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Adsorption properties for heavy metal ions from aqueous solution of the novel chelating resin loaded quaternary ammonium salt

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ABSTRACT

The novel Schiff bases chelating resin loaded quaternary ammonium salt (CPS-QA-SBGL) had been synthesized with the cross-linked Polystyrene-Dimethylamine porous beads (CPS-DMA) as raw materials, which were bonded with glycine on the surface via a quaternary ammonium unit as a linker. CPS-QA-SBGL had been characterized by elemental analysis, infrared spectra and thermo-gravimetric analysis (TGA)-differential scanning calorimetry (DSC) analysis. Factors influencing the uptake of heavy metal ions such as pH, initial metal ion concentrations, shaking time and temperature were investigated systematically by batch experiments. The adsorption equilibrium study exhibited that heavy metal ions adsorption of CPS-QA-SBGL followed a Langmuir isotherm model better than a Freundlich model. The optimum adsorption conditions at 298 K were obtained: the initial concentrations of Pb^{2+}, Cd^{2+}, Cu^{2+} and Cr^{3+} were 600 mg/L, pH 5, the equilibrium adsorption uptake time was in 30 min. The kinetic data of adsorption of heavy metal ions on CPS-QA-SBGL were best described by a pseudo-second-order equation, indicating their chemical adsorption process. In addition, dynamic parameters suggest that adsorption of heavy metal ions onto CPS-QA-SBGL is involving an exothermic reaction and occurs spontaneously ($\Delta G < 0$).

Keywords: Schiff base; Quaternary ammonium salt; Adsorption; Heavy metal ions

1. Introduction

With the development of industry and technology, water pollution by heavy metal ions is becoming one of the most serious problems and has attracted widely attention all over the world due to their toxicity [1] and tendency to bioaccumulation [2]. Heavy metal pollution caused by Pb^{2+} and Cd^{2+} is most serious to the human body. These heavy metal ions enter into the ecosystem by adsorbing in soil, plants and animals. Lead enters the organism primarily via the alimentary and/or the respiratory tract. The accumulation of Pb in human body may lead to anemia and paralysis of the muscles. Cadmium poisoning may lead to the damage of the lung, kidney and bones.

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Thus, it is necessary to remove them from the contaminated water.

Various technologies had been applied to the removal of heavy metal ions, such as precipitation and flocculation [3,4], active carbon adsorption [5], biochemical regeneration [6] and ion exchange [7]. However, these methods are limited in their use due to their different disadvantages of secondary pollution, high costs or poor results.

Chelating resin [8] had been widely designed and synthesized to remove heavy metal ions due to its higher adsorption capacity, desorption and regeneration easily and long-service life. The novel Schiff bases chelating resin loaded quaternary ammonium salt (CPS-QA-SBGL) had been designed and synthesized with advantages such as steady chemical property, good hydrophilic property, higher adsorption capacity and the spherical structure make it is easy to separate from waste water. In view of above, the novel chelating can be applied to the waste water measurement.

2. Experimental

2.1. Instruments

Fourier transform infrared spectra were recorded on a Nicolet MAGNA-IR 550 (II) spectrophotometer using KBr pellets. The concentration of various metal ions was determined by TAS-990 flame atomic absorption spectrometry (FAAS) with an air-acetylene flame (Beijing Purkinje General Instrument Limited Company, Beijing). A Shimadzu TG/DTA simultaneous measuring instrument DTG-60/60H (Kyoto, Japan) was used for TGA and DSC. Elemental analysis was carried out on Carlo Erba-EA1108 elemental analyzer.

2.2. Reagents and solutions

Chloromethylated cross-linked polystyrene porous beads (CPS) were purchased from Hecheng Science and Technology Factory, Tianjin, China and used as received. 5-Cholromethylsalicylaldehyde (5-CS) was synthesized according to our previous work [9]. The dimethylaminomethylated CPS (CPS-DMA) were prepared with CPS and dimethylamine in dioxane solution, in which nitrogen content is 5.58%. Glycine and the other reagents were reagent grade and were used without further purification.

The stock solutions of nitrate salts of Pb^{2+}, Cd^{2+}, Cu^{2+} and Cr^{3+} at the concentration of 2,000 mg/L in 1%HNO_3 were prepared in distilled water and standardized by complexometric titration before use. The working solutions of the heavy metal ions were prepared appropriately to dilute the stock solutions. The buffer solutions for pH adjustment were acquired containing suitable amounts of NaOH and HNO_3.

2.3. Synthesis of CPS-QA-SBGL

The general synthetic route for chelating resin is shown in Fig. 1.

2.3.1. Synthesis of CPS-QA-S

CPS (5.0 g) were added into the dioxane solution (35 mL) containing dimethylamine (15 g) and swollen for 2 h in a 3-necked flask, then refluxed for 48 h under the condition of mechanical stirring. The aminated CPS (CPS-DMA) were obtained and its nitrogen content was 5.58%. Then CPS-DMA were added into the ethyl acetate solution (50 mL) containing 5-CS (21 g) and then were stirred at the room temperature for 48 h. The pale white spherical product (CPS-QA-S) was filtered off, washed with heated ethanol and distilled water. After being dried under vacuum at 50℃ for 48 h, 8.26 g of the resin containing the quaternary ammonium and salicylaldehyde units was obtained. Elemental analysis of CPS-QA-S gave N 2.63%, Cl 6.91%.

2.3.2. Preparation of CPS-QA-SBGL

CPS-DMA-S (8.26 g) and glycine (2 g) were added into the alcohol solution (30 mL), and then refluxed for 50 h. The pale yellow spherical product was filtered off, washed with 20 mL of the acidic ethanol for three times and dried at 50℃ for 48 h under vacuum condition, 9.13 g of the pale yellow chelating resin (CPS-QA-SBGL)was obtained. Elemental analysis of CPS-QA-SBGL gave N 3.22% and Cl 6.54%.

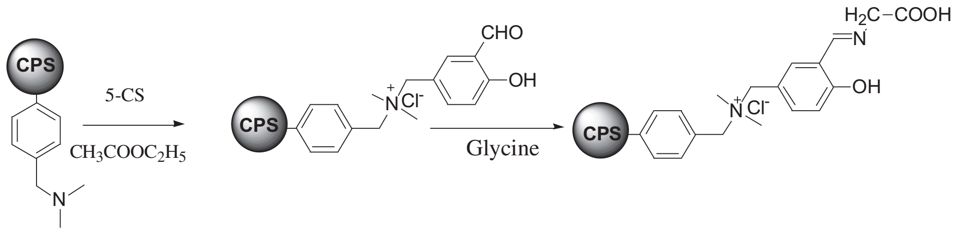

Fig. 1. Synthetic route.

2.4. Batch experiment

Adsorption of Pb^{2+}, Cd^{2+}, Cu^{2+} and Cr^{3+} was carried out by a batch technique at 298 K. 100 mg of the chelating resin was added into 20 mL aliquot of tested metal ion solution in a shaker adjusted to desired shaking speed.

The residual concentration of heavy metal ion was detected by FAAS. The adsorption capacities were calculated using the following equation [10]:

$$q = \frac{c_0 - c}{c_0} \times 100 \tag{1}$$

$$Q_m = \frac{c_0 - c}{W} \times V \times 1,000 \tag{2}$$

where q is the adsorptivity (%), Q_m is the metal ion adsorption capacity, mg/g; C_0 and C are the initial and final concentrations of the metal ion solutions, mg/mL; V is the volume of the solution, mL; W is the weight of dry resin, g.

2.4.1. Shaking time

The effect of shaking time on adsorption capacity of CPS-QA-SBGL for metal ions had been studied. 100 mg of the chelating resin was added into 20 mL aliquot of tested metal ion solution in a shaker adjusted to desired shaking speed and optimum pH.

2.4.2. Optimum pH

100 mg of the dried resin was added into a heavy metal ion solution (20 mL, 600 mg/L) in the beaker flask, and then adjusted the heavy metal ion solution prior to equilibration over a pH range from 2 to 8 by HNO_3 and NaOH, and shaken for 24 h at 298 K to reach equilibrium.

2.4.3. Initial concentration

100 mg of the dried resin was added into a heavy metal ion solution (20 mL, 25, 50, 100, 200, 400, 600, 800, 1,000, 1,200 mg/L) in the beaker flask, and then adjusted the heavy metal ion solution to optimal pH by HNO_3 and NaOH, and shaken for 24 h at 298 K to reach equilibrium.

2.4.4. Dynamic experiments

100 mg of the dried resin was added into a heavy metal ion solution (20 mL, 600 mg/L) in the beaker flask, and then adjusted the heavy metal ion solution to optimal pH by HNO_3 and NaOH, and shaken for 24 h at 298, 303 and 313 K to reach equilibrium, respectively.

2.4.5. Elution experiments

Elution experiments were performed by equilibrating 100 mg of dried resin with 20 mL (600 mg/L) of heavy metal ions (Pb^{2+}, Cd^{2+}, Cu^{2+} and Cr^{3+}) for 24 h at 298 K. The total uptake was estimated then the resin was decanted and the resin was washed thoroughly with distilled water. The resin (loaded heavy metal ions) was equilibrated for 12 h in 20 mL of the eluent 1.5 mol/L HNO_3 solutions. The concentrations of the released metal ions were detected via FAAS. Afterwards, the resin was washed repeatedly with distilled water till the filtrate become free of the eluent. The previous steps were repeated for 10 cycles.

3. Results and discussion

3.1. Structural characterization

3.1.1. FTIR analysis

The main peaks of CPS-QA-S in FT-IR spectrum are 3,428, 3,031, 2,925, 2,720, 1,698, 1,550 and 1,437 cm^{-1} correspond to the stretching vibration of OH (phenolic), =CH, CH_3, CH_2, C=O, C=C and C–O, respectively (Fig. 2).

The FTIR spectrum of CPS-QA-SBGL showed bands at 3,349–3,303, 3,051, 2,935, 1,680, 1,548, 1,435 and 1,112 cm^{-1}, which were assigned to the stretching frequencies of COOH, OH, =CH, CH_3, C=C and C–O, respectively. The peak at 1,698 cm^{-1} belongs to aldehyde C=O had disappeared in CPS-QA-S IR spectrum, the new peaks at 1,680 and 1,435 cm^{-1} were due to carboxylate COOH stretching vibration.

3.1.2. TGA–DSC analysis

CPS-QA-SBGL weight loss about 6% when temperature went up to 109℃ could be seen in the TGA curve, which may be attributed to the loss of free H_2O. The thermo-degradation temperature is between 195 and 287℃ of CPS-QA-SBGL, which may be due to the loss of quaternary ammonium salt and aromatic carboxylate. The final stage of thermo-degradation of CPA-QA-SBGL is between 380 and 453℃, which may be due to the degradation of the chloromethylated CPS. TGA–DSC results showed that the chelating resin possessed good thermal stability (Fig. 3).

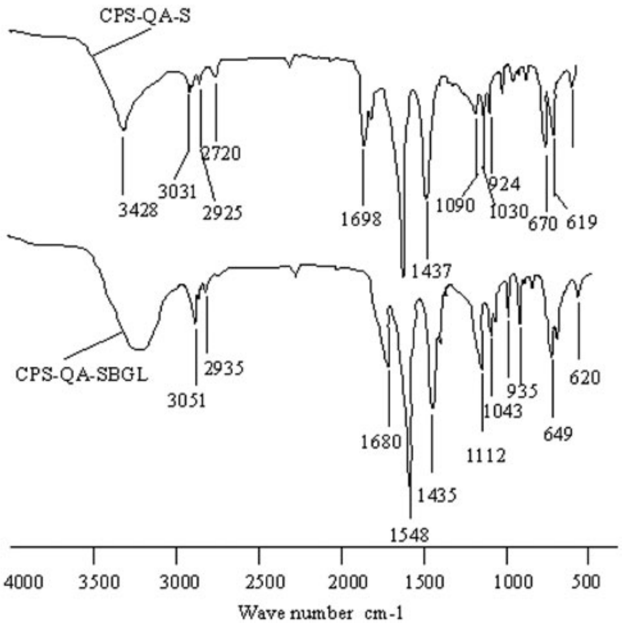

Fig. 2. FTIR spectrum of CPS-QA-SAGL.

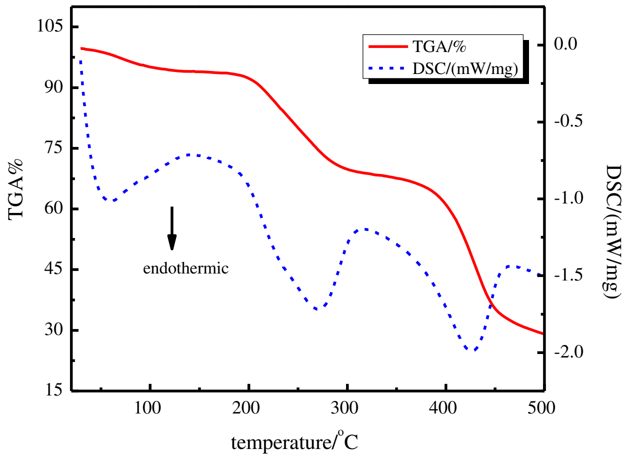

Fig. 3. TGA–DSC of CPS-QA-SBGL.

3.2. *Effect of shaking time on metal ions sorption capacity*

As seen from the Fig. 4, the heavy metal ion adsorption rate and capacity of the chelating resin had increased sharply in 20 min. Then 30 min later, adsorption capacity would have no further variation with time. The chelating resin had quick adsorption rates for heavy metal ions mainly due to the structure. The chelating resin containing quaternary ammonium salts loaded Schiff bases would increase the hydrophilic property of beads surface, and the diffusion time of the heavy metal ions from the surface to the inner parts of the beads had been shortened. As a result, CPS-QA-SBGL has good accessible and high-hydrophilic chelating sites to bind heavy metal ions in aqueous solutions.

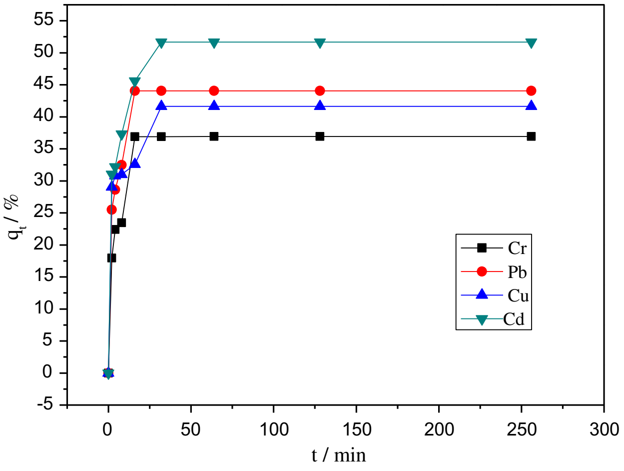

Fig. 4. Relation between adsorptive capacity and time of CPS-QA-SBGL.

3.3. *Adsorption kinetics of heavy metal ions adsorption process*

The kinetics of adsorption is important from the point of view that it controls the adsorption rates. In order to achieve a depth sight into the mechanisms of metal ions adsorption process better, pseudo-first-order and pseudo-second-order had been used for analyzing adsorption behaviour.

The pseudo-first-order kinetic model assumes that the adsorption process mechanism is involving in physical adsorption and the equation is used as following [11]:

$$\log{(q_e - q_t)} = -\frac{K_1}{2.303}t + \log q_e \tag{3}$$

where K_1 is the constant of the pseudo-first-order adsorption in min^{-1}.

The pseudo-second-order kinetic model is based on chemical adsorption (chemisorption) and the equation is given by [12]:

$$\frac{t}{q_t} = \frac{t}{q_e} + \frac{1}{K_2 q_e^2} \tag{4}$$

The K_2 is the rate constant of pseudo-second-order adsorption in $\text{g mg}^{-1}\,\text{min}^{-1}$.

As seen from Fig. 5, the linear spots fit with the pseudo-second-order kinetic model better than the first one. The estimated parameter values and regression correlations calculated by nonlinear regression analysis are listed in Table 1. The pseudo-second-order kinetic

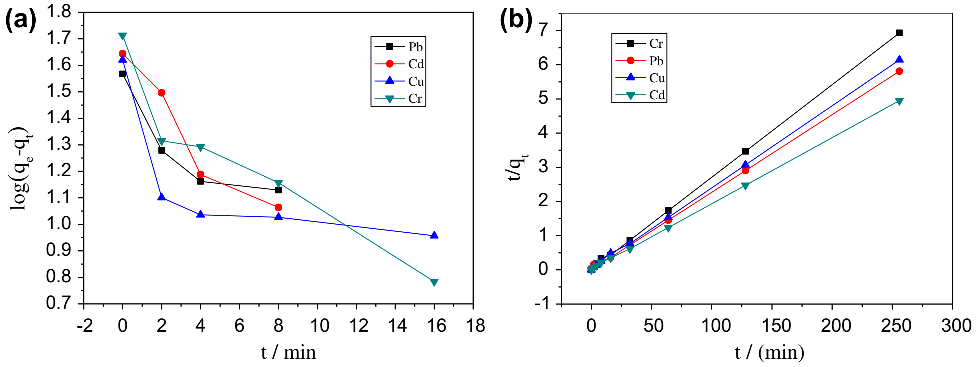

Fig. 5. Pseudo-first-order (a) and pseudo-second-order (b) kinetics adsorption of Pb^{2+}, Cd^{2+}, Cu^{2+} and Cr^{3+} onto CPS-QA-SBGL.

model got the higher correlations coefficients ($R^2 > 0.999$) according to the Table 1. In sum, metal ions adsorption process on the CPS-QA-SBGL is promoted by a chemical process.

3.4. Effect of pH on metal ions sorption capacity

The pH of the aqueous solution was clearly an important parameter that affected the adsorption process. It is generally known that pH can affect the surface charge properties of the chelating resins in terms of dissociation and protonation of the chelating groups. Maybe this is the reason why pH has significant influence on metal ions sorption capacity. Fig. 6 shows that the effect of pH on metal ions sorption capacity.

As seen from Fig. 6, the adsorption capacities were highest at pH 4–6. It can be indicated that the adsorption capacities increase with a pH rise from 2 to 4, but decrease with pH rise when pH > 6. The maximum adsorption capacities of CPS-QA-SBGL were 44.13 mg/g for Cr^{3+} with pH at 5.15, 44.17 mg/g for Cu^{2+} with pH at 4.92, 50.58 mg/g for Pb^{2+} with pH at 5.45 and 62.32 mg/g for Cd^{2+} with pH at 4.15. Based on the electron donating nature of the oxygen and nitrogen groups in the CPA-QA-SBGL and the electron accepting nature of Pb^{2+}, Cd^{2+}, Cu^{2+} and Cr^{3+}, the ion exchange mechanism and complexation could be considered. However, the adsorption capacities decline sharply when the pH value is lower than 4 or higher than 6. The probably reasons [13] are: (1) at lower pH conditions, the concentration of H^+ is higher, the H^+ ions competed with the metal ions for the biding sites on the chelating resin, the positive charge repeled and prevented metal ions from transferring into the resin surface, and the complexation abilities of the resins ligands became weak, resulted in lower adsorption capacities of the resins. Thereby, the metal ions may be adsorbed at the surface of CPS-QA-SBGL by ion exchanging and complexation. (2) Under higher pH conditions, the abundant OH ions may react with metal ions to produce precipitation; this may lead the adsorption capacities to getting sharply down.

3.5. Effect of initial metal ions concentration with adsorption isotherm models

The relation between the amount of adsorbate on adsorbent as a function and its concentration at a constant temperature in the equilibrium solution is called adsorption isotherm. And it can help researchers to understand the mechanism in adsorption progress. As seen from Fig. 7, when the heavy metal ions initial concentration was lower than 200 mg/L, the adsorption capacity relatively rapidly increased with increasing of the heavy metal ions concentrations. When the

Table 1
Kinetic adsorption parameters

	Pseudo-first-order		Pseudo-second-order		
Heavy Metal ions	K_1	R^2	K_2	q_e	R^2
Cr^{3+}	0.1147	0.728	3.404×10^4	37.175	0.9997
Pb^{2+}	0.1720	0.9062	5.412×10^4	44.444	0.9997
Cu^{2+}	0.0659	0.4526	4.987×10^4	42.017	0.9997
Cd^{2+}	0.1147	0.8892	9.864×10^4	52.083	0.9998

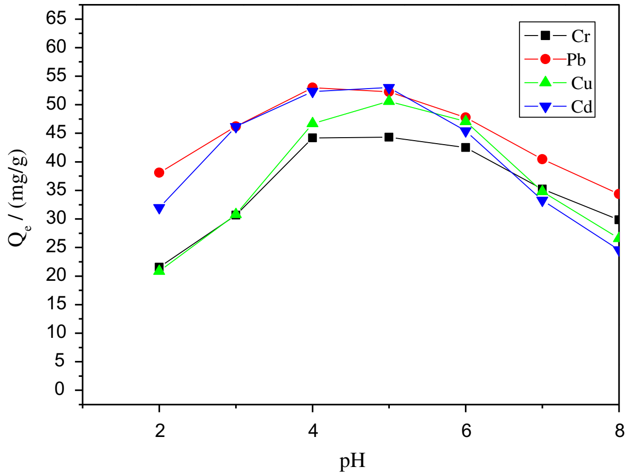

Fig. 6. Dependence of sorption capacity of CPS-DMA-SABA on the pH.

heavy metal ions initial concentration was higher than 600 mg/L, the adsorption capacity increased slowly except for Cd^{2+}. The capacity of CPS-QA-SBGL was found to be 51, 59, 66.2 and 88.9 mg/g for Cr^{3+}, Cu^{2+}, Pb^{2+} and Cd^{2+}, respectively. The reason for the enhancement in adsorption capacities is that the chemical modification generated more new functional groups such as hydroxyl radicals and carboxyl groups suitable for metal ion adsorption. Moreover, the difference in the adsorption capacity of different metal ions is in accord with the ionic radius of metal ions and atomic weight. The Pauling ionic radius [14] of Cu^{2+}, Pb^{2+}, Cd^{2+}, Cr^{3+} is 0.72, 1.20, 0.97, 0.615 Å, respectively. Therefore, Pb^{2+} and Cd^{2+} presented a better affinity to the resin surface than Cu^{2+} and Cr^{3+}.

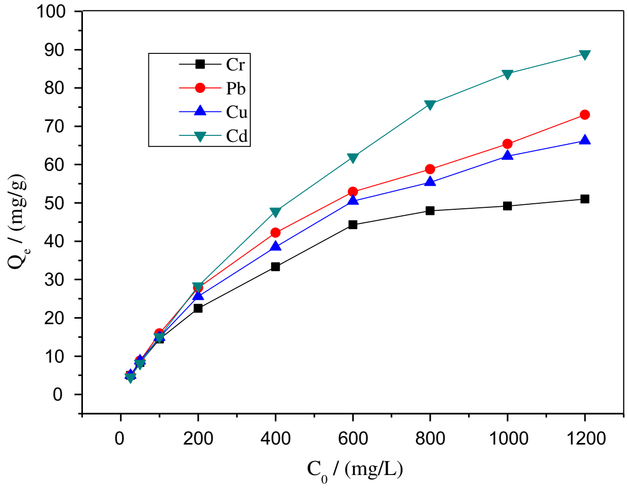

Fig. 7. Adsorption isotherm.

Two well-known adsorption isotherm models, Langmuir and Freundlich, are widely employed to expose the adsorption relationship. Two models' equations are given by [15,16]:

$$\frac{C_e}{Q_e} = \frac{1}{Q_{m^b}} + \frac{C_e}{Q_m} \tag{5}$$

$$\log Q_e = \frac{1}{n}\log C_e + \log K_f \tag{6}$$

where C_e is equilibrium solution concentration, mg/L; Q_e is the equilibrium adsorption capacity, mg/g; Q_m is representing the maximum amount of metal ion adsorption capacity, mg/g; b is the equilibrium Langmuir adsorption constant that refers to the bonding energy of adsorption, L/mg, n is the constant related the adsorption intensity and K_f is the Freundlich constant related to the adsorption capacity of the adsorbent, mg/g.

The quantitative relationship between initial heavy metal ion concentrations and the adsorption capacity is shown in Fig. 8. The calculated coefficient (b, Q_m, n and K_f) and linear regression coefficient (R^2) values for each Langmuir and Freundlich are shown in Table 2.

On the basis of characteristics of adsorption isotherms and regression coefficient (R^2) values, Langmuir equation ($R^2 > 0.99$) fits better than Freundlich model for the experiment data.

3.6. *Effect of temperature on metal ions sorption capacity*

The effect of temperature on metal ions sorption capacity was also discussed in this paper. The distribution ratio ($D = Q_e/C_e$) was measured under the condition of the same metal ion, same initial metal ion concentration, same pH and different temperature (298, 303 and 313 K).

The dynamic constants were calculated by the following equation [17]:

$$\log D = -\frac{\Delta H}{2.303RT} + \frac{\Delta S}{R} \tag{7}$$

$$\Delta G = \Delta H - T\Delta S \tag{8}$$

where R is constant, 8.314 J/(K mol); T is the temperature, K; ΔH is the heat of adsorption, kJ/mol; ΔS is the reaction entropy, J/(K mol); ΔG is the Gibbs free energy of adsorption, kJ/mol. It is supposed that ΔH and ΔS are not influenced by temperature in metal ion adsorption process. As shown in Fig. 9, the distribution ratio decreases with temperature increasing. It can also be indicated that the adsorption process is an exothermic reaction.

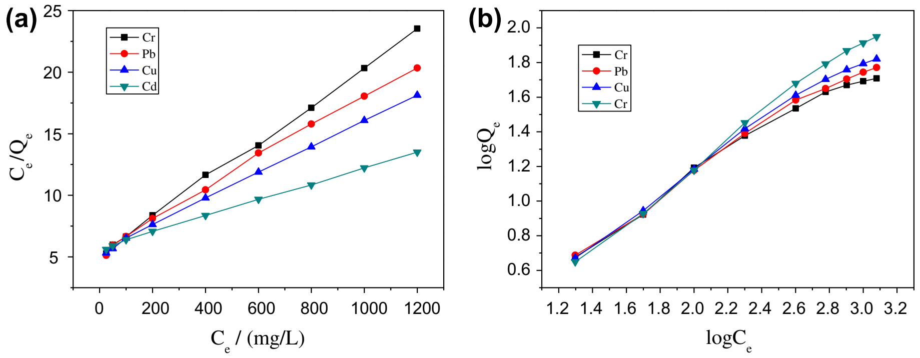

Fig. 8. Adsorption isotherms of metal ions onto CPA-QA-SBLG (a) Langmuir and (b) Freundlich model.

Table 2
Adsorption parameters of the Langmuir and Freundlich isotherm models

	Langmuir			Freundlich		
Heavy metal ions	Q_m	b	R^2	n	K_f	R^2
Cr^{3+}	65.78	2.948×10^{-3}	0.9989	1.690	0.9003	0.9827
Cu^{2+}	78.125	2.402×10^{-3}	0.9972	1.605	0.8030	0.9895
Pb^{2+}	95.59	2.032×10^{-3}	0.9989	1.518	0.7066	0.9914
Cd^{2+}	151.52	1.171×10^{-3}	0.9987	1.329	0.4775	0.9956

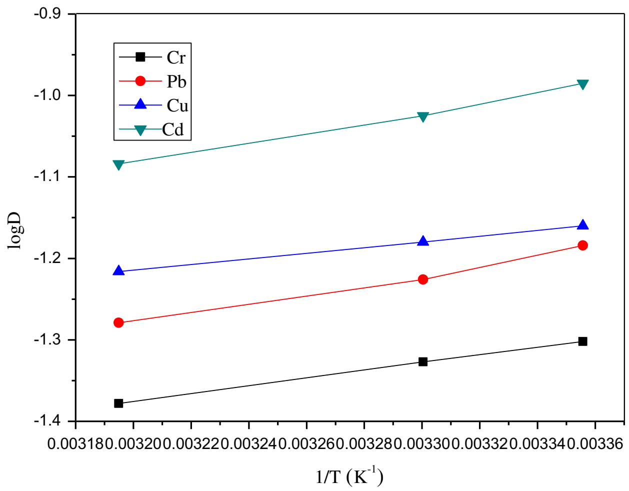

Fig. 9. Influence of temperature on distribution ratio.

The calculated coefficient (ΔH, ΔS and ΔG) and linear regression coefficient (R^2) values are shown in Table 3. The adsorption process is involving an exothermic reaction and occurs spontaneously ($\Delta G < 0$).

3.7. Repetitive use performance

HNO_3 (1.5 mol/L) was used as eluent for heavy metal ions (Pb^{2+}, Cd^{2+}, Cu^{2+} and Cr^{3+}), and this progress may be due to the possibility of ion exchange. The desorption ratios of heavy metal ions were all higher than 98%. Statistic experiments data showed that the adsorption capacity for heavy metal ions of the resin (CPS-QA-SBGL) decreased only about 3% after 10 times of adsorption-desorption-adsorption tests. The regenerated resins showed the heavy metal ions uptake capacity comparable wtih the fresh resins (Table 4).

Table 3
Dynamic parameters of adsorption

Heavy metal ions	ΔH (kJ/mol)	ΔS (J/(K mol))	ΔG (kJ/mol))	R^2
Pb^{2+}	−9.08	−24.05	−1.9131	0.9997
Cd^{2+}	−11.1	−26.03	−3.3431	0.9877
Cu^{2+}	−6.65	−19.56	−0.8211	0.9998
Cr^{3+}	−11.6	−25.18	−4.0964	0.9955

Table 4
Stability of CPS-QA-SBGL

Ions uptake	0	1	2	3	4	5	6	7	8	9
Pb^{2+}	42.7	42.27	42.16	42.14	42.09	41.85	41.63	41.54	41.48	41.42
Cd^{2+}	50.47	49.97	49.91	49.46	49.24	49.13	49.05	48.97	48.93	48.86
Cu^{2+}	44.66	44.23	44.17	44.08	43.91	43.67	43.53	43.48	43.42	43.31
Cr^{3+}	62	61.39	61.34	61.27	61.15	61.08	60.85	60.73	60.39	60.14

4. Conclusion

The novel Schiff bases chelating resin loaded quaternary ammonium salts (CPS-QA-SBGL) had been synthesized with the CPS-DMA porous beads as raw materials, which were bonded with glycine on the surface via a quaternary ammonium unit as a linker and can be used repeatedly. CPS-QA-SBGL had been characterized by elemental analysis, infrared spectra and TGA–DSC analysis. Based on the metal ion binding, complexation and exchange properties of the CPS-QA-SBGL, the adsorption properties for Pb^{2+}, Cd^{2+}, Cu^{2+} and $Cr3^{+}$ of CPS-QA-SBGL had been investigated with various experimental conditions. Results showed that pH, shaking time, temperature and initial concentrations of the heavy metal ions had remarkably influences on the adsorption behavior of the CPS-QA-SBGL. The static test showed that the equilibrium adsorption data fits to Langmuir isotherm model better than Freundlich model and it is an exothermic process. The optimum adsorption conditions at 298 K were obtained: the initial concentrations of Pb^{2+}, Cd^{2+}, Cu^{2+}, Cr^{3+} were 600 mg/L, pH 5, the equilibrium adsorption uptake time was in 30 min. In addition, the kinetic study indicates that the heavy metal ions adsorption process on the CPS-QA-SBGL can be described by pseudo-second-order kinetic model well and is mainly promoted by a chemical process.

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