Separation and recovery of lead from a low concentration solution of lead(II) and zinc(II) using the hydrolysis production of poly styrene-co-maleic anhydride

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ABSTRACT

The Pb—Zn separation/preconcentration technique, based on the complex formation reaction of Pb(II) and Zn(II), using a copolymer poly(styrene-co-maleic anhydride) (PSMA), without adding any carrier element was developed. The effects of several experimental parameters such as solution pH, temperature and adsorption time were studied. The experimental results show that the PSMA resin–Pb equilibrium was achieved in 2 min and the Pb(II) loading capacity is up to 641.62 mg g^{-1} in aqueous solution under optimum conditions, which is much higher than the Zn(II) loading capacity within 80 min. The adsorption test for Pb(II) indicates that PSMA can recover Pb(II) from a mixed solution of Pb(II), Zn(II) and light metals such as Ca(II) and Mg(II) with higher adsorption rate and larger selective coefficient. A further study indicates that PSMA as chelating resins recovering Pb(II) can be regenerated via mineral acid (6 M H$_2$SO$_4$). PSMA was synthesized by radical polymerization and tested as an adsorbent for the selective recovery of Pb(II). In addition, the formation procedure and structure of Pb–PSMA complex were also studied. Both the PSMA and the Pb–PSMA complex were characterized by means of FTIR spectroscopy, elemental analysis, gel permeation chromatography (GPC) and atomic absorption spectrometry (AAS).

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1. Introduction

Lead presents a threat to human health and environment when it exists in or is discharged into water resources because of its high toxicity and carcinogenicity, and it is not biodegradable in nature [1–4]. The development of high-performance adsorbents for removing lead from wastewater and drinking water is considered as a research priority in the environmental protection field [5–11]. However, lead and zinc ions are usually found to coexist in minerals and wastewater, which makes the separation of them to be difficult. Therefore, the separation and recovery of lead from the mixture of lead and zinc is very urgent.

A vast literature body now exists that deals with lead separation [12–14], but the methods are either costly or ineffective. The use of polymer-bonded ligands in selective lead removal has been the subject of many research articles [15–18] and reviews [19–21], and there are many articles describing the use of polymer-supported ligands such as xanthate [22], thiourea [23], pyridine-based thiols [24], and dithiozone [25] for highly selective lead removal. However, these materials also adsorb other metal ions in significant quantities. Therefore, development of new and more effective adsorbents for selective recovering Pb(II) has become essential, at the same time they should be cost effective.

In our previous work, we have reported the synthesis of MMC (macromolecular metal complexes) containing rare earth metals with polymeric ligands and their application as florescent materials [26,27]. As part of our continuing investigation into the development of MMC, herein we evaluate the Pb—Zn separation through formation of Pb–PSMA complex and the lead loading capacity of PSMA under competitive and noncompetitive conditions. Adsorption experiments reveal that the maximum adsorption capacity of PSMA for Pb(II) is 641.62 mg g^{-1}, which is much higher than that of other polymer resins for Pb(II) reported in previous references, for example, 21.91 mg g^{-1} in Ref. [15] and 117.9 mg g^{-1} in Ref. [18]. At the same time, the PSMA resin does not adsorb Zn(II) and light metals (such as Ca(II) and Mg(II) ions) within 80 min, so the high adsorption selectivity of the PSMA resin to Pb(II) can efficiently separate lead from zinc. A detailed structure and properties of both PSMA and Pb–PSMA complex was also performed. The results indicate that the as-prepared Pb–PSMA complex possesses three-dimensional network structure, which does not dissolve in water or typical organic solvents, revealing that the PSMA resin can be used as a potential chelating adsorbent to recover lead from a mixed solution. For the purpose of developing practical approach for separating lead from zinc in industrial solutions, the adsorption behavior of the PSMA resin for Pb(II) and Zn(II) has been investigated.

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2. Experimental

2.1. Instruments

Vibration spectra from 4000 to 400 cm^{-1} were recorded on a Thermo Nicolect Mattson 2110 spectrometer (KBr discs). Element analysis for C and H was carried on an Elemental Vario-EL elemental analyzer. Metal contents were determined on a Hitachi 180-80 polarized Zeeman atomic adsorption spectrometer (AAS). The average molecular weight of PSMA was estimated by a SN-01A gel permeation chromatography (GPC), using THF as fluent.

2.2. Materials

Styrene (supplied by Sigma–Aldrich, Shanghai, China) was washed with 10% aqueous sodium hydroxide to remove the inhibitor, and followed by washing with water until it was neutral. All the other chemicals used in this work were purchased from Alfa Aesar (Beijing, China) and used as received without further purification, and they were of analytical reagent grade. Stock standard solutions of 1 mg mL^{-1} Pb^{2+}, Zn^{2+} and a Pb^{2+}, Zn^{2+} mixture containing light metal ions (Ca^{2+} and Mg^{2+}) were prepared by dissolving the appropriate amount of analytical or spectrograde metal-nitrates in de-ionized water, respectively.

2.3. Preparation of the PSMA resin

PSMA resin was obtained by free radical copolymerization of maleic anhydride and styrene with benzoyl peroxide as initiator at 70 °C. The synthetic route of PSMA is shown in Fig. 1, and the details of the synthetic procedure are described in the literature [28]. The average molecular weight was evaluated by GPC method. The molar ratio of maleic anhydride to styrene in PSMA, calculated from the elemental analysis, is 1:2, so the content of maleic anhydride in PSMA is 3.162 mmol g^{-1}.

2.4. Adsorption capacities

1 g of dried PSMA resin was put into 35 mL of dilute KOH (0.1 M) solution; the mixture was stirred at room temperature until it dissolved completely. The resulting polymeric potassium carboxylate (K-PSMA) solution was adjusted to pH 6–7, and then added to a Pb(NO$_3$)$_2$ aqueous solution containing 1.02 mg mL^{-1} Pb(II) ion. The mixture was stirred continuously, the concentration of Pb(II) ion was determined by AAS at different intervals (such as 2 min, 5 min, 30 min, 60 min and so on) till it remained unchanged. The formed precipitates (Pb–PSMA) were separated from the solutions by centrifugation, then washed with water to remove aqueous Pb(II) and any other impurities, finally washed with ethanol and dried in a vacuum desiccator. The adsorption capacity Q (mg g^{-1}) was calculated according to the following formula (1):

$$Q = \frac{(C - C_0)V}{2W} \tag{1}$$

where C_0 (mg mL^{-1}) and C (mg mL^{-1}) represent the initial concentration of Pb(II) and the equilibrium concentration of Pb(II) in supernatant after adsorption, respectively; V is the volume of the solution used for adsorption (mL); and W is the weight of the resin
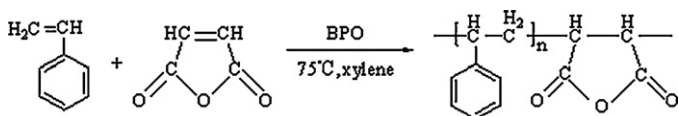

Fig. 1. Synthetic route of the PSMA resin.

Table 1
Elemental analysis of PSMA resin and Pb–PSMA complex.

	C	H	O	Pb
PSMA	72.05	5.34	22.61	
Pb–PSMA	46.93	4.25	35.76	13.04

(g). The formation procedure and structure unit of the Pb–PSMA complex are shown in Fig. 2.

2.5. Adsorption selectivity

To determine the adsorption selectivity of PSMA to Pb(II), the resin was placed in a binary mixed solution of Pb(II) and Zn(II) ions, in which the concentration of each metal ion was equal moles. Then, accurate amount of K-PSMA solution was put into this binary mixed solution and stirred continually. Finally, the Pb–PSMA complex was separated by centrifugation, and the concentrations of the metal ions in the binary mixture were determined by AAS.

2.6. Desorption and repeated use of the PSMA resin

For desorption of Pb(II), the Pb–PSMA complex was treated with 6 M H$_2$SO$_4$ solution. After filtration and being washed with water, the PSMA resin was dried under vacuum at 60 °C for repeated use. The detailed procedure is similar to the previous report [29], here, we do not expatiate it.

3. Results and discussion

3.1. Characterization of Pb–PSMA complex

3.1.1. Elemental analysis

The maleic anhydride content in the PSMA resin was determined by being titrated against potassium hydroxide aqueous solution (0.1 M) and further validated by elemental analysis. It was calculated with Eq. (2) as shown below:

$$\text{MA content } (\text{mmol g}^{-1}) = \frac{(V_1 - V_2)C}{2W} \tag{2}$$

where MA is maleic anhydride, V_1 and V_2 are the volume (mL) of KOH consumed by the resin and blank, respectively; C is the concentration of KOH aqueous solution; and W is the weight of the PSMA resin used. The content of maleic anhydride in the PSMA resin is 3.16 mmol g^{-1}. The elemental analysis results of the PSMA resin and Pb–PSMA complex were shown in Table 1.

The data in Table 1 show that the mole ratio of the maleic anhydride in the PSMA resin to Pb(II) is near to 1:1. This means that the mole ratio of carboxyl in K-PSMA to Pb(II) is near to 2:1.

3.1.2. Infrared spectra analysis

The bonding mode of Pb(II) to PSMA was examined by comparing the FTIR spectra of Pb–PSMA complex with that of the PSMA resin. As shown in Fig. 3, the FTIR spectra of Pb–PSMA complex are quite different from those of PSMA resin. The characteristic bands of the PSMA resin and Pb–PSMA complex are listed in Table 2. The results indicate that Pb–PSMA complex still has a weak absorption band at 1700–1705 cm^{-1} ascribed to $\nu_{C=O}$ of the uncoordinated carboxylic group. However, the absorption intensity of this band is

Table 2
FTIR spectra data of PSMA resin and Pb–PSMA complex (cm^{-1}).

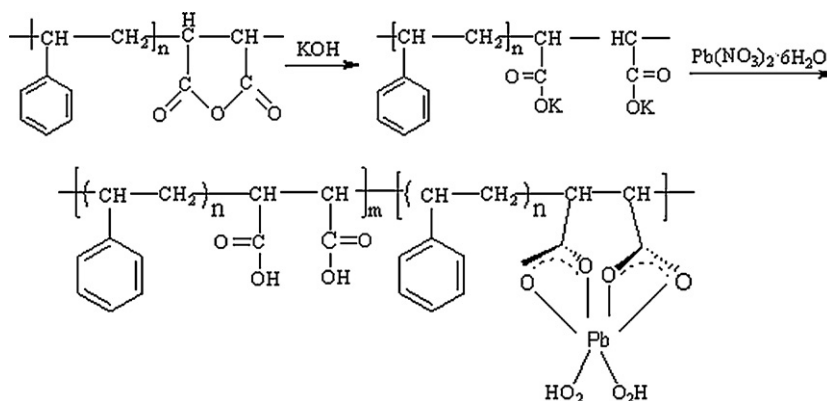| complexes	ν_{O-H}	$\nu_{C=O}$	ν_{as} COO$^-$	ν_{s} COO$^-$	$\Delta\nu_{as-s}$
PSMA	2923/3434	1705			
K-PSMA	2924/3423		1563	1403	160
Pb–PSMA	2926/3433	1704	1544	1401	143

Fig. 2. Formation procedure and structure unit of Pb–PSMA complex.

apparently lower than that of the PSMA resin. Therefore, the $\nu_{C=O}$ absorption band at ca. 1704 cm^{-1} in the Pb–PSMA complex cannot disappear completely, this is due to a larger hindrance caused by polystyrene moiety in the polymer chains of PSMA, which makes Pb–PSMA complex appear in nonstoichiometric form as illustrated in previous report [30]. In addition, two strong absorption bands at 1401 cm^{-1} and 1544 cm^{-1} are observed in the Pb–PSMA complex, which are assigned to the symmetric vibration absorption (ν_s, COO$^-$) and asymmetric vibration absorption (ν_{as}, COO$^-$) of the carboxylic groups, respectively [31,32].

The data in Table 2 show that the $\Delta\nu$ of Pb–PSMA is equal to 143 cm^{-1} ($\nu_{as} - \nu_s = 1544-1401$), which is far smaller than that ($\nu_{as} - \nu_s = 160$ cm^{-1} = 1563–1403) of K-PSMA, this indicates that the symmetry of the carboxylic groups in the Pb–PSMA complex is C$_{2\nu}$, the same as the free ions. This clearly proves that the carboxylic groups acting as a bidentate chelate coordinate to Pb(II) ions in the complex [33–35].

3.2. Adsorption for Pb(II) ion and corresponding influencing factor

3.2.1. Effect of solution pH on adsorption capacity

The effect of solution pH on the adsorption of Pb(II) at a constant temperature of 30 °C is presented in Fig. 4.

It can be seen that the chelating of the PSMA resin with Pb(II) is obviously dependent on the solution pH. At lower pH values, carboxylates (COO$^-$) in PSMA resin are in the form of protonation, which induces an electrostatic repulsion of Pb(II). Therefore, competition exists between protons and Pb(II) ion for adsorption sites and leads the adsorption capacity to decrease. The results indicate that Pb(II) ion can compete with hydrogen ions for adsorption by the carboxylate ions of PSMA [9]. At the pH value of 2.1, Pb(II) starts precipitating, the adsorption capacity increases with the increase of the solution pH values and reaches the maximum at the pH value of 7.8. At higher pH values (in the range of 7.8–8.2), the adsorption capability starts decreasing with the increase of the solution pH values. This phenomenon can be illustrated as the strong tendency that Pb(II) forms coordinated anion with carboxylate, which causes Pb–PSMA complex dissolving in water. On the contrary, above the pH value of 8.5, the adsorption capability increases with the increase of the solution pH values, this phenomenon can be illustrated as the formation of Pb(OH)$_2$, this is not favorable to selectively recovering Pb(II), so the solution pH was adjusted in the range of 2.1–8.2. The results indicate that the optimal solution pH for the adsorption of the PSMA resin to Pb(II) is 7.8 and the maximum adsorption capacity of that is 641.62 mg g^{-1} at the pH value of 7.8.

3.2.2. Effect of temperature on adsorption capacity

The effect of temperature on the adsorption capacity of the PSMA to Pb(II) at the solution pH value of 7.8 is shown in Fig. 5.

The adsorption capacity of PSMA to Pb(II) increases with the temperature increasing below 30 °C and reaches a peak value at 30 °C, then it decreases with the temperature increasing above 30 °C. This results indicated that the adsorption capacity of PSMA to Pb(II) ion is affected by the temperature, and the optimal temperature for Pb(II) adsorption by the PSMA is at 30 °C.

3.2.3. Effect of time on adsorption capacity (selectivity of lead recovery)

50 mL of 3.64×10^{-3} mol L^{-1} Pb(II) and Zn(II) ions mixed solution interacted with 0.0513 g of PSMA resin under stirring for 120 min at 30 °C, then the solution was centrifuged to get a supernatant. The effect of the adsorption time on the adsorption of PSMA to Pb(II) and Zn(II), monitored through determining the concentration variation of these two ions with time, are shown in Figs. 6 and 7, respectively. It can be seen that the adsorption processes of the PSMA resin for Pb(II) and Zn(II) are obviously different. The concentration of Pb(II) ion sharply decreases to 4.62×10^{-4} mol L^{-1} within about 2 min and then remains unchanged in the following time even to 120 min. So, the adsorption rate of PSMA for Pb(II) is extremely fast and the adsorption capacity can up to 641.62 mg g^{-1} calculated from formula (1). Different from Pb(II), the concentration of Zn(II) ion has nearly no any variation (keeps at 3.64×10^{-3} mol L^{-1}) within 80 min, but it quickly decreases to 6.67×10^{-4} mol L^{-1} in the range of 80–100th min and then keeps this value in the following time. It also needs to be noted that the adsorption equilibrium between the PSMA resin and Pb(II) is reached within 2 min, while that for Zn(II) is reached in the range of 80–100th min. This means that there is a adsorption hysteresis in the adsorption of the PSMA to Zn(II), and the hysteresis time is about 80 min, which provides a chance to selectively adsorb Pb(II) from the mixed solution of Pb(II) and Zn(II) ions. From results above, we know that the Pb(II) ions can be selectively separated from the mixed solution of Pb(II) and Zn(II) ions within 2 min.

Similar adsorption experiment was carried out in the aqueous solution containing Pb(II), Zn(II), Ca(II), and Mg(II) ions, and the relevant data are listed in Table 3. The detection limits of the atomic

Table 3
Adsorption capacity of PSMA resin for Pb(II) and Zn(II).

	Pb^{2+}	Zn^{2+}	Ca^{2+}	Mg^{2+}
L (µg mL^{-1})	2.0×10^{-2}	2.0×10^{-3}	1.0×10^{-2}	1.0×10^{-3}
q_i (mol L^{-1})	3.64×10^{-3}	3.64×10^{-3}	3.64×10^{-3}	3.64×10^{-3}
q_e (mol L^{-1})	4.62×10^{-4}	3.64×10^{-3}	3.64×10^{-3}	3.64×10^{-3}
Q (mg g^{-1})	641.62	$<1.95 \times 10^{-3}$	$<0.97 \times 10^{-2}$	$<0.97 \times 10^{-2}$

L: limit of detection.
C_i: initial concentration.
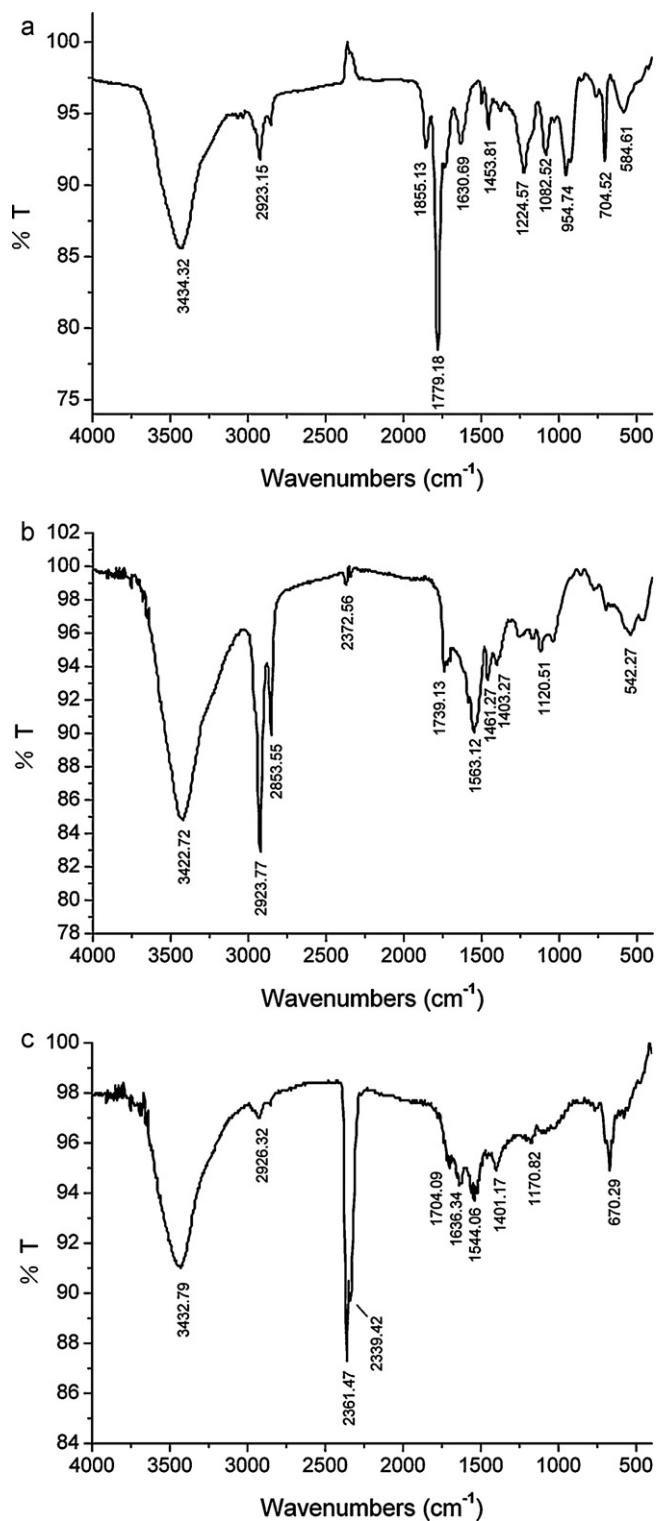C_e: equilibrium concentration.
Q: adsorption capacity.

Fig. 3. FTIR spectra of (a) PSMA resin, (b) K-PSMA and (c) Pb–PSMA complex (cm^{-1}).
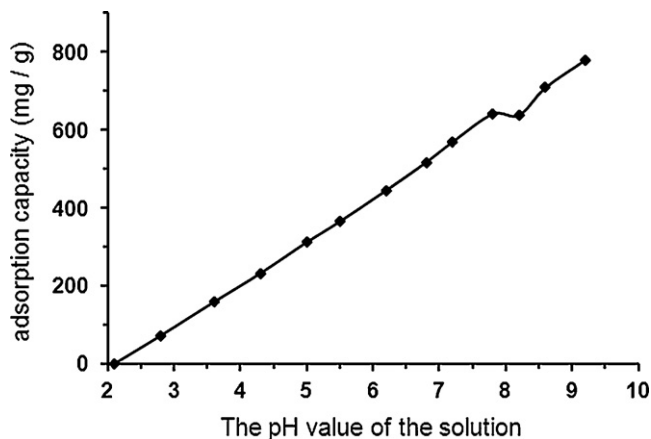

Fig. 4. Adsorption capacity of PSMA resin for Pb(II) vs. The pH value of the solution.
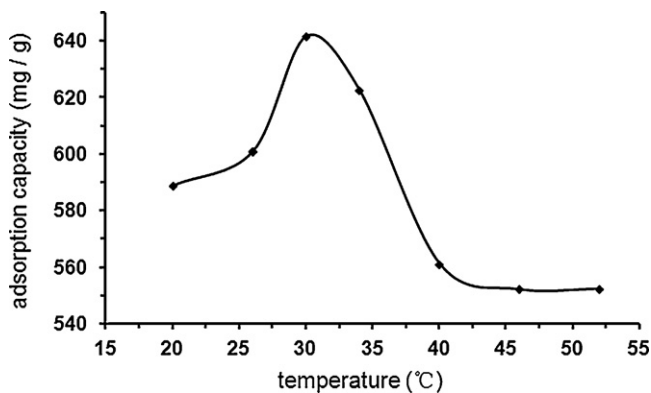

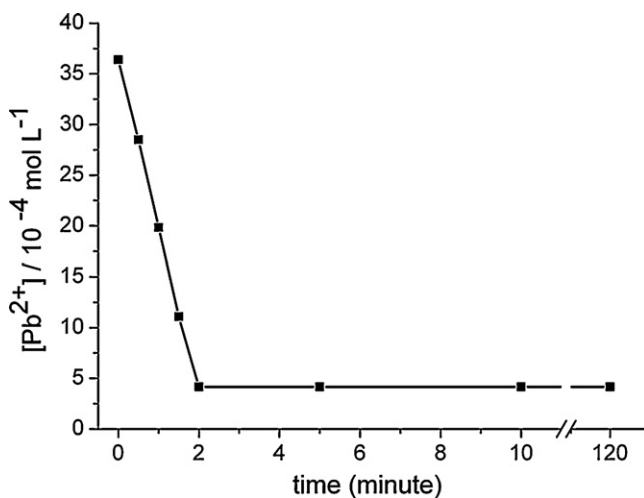**Fig. 5.** Adsorption capacity of PSMA resin for Pb(II) vs. Temperature of the solution.

Fig. 6. The concentration of Pb(II) in the solution vs. Adsorption time.

adsorption spectrometer used in our experiment are 0.02 μg mL^{-1} for Pb(II), 0.002 μg mL^{-1} for Zn(II), 0.01 μg mL^{-1} for Ca(II), and 0.001 μg mL^{-1} for Mg(II), respectively. Under our detection condition, we cannot detect the concentration variation of Zn(II), Ca(II) or Mg(II) ions before and after adsorption at any intervals within 80 min, so the concentration of them remain unchanged within 80 min. That means the adsorption capacities of the PSMA resin are <1.95 × 10^{-3} mg g^{-1} for Zn(II), <0.97 × 10^{-2} mg g^{-1} for Ca(II), and

<0.97 × 10^{-3} mg g^{-1} for Mg(II) within 80 min, respectively, calculated according to Eq. (3):

$$Q = \frac{L \times V}{1000\,W} \qquad (3)$$

where Q represents adsorption capacity (mg g^{-1}); L is the limit of detection (μg mL^{-1}); V is the volume of the solution used for adsorption (mL); and W is the weight of the resin (g). It needs to be noted that the PSMA resin only adsorbs Pb(II) ion and its adsorption capacity for Pb(II) is 641.62 mg g^{-1}, while the concentrations of
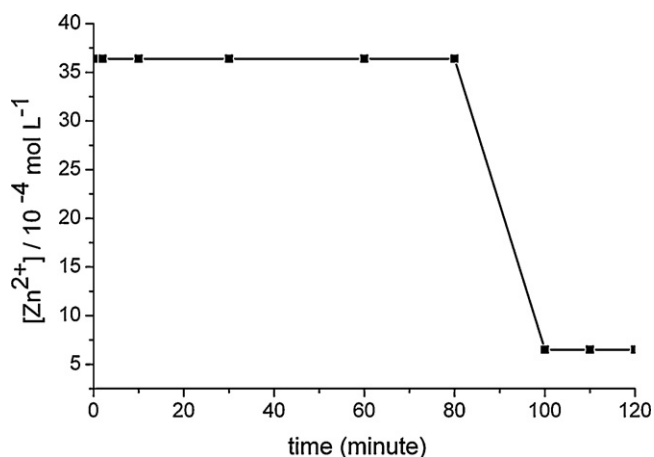
Fig. 7. The concentration of Zn(II) in the solution vs. Adsorption time.

Zn(II), Ca(II), and Mg(II) ions in the solution remain the same within 80 min. The result indicates that the PSMA resin has a higher selectivity to Pb(II) than that to Zn(II) and other light metals such as Ca(II) and Mg(II). Therefore, PSMA is a excellent adsorbent for selective recovery of Pb(II) from the mixed solutions.

3.3. Desorption of lead

As mentioned above, the higher adsorption selectivity of PSMA resin to Pb(II) can be exploited to recover Pb(II). The Pb–PSMA precipitates do not dissolve in water or typical organic solvents, but the Pb(II) can be easily desorbed by inorganic acid to release Pb(II) ion by which the PSMA resin would be regenerated. Under the inorganic acid condition ($6\,M\,H_2SO_4$), the solid Pb–PSMA complex changes into Pb(II) ion and the solid PSMA resin, which can be used repeatedly after transforming into K-PSMA.

4. Conclusions

The PSMA resin has rather strong coordination ability to Pb(II) to form Pb–PSMA complex which possesses three-dimensional network structure and can not be dissolved in water or typical organic solvents. Adsorption experiments reveal that the maximum adsorption capacity of PSMA resin to Pb(II) is $641.62\,mg\,g^{-1}$, while it does not adsorb Zn(II) and light metals (Ca(II) and Mg(II) ions) within 80 min, so the high adsorption selectivity of PSMA to Pb(II) ion can be used to separate Pb(II) from Zn(II) efficiently. The Pb(II) adsorbed on the PSMA resin can be desorbed easily by $6\,M\,H_2SO_4$, and the PSMA resin can be used repeatedly.

Acknowledgment

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