphate (ZrP) onto a strongly acidic cation exchanger D-001. Sorption of Pb^{2+}, Zn^{2+} and Cd^{2+} onto ZrP-001 was found to be pH-dependent due to the ion exchange mechanism. Competitive effect of Ca^{2+} on sorption of heavy metals indicated that ZrP-001 exhibited more preferable sorption of all the metals than D-001 with the preference order $Pb^{2+} >> Zn^{2+} \approx Cd^{2+}$. Fixed-bed sorption and regeneration results further demonstrated that ZrP-D001 is a potential candidate for removing heavy metals from contaminated water.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jhazmat.2007.07.012.

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