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Synthesis and characterization of a high selective mercury(II)-imprinted polymer using novel aminothiol monomer

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ABSTRACT

A novel aminothiol monomer was synthesized from' 2-mercaptoethylamine for complexing the Hg(II) ion. The synthesized complex monomer has been used for preparing the Hg(II)-imprinted polymer by radical copolymerization with methacrylic acid and ethylene glycol dimethacrylate and AIBN as the functional monomer, cross-linker and initiator respectively in the presence of a binary porogen of DMSO and toluene. The results of batch procedures showed that the Hg(II)-imprinted polymer has adsorption capacity of 28 mg g^{-1}. The distribution ratio (K_d) values of the Hg(II)-imprinted polymer increases for mercury with respect to both K_d values of CH_3Hg(I), Zn(II), Cd(II) and non-imprinted polymers. The relative selective factors k for Hg(II)/ CH_3Hg(I), Hg(II)/Cd(II) and Hg(II)/Zn(II) are 15.5, 12.5, and 20.6, which are greater than 1. The thermal stability has also been investigated by thermogravimetric analysis which shows that the polymerization process increases the thermal stability up to 125 °C.

1. Introduction

Molecular imprinting is a technology that creates recognition sites in a macromolecular matrix using a molecular template [1], in which the template molecule is usually the target analyte. Both the shape image of the target and alignment of the functional moieties to interact with the target are memorized in the macromolecular matrix after removing the target molecule [2]. Contrary to MIPs, ion-imprinted polymers (IIPs) are prepared using a target ion as an ion template during the polymerization of IIPs for recognizing and preconcentrating a specific ion [3]. One of the first studies on the synthesis of IIPs was reported by Nishide et al. [4–6] in 1970 s. They crosslinked the linear chain polymers (poly(4-vinyl pyridine)) with a bifunctional agent (dibromoalkane) in the presence of metal ions, such as Cu(II), Zn(II), Co(II), Ni(II), Hg(II) and Cd(II) as templates. Rao et al. [7,8] have recently published two reviews, describing IIPs and their special applications in selective recognition and solid phase extraction of inorganic ions.

Three steps are involved in ion-imprinting process: The complexation of metal ions to a polymerizable ligand, then the polymerization of complex monomer, and finally the removal of template produces the ion-imprinted polymer. The monomers are chosen according to their ability to interact with the functional groups of the template molecule. Since the affinity of ligand affects directly on selectivity for an ion template, the right selection of the functional monomer is important [9–14].There are some commercial monomers such as 4-vinyl pyridine, styrene, and methacrylic acid that have been used extensively in IIPs. These monomers are used by themselves or with another compound for complexing the ion template before or during the polymerization [15–17]. Liu et al. [18] have reported Hg(II) imprinted copolymers by copolymerizing mercury chloride, diazoaminobenzene (DAAB) and 4-vinylpyridine (VP) using ethylene glycol dimethacrylate (EGDMA) as a crosslinker in the presence of 2,2-azobisisobutyronitrile (AIBN) as an initiator. Their Hg(II) imprinted copolymers have high adsorption capacity and good selectivity for Hg(II) compared with non-imprinted copolymers but their thermal stabilities have not been reported. 5,7-dichloroquinoline-8-ol is another compound that has been used for complexing many ion templates in IIPs [19–21]. Although these compounds increase the selectivity in ion-imprinted polymers, as they do not involve the polymerizable group, they cannot be copolymerized with the functional monomers and will be physically trapped into the bulk of polymer after removing the template that can cause thermal instabilities and they will decompose before thermal decomposition of polymer.

Since heavy metals in general and mercury in particular show very great affinity to sulfur, the functional monomers containing the sulfhydryl group can produce good stable complex with mercury. On the other hand, an amine as a nucleophilic element can react with an activated acryl donor such as methacryloyl chloride and produce the acryl-type monomers. The aminothiols have both of amine and sulfhydryl groups therefore they are good candidates for producing thiol-functional monomers for imprinting the mercury ions.

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2-Mercaptoethylamine (MEA) or Cysteamine is the simplest stable aminothiol without additional reactive functional group that is able to form the stable complex with some metal ions [22–24]. In the present work, this compound has been used for synthesizing N-Methacryloyl-2-mercaptoethylamine (MMEA) then complexing the mercury ion. The produced complex monomer copolymerized with methacrylic acid and ethylene glycol dimethacrylate as a functional monomer and crosslinker respectively. After removing the mercury ions the obtained Hg(II)-imprinted polymer shows high selectivity to Hg(II) ions in comparison to non-imprinted polymers and in competition with other metal ions such as Cd(II), Zn(II) and CH_3HgCl.

2. Experimental

2.1. Apparatus

The concentrations were determined by a Perkin–Elmer ELAN DRC II ICP-MS (Sciex, Canada) and a non-dispersive atomic fluorescence spectrometer (Beijing Rayleigh Analytical Instrument Corporation, China) equipped with a high performance Hg hollow cathode lamp (253.7 nm, Beijing Institute of Vacuum Electronics Research, China).A Nicolet Avatar 360 FTIR and a Laser Raman Renishaw (633 nm, 50 × , 6 mw, 30 s) were used for IR and Raman spectra respectively. The pH measurements of all solutions were performed using a Mettler Toledo Delta 320 pH meter (Mettler-Toledo instruments Co. Ltd., Shanghai, China). A NETZSCH TG 209 F1 was used for thermogravimetric analysis.

2.2. Reagents

All used solutions were prepared with ultrapure water (UPW) produced by a Milli-Q system (Millipore Filter Co., Bedford, MA, USA). All reagents used in this work were of the highest purity available and at least of analytical grade. Hg(II) Stock solution (1007 mgL^{-1}, Sigma) was used for preparation of mercury standard solutions. Methacryloyl chloride was from Zhengzhou Sigma chemical Co., Ltd. (Henan, China). Methacrylic acid was from Shantou XiLong Chemical Factory (Guandong, China). Azobisisobutyronitrile (AIBN 97%, recrystallized before use), Dimethyl Sulfoxide (DMSO), toluene, tetrahydrofuran (THF), Hydrochloric Acid, thiourea, $CdCl_2 \cdot 2.5H_2O$, $ZnCl_2$, triethyl amine, anhydrous diethylether, $NaHCO_3$, anhydrous sodium sulfate and mercury(II) chloride were from Sinopharm Chemical Reagent (Shanghai, China). EGDMA (97%) and 2-mercaptoethylamine were purchased from TCI (Tokyo, Japan), CH_3HgCl (> 95%) was obtained from Alfa Aesar (Ward Hill, MA, USA).

The pH was adjusted with the following buffer solutions: $NaCH_3COO/CH_3COOH$ for pH 4.0–6.0; Na_2HPO_4/NaH_2PO_4 for pH 7.0–7.5; $Na_2B_4O_7/HCl$ for pH 8.0–9.0

2.3. Synthesis of N-methacryloyl-2-mercaptoethylamine monomer (MMEA)

MMEA was synthesized via the Schotten–Baumann reaction [25] as shown in Fig. 1a. 2-mercaptoethylamine (MEA) (10 mmol) dissolved in THF (50 mL) in a three-necked flask, and then methacryloyl chloride (12 mmol) was added dropwise into the flask while triethylamine (TEA) (12 mmol) simultaneously was added as a catalyst under nitrogen atmosphere at 0 °C (ice-water bath), and then the reaction mixture was allowed to react for 5 h at room temperature. The resulting precipitate was filtered off, and the THF was evaporated using a rotavapor device. The remaining yellow liquid was dissolved in anhydrous diethylether and then washed with 0.5 M $NaHCO_3$ solution and finally was dried using anhydrous sodium sulfate to yield MMEA after the diethylether had evaporated completely.

2.4. Synthesis of Hg(II)–N-methacryloyl-2-mercaptoethylamine complex monomer (Hg(II)-MMEA)

MMEA monomer (4 mmol) was dissolved in ethanol, and was added to an $NH_3 \cdot H_2O–NH_4Cl$ buffer solution (pH 9) under nitrogen atmosphere, then $HgCl_2$ (2 mmol) was added slowly and allowed to further reaction for 2 h with stirring. Then the formed complex (Hg(II)-MMEA) was filtered and washed with water, ethanol and acetone then dried in a hydrate silica gel vacuum desiccator (Fig. 1b).

2.5. Preparation of Hg(II) Imprinted and non-imprinted copolymers

Hg(II)-MMEA complex monomer (0.032 mmole), methacrylic acid (0.118 mmol), EGDMA (0.786 mmole) and AIBN (4 mg) were dissolved in the binary porogen mixture (DMSO 170 μL, Toluene 80 μL) and homogenized ultrasonically for 10 min. The prepolymerization solution was deoxygenized by a nitrogen stream for 5 min then sealed and placed in an oven at 70 °C for 24 h. Afterwards, the obtained polymer was separated and washed thoroughly and frequently with ethanol and water to remove any unreacted monomer, initiator and solvent then dried in a hot air oven at 60 °C for 2 h and grounded in a mortar and sieved to acquire the powdered particles and then stirred with the solution of HCl 2 M–thiourea 5% at 60 °C for 48 h for complete removal of Hg(II) from copolymer. Hg(II)-imprinted polymer was washed with ultra-pure water and dried again in an oven at 60 °C.

The non-imprinted polymer particles were prepared in a similar way but without the imprinted ion, Hg(II), i.e. the MMEA monomer was used in prepolymer solution instead of Hg(II)-MMEA complex monomer for obtaining the non-imprinted polymer.

2.6. Batch procedures

The adsorption behavior of Hg^{2+} on 25 mg Hg-imprinted copolymer in 25 mL solution was tested by stirring 25 mg IIP in 25 ml solution buffered at pH 7.5 except for pH effect test at pH 4–9 containing Hg(II) 0.1 mgL^{-1} except for adsorption capacity measurement with the metal ion concentration of 20 to 100 mgL^{-1}. At prefixed time, final concentrations were determined by AFS and ICPMS. For the selectivity measurements 25 mL solution buffered at pH 7.5 containing 5 μM of Hg^{2+}, Cd^{2+}, Zn^{2+} and CH_3Hg^+ ions was stirred magnetically at 500 rpm with 25 mg IIP or NIP. The adsorption capacities, the distribution ratios, the selectivity factors of Hg(II) with respect to Zn(II), Cd(II) and CH_3HgCl and the relative selectivity factor were calculated using the following equations:

$$Q = (C_i - C_f)V/m \tag{1}$$

$$K_d = [(C_i - C_f)/C_f][V/m] \tag{2}$$

$$k = K_{Hg}/K_M \tag{3}$$

$$k' = k_{imprint}/k_{non-imprint} \tag{4}$$

where Q represents the adsorption capacity (mg g^{-1}), C_i and C_f are the initial and equilibrium concentrations of Hg(II) (mgL^{-1}), m is the mass of copolymers (g) and V is the volume of metal ion solution [26]. K_d is distribution factor and K_{Hg} and K_M represent the distribution ratios of Hg(II) and Cd(II), Zn(II) or CH_3HgCl. k and k are selectivity factor and relative selectivity factor respectively.

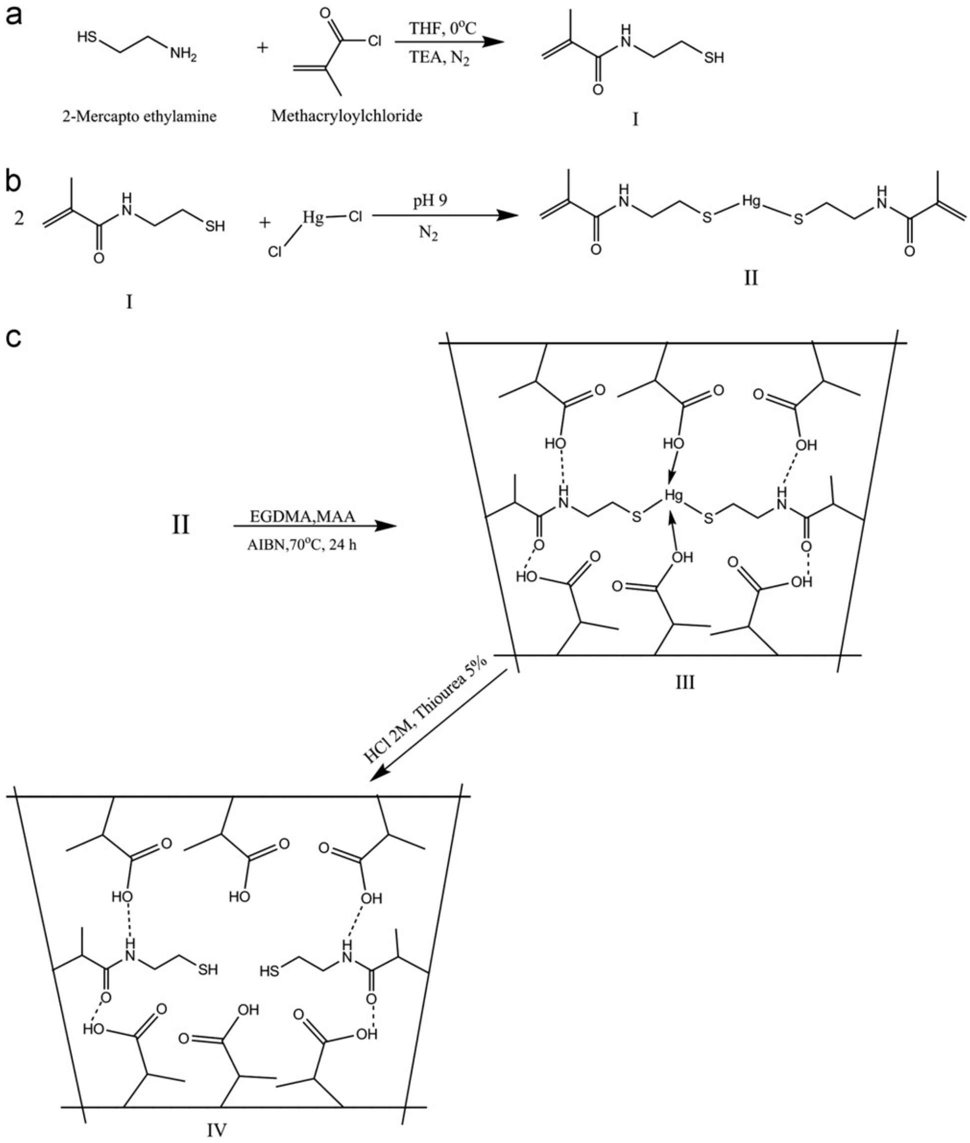

Fig. 1. (a) Synthesis of MMEA monomer (I), (b) synthesis of Hg(II)-MMEA complex monomer (II) and (c) synthesis of Hg(II)-imprinted polymer (IV).

3. Results and discussion

3.1. Characterization of Hg(II) –N-Methacryloyl-2-Mercaptoethylamine Complex

FTIR spectrum of Hg(II)-MMEA (Table 1, Suppl. A) has the characteristic stretching vibration amide I and amide II absorption bands at 1660 and 1533 cm^{-1} that confirm the entering of methacrylate functional group to mercaptoethylamine molecule and formation of a secondary amide. The S–H stretching vibration band at 2557 cm^{-1} is absent from the spectrum of Hg(II)-MMEA complex monomer (Table 1, Suppl. B) which confirms the forming of the complex. Hoffmann et al. have studied the FTIR and FT-Raman spectroscopy for Hg(SR) compounds in the solid state [27]. Their results show that the frequency window of the Hg–S stretching vibrations is approximately 180–500 cm^{-1}. Therefore, Raman spectra in this range were employed to reconfirm the formation of the complex (Fig. 2). Raman spectrum presents two bands at 273 and 410 cm^{-1} that are assigned to the asymmetric vibration of δ(C–S–Hg) and the bending mode of υ(Hg–S) respectively.

3.2. Characterization of Hg(II)-IIP copolymers

3.2.1. Raman spectra

The mercury ion was removed by stirring the imprinted polymer particles with acidic thiourea to obtain the leached IIP particles. The unleached and leached Hg(II)-imprinted polymers were characterized by Raman spectroscopy. The Raman spectrum of leached polymer (Fig. 3a) revealed a band at 2557 cm^{-1} assigned to S–H stretching band that was absent in the spectrum of unleached polymer (Fig. 3b). So, it is clear that the imprinting process has taken place successfully.

3.2.2. Thermal stability

The polymerization effect on thermal stability was also investigated by thermogravimetric analysis of Hg(II)-MMEA complex monomer (Fig. 4a) and Hg(II)-imprinted polymer (Fig. 4b). In the measurement process, the samples were heated from 25 °C to 600 °C with a heating rate of 10° K min^{-1} in N_2 atmosphere. The results showed that Hg(II)-MMEA complex decomposes at 175 °C

Table 1
Some characteristic infrared bands.

N-Methacryloyl-2-Mercaptoethyl amine (cm^{-1})	Hg(II)-N-Methacryloyl-2-Mercaptoethyl amine Complex (cm^{-1})	Assignment
3330 s,br	3384 s,br	OH and NH stretch
2973 m	2970 m	CH_2 asym
2930 m	2928 m	CH_2 sym.
2557 w	–	S–H stretch.
1656 s	1653 s	C=O stretch. (Amide I)
1618 w	1616 w	C=C stretch
1533 s	1529 s	N–H bend. (Amide II)
1451 m	1452 m	CH_2 bend.

s=strong, *m*=medium, *w*=weak, *br*=broad.

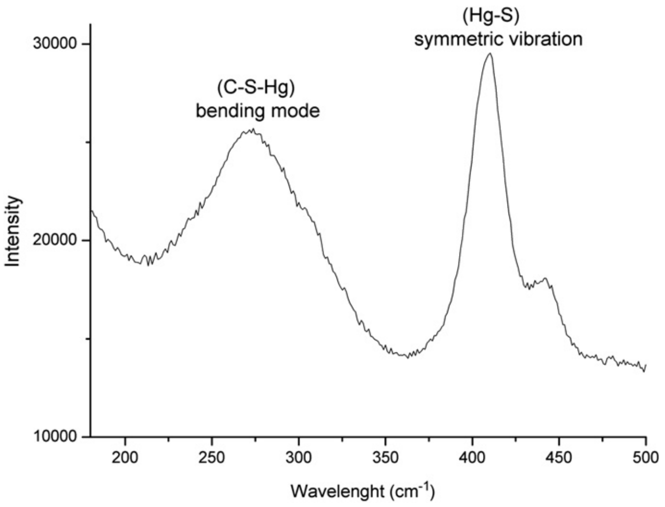

Fig. 2. Raman spectrum of Hg(II)-MMEA(Raman).

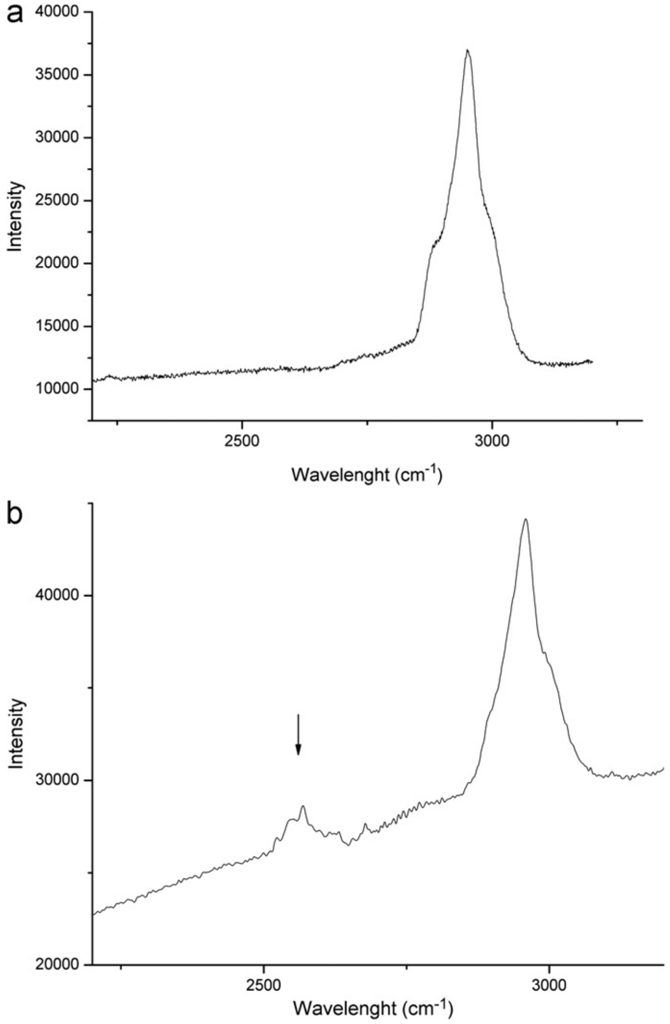

Fig. 3. (a) Raman spectrum of unleached Hg(II)-imprinted polymer. (b) Raman spectrum of leached Hg(II)-imprinted polymer.

while the Hg(II)-imprinted started thermal decomposition at about 300 °C and ended at 480 °C which shows that the polymerization process increases the thermal stability up to 125 °C. Also, remaining mercury in unleached imprinted polymer at 300 °C suggested that no thermal desorption of mercury ion takes place before the decomposition of polymer.

3.3. *Adsorption behaviors*

3.3.1. *Adsorption capacity of imprinted copolymer for Hg(II)*

Fig. 5 shows the effects of initial concentration of Hg(II) ions on the adsorption capacity of the Hg-imprinted polymer at pH 7.5. The amount of Hg(II) ions adsorbed per unit mass of the polymer (i.e., adsorption capacity) increases with increasing the initial concentration of Hg(II) ions. The maximum adsorption, which represents saturation of active points on the polymer by Hg(II) ions, was 28 mg Hg(II) per g of polymer (corresponding to a 88 mgL^{-1} Hg(II) ion initial concentration).

3.3.2. *Effect of pH on the absorption behaviors*

The pH values play an effective role on complexing reaction between metal ions and ligands. In order to evaluate the effect of pH on the adsorption of the Hg(II), the pH values of sample solutions were adjusted in the range of 4–9. Fig. 6 shows that the adsorption of Hg(II) increases with the increase of pH values from 4 to 7. The adsorption is low in acidic conditions owing to the protonation of Hg(II) imprinted copolymers and diminishes after pH 8 owing to the formation of metal hydroxide. The adsorption capacity is near the maximum between pH 7.0–8.0. So, pH 7.5 was chosen as an optimum pH for all adsorption experiments.

3.3.3. *Kinetics*

The kinetics of the Hg(II) adsorption on the polymers were investigated in a batch system. The Hg(II) concentration in suspensions was analyzed at times of 5, 10, 20, 30, 40, 50, and 120 min from the start. Fig. 7 shows the time dependence of the adsorption capacity of Hg^{2+} ions on the imprinted polymer. It can be seen that the adsorption of Hg^{2+} ions increases with the time during the first 50 min and then the curve levels off as equilibrium is reached most probably due to complexation and geometric shape affinity (or memory) between Hg^{2+} ions and Hg^{2+} cavities in the polymer structure. It is well known that removal of the template from the polymeric matrix leaves cavities of complementary size, shape and chemical functionality to the mercury ions.

3.3.4. *Selectivity*

Competitive adsorption of Hg(II), Cd(II), Zn(II) and $CH_3Hg(I)$ from their mixtures was also investigated in a batch system. After adsorption equilibrium of 50 min, the concentration of Cd(II) and Zn(II) ions in the remaining solution was measured by ICP-MS and

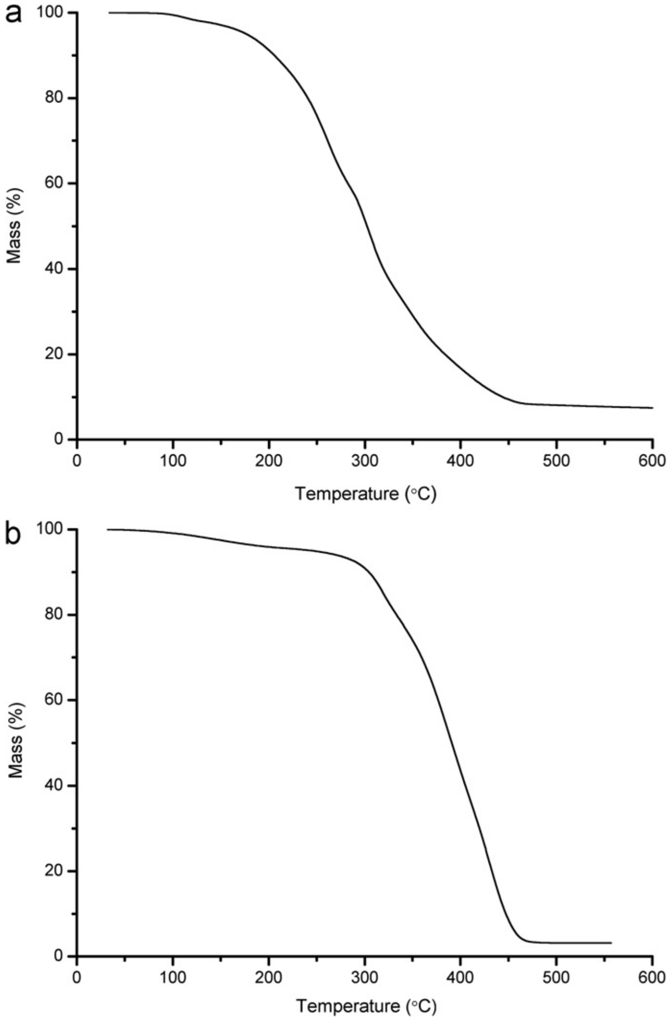

Fig. 4. (a) Thermogravimetry curve of Hg(II)-MMEA complex monomer. (b) Thermogravimetry curve of Hg(II)-imprinted polymer.

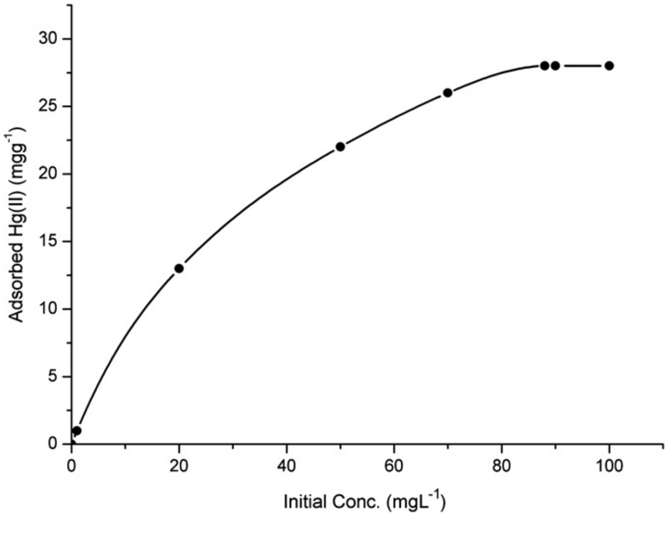

Fig. 5. Adsorption capacity of Hg(II) ions on the imprinted polymer; other conditions, 25 mg of Hg(II) imprinted copolymers; *V* 25 ml; pH 7.5; adsorption time 24 h; *T* 25 °C.

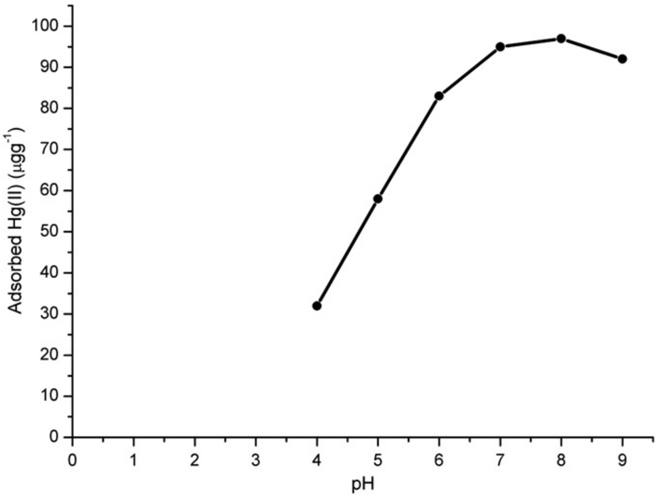

Fig. 6. Effects of pH on Hg(II) adsorption; other conditions, 25 mg of Hg(II) imprinted copolymer; Hg(II) initial concentration, 0.1 mgL^{-1}; *V* 25 ml; *T* 25 °C.

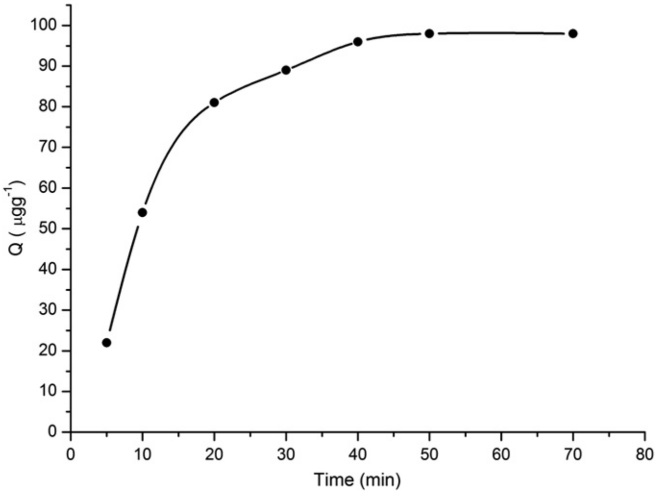

Fig. 7. Adsorption rates of Hg(II) ions on the imprinted polymer; other conditions, 25 mg of Hg(II) imprinted copolymer; Hg(II) initial concentration, 0.1 mgL^{-1}; *V* 25 ml; *T* 25 °C.

Table 2
The selectivity parameters.

Metal ions	Hg(II)-imprinted		Non-imprinted		k'
	K_d (mL g^{-1})	k	K_d (mL g^{-1})	k	
Hg(II)	19,896		5230		
Cd(II)	307	64.8	1007	5.2	12.5
Zn(II)	69	288.3	374	14	20.6
CH_3HgCl	217	91.7	887	5.9	15.5

the concentration of Hg(II) and CH_3HgCl was measured by HPLC-AFS. The results of competitive ion binding batch tests are summarized in Table 2. A comparison of the selectivity factor of Hg(II)-imprinted copolymer with the selectivity factor of non-imprinted copolymer showed that the imprinted matrix for Hg(II)/Cd(II), Hg(II)/Zn(II) and Hg(II)/CH_3HgCl was 12.5, 20.6 and 15.5 times greater than the non-imprinted matrix, respectively. These results confirm the size effect of imprinted cavities on selectivity. Although Zn^{2+} and Cd^{2+} have similar electrical charges with Hg^{2+}, since they are smaller than Hg^{2+} ($Cd^{2+}=97$ pm, $Zn^{2+}=74$ pm and $Hg^{2+}=110$ pm), they pass through the cavities without effective interactions; conversely, CH_3Hg^+ ion is larger and cannot enter the cavities so, it is slightly adsorbed on the polymer.

Table 3
Determination of Hg(II) in water samples.

Samples	Hg(II) added(μgL^{-1})	Hg(II) founded (μgL^{-1})[a]	Recovery (%)
Tap water	0.00	0.23 ± 0.012	–
	0.50	0.69 ± 0.021	94.5
	1.00	1.21 ± 0.045	98.3
	2.00	2.30 ± 0.113	103.1
	5.00	5.53 ± 0.251	105.5
Sea water	0.00	0.09 ± 0.008	–
	0.50	0.55 ± 0.032	93.2
	1.00	1.05 ± 0.053	96.3
	2.00	2.07 ± 0.089	99.0
	5.00	5.31 ± 0.324	104.3

[a] Average of three replicate determinations ± S.D.

3.3.5. *Analytical applications*

The prepared Hg(II)-imprinted polymer was used for the determination of Hg(II) in the tap water and Xiamen coastal sea water samples, China. The standard addition method was applied to check the selectivity of the ion-imprinted sorbent for Hg(II) against matrix elements. The results are listed in Table 3. When 200 ml of the water samples spiked with Hg(II) standards of different concentrations (0.5, 1, 2, and 5 μgL^{-1}) with a pH of 7.5 were treated by the imprinted sorbents and desorbed by the acidic solution of thiourea, the recoveries were in the range of 93–105%. These results demonstrate that the interference species in the matrix were eliminated satisfactorily after extraction and preconcentration procedures and indicate the suitability of the Hg(II)-imprinted polymer for selective extraction and determination of trace Hg(II) in environmental samples.

4. Conclusions

In this work, an Hg(II)-imprinted polymer has been prepared by radical copolymerization of a novel synthesized Hg(II)-aminothiol monomer with methacrylic acid using ethylene glycol dimethacrylate and AIBN as a cross-linker and initiator in the presence of a binary porogen. The imprinted polymer has a good selectivity towards Hg(II) over Cd(II), Zn(II) and CH_3HgCl and in comparison with non-imprinted polymer. The thermogravimetric analysis of the synthesized complex monomer shows good thermal stability up to 300 °C and also shows that the thermal stability is not enough for releasing the Hg(II) ion before the decomposition of polymer. In addition, the prepared Hg(II)-imprinted polymer exhibits high selectivity for Hg(II) present in environmental samples matrix.

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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at http://dx.doi.org/10.1016/j.talanta.2012.09.023.

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