Syntheses and Adsorption Properties for Hg²⁺ of Chelating Resin of Crosslinked Polystyrene-Supported 2,5-Dimercapto-1,3,4-thiodiazole

Rongjun Qu,^{1,2} Chunhua Wang,² Changmei Sun,² Chunnuan Ji,² Guoxiang Cheng,¹ Xiaoqin Wang,² Gang Xu²

¹School of Materials Science & Engineering, Tianjin University, Tianjin 370002, People's Republic of China ²School of Chemistry and Materials Science, Yantai Normal University, Yantai 264025, People's Republic of China

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ABSTRACT: A novel chelating resin with functional group containing S and N atoms was prepared using chloromethylated polystyrene and 2,5-dimercapto-1,3,4-thiodiazole (also called bismuththiol I, BMT) as materials. Its structure was characterized by infrared spectra and elementary analysis. The results showed that the content of the functional group was 2.07 mmol BMT g⁻¹ resin, 47% of which were in the form of monosubstitution (PS-BMT-1) and 53% in the form of double substitution (PS-BMT-2). The adsorption for mercury ion was investigated. The adsorption dynamics showed that the adsorption was controlled by liquid film diffusion. Increasing the temperature was beneficial to adsorption. The Langmuir model was much better than the Freundlich model to

tional describe the isothermal process. The adsorption activation energy (E_a), ΔG , ΔH , and ΔS values calculated were 18.56 kJ·mol⁻¹, -5.99 kJ·mol⁻¹, 16.38 kJ·mol⁻¹, and 37.36, prials. J·mol⁻¹·K⁻¹, respectively. The chelating resin could be easily a and regenerated by 2% thiourea in 0.1 mol·L⁻¹ HCl with higher effectiveness. Five adsorption–desorption cycles demonstrated that this resin was suitable for repeated use without considerable change in adsorption capacity. © 2004 Wiley Periodicals, Inc. J Appl Polym Sci 92: 1646–1652, 2004

> **Key words:** crosslinked polystyrene-supported 2,5-dimercapto-1,3,4-thiadiazole; chelating resin containing S and N atom; adsorption; mercuric ion

INTRODUCTION

Heavy metals are toxic to aquatic flora and fauna even in relatively low concentrations.¹ Some of these are capable of being assimilated, stored, and concentrated by organisms. Mercury is a commonly occurring toxic metal in natural ecosystems. It is used in a wide variety of industries such as electrical, paints, fungicides, chlor-alkali, paper and pulp, and pharmaceutical.¹ Of these, the color-alkali industry is the largest consumer of mercury and the single largest source of mercury pollution.

Mercury and its related compounds are toxic when ingested in living organisms. A special characteristic of mercury is its strong attraction to biological tissues and in general the slow elimination from the biological system. The health hazards due to the toxic effect of mercury at Minamata, Japan, and Iraq are very well known.² Although it is desirable to have zero mercury level in effluents, it is practically impossible to accomplish this. The world health organization recommends a maximum uptake of 0.3 mg·week⁻¹ and 1 ug·L⁻¹ as the maximum acceptable concentration in drinking water.³ A number of approaches have been suggested for the removal of mercury from aqueous solutions.^{4,5} The techniques include reduction, precipitation, ion exchange, reverse osmosis, adsorption, coagulation, etc. Among these, selective adsorption utilizing biological materials, mineral oxides, carbon, or polymer resins has generated much interest among researchers and practitioners of environmental engineering and science. Activated carbon,^{6–9} rubber tire,^{10–13} starch xanthate,¹⁴ and chitosan beads¹⁵ are found to be very effective for the removal of mercury.

Because of the chelating natures with metal ions, chelating resins are widely used in the preconcentration, separation, recovery, and hydrometallurgy of metals. Many efforts have been made to synthesize the chelating resins to remove mercury.^{16–25} There are two nitrogen atoms and three sulfur atoms in the structure of bismuththiol I ($C_2H_2N_2S_3$, BMT), which can chelate metal ions such as bismuth, copper, and lead. It can be expected that the chelating resin containing the functional group of BMT will have excellent adsorption properties toward metal ions. So we synthesized a

Correspondence to: R. Qu (qurongjun@sohu.com).

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Scheme 1

novel chelating resin containing BMT groups and studied the adsorption property for mercury ion.

EXPERIMENTAL

Methods

Commercial macroporous polychloromethylstyreneco-divinylbenzene beads (chlorobeads, PS), with degree of crosslinking 10% DVB, chlorine content 19.85%, BET surface area 40.0891 \pm 0.1364 m²/g (ASAP-2000 Surface Area Analyzer, USA, analysis gas N₂), were purchased from the Chemical Factory of Nankai University of China. Micromeritics. Before use PS-Cl was further purified by extraction to remove surface impurities in a Soxhlet extraction apparatus for reflux-extraction in 95% ethanol for 10 h. BMT was purchased from Aldrich Chemical Co. Infrared spectra were recorded on a Nicolet MAGNA-IR 550 (series II) spectrophotometer. Test conditions were as follows: potassium bromide pellets, scanning 32 times, resolution 4 cm⁻¹. The content of chlorine was measured according to ref.²⁶ The content of S and N elementary analyses was carried out by the central laboratory of the Elemental Research Institute of Nankai University. The concentration of mercury ions was measured on a GBC-932 atomic absorption spectrophotometer (AAS) made in Australia according to the cold AAS method. Test conditions were as follows: wavelength 253.7 nm; slit 0.7 nm; lamp current 3.0 mÅ; standard substance HgCl₂; reductant, potassium borohydride.

Synthesis of polystyrene-supported BMT resin (PS-BMT)(typical reaction)

The synthesis route of PS-BMTwas described as follows (Scheme 1).

A total of 1.0 g of PS-Cl, 1.628 g of BMT, 4.0 g K_2CO_3 powder, and 20 mL of acetone was placed in a oneneck flask. With magnetic stirring, the mixture was refluxed for 8 h. The solvent acetone was distilled, and then 100 mL of distilled water was added to dissolve the salts. The polymeric product was filtered and washed with distilled water, 2% solution of KOH, 1% hydrochloric acid, and finally with distilled water. The product was transferred to a Soxhlet extraction apparatus for reflux-extraction in 95% ethanol for 10 h and then dried under vacuum at 50°C over 48 h. Elemental analysis (%) was as follows: Cl, 4.84; S, 19.51; N, 5.80. BET surface area was determined to be 21.4859 \pm 0.1399 m²/g.

Adsorption kinetics

Batch tests were performed to determine adsorption kinetics. A typical procedure is as follows: Pyrex glass tubes (50 mL) were prepared with the desired amounts of reagent solution (10 mL) and placed in a thermostat-cum-shaking assembly. When the desired temperature was reached, a known amount of resin (0.05 g) was added into each tube, and the mixed solutions were mechanically shaken. At predecided intervals of time, the solutions in the specified tubes were separated from the adsorbent and the concentration of Hg²⁺ was determined by means of AAS. The adsorption amounts were calculated according to eq. (1):

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

where *Q* is the adsorption amount (mmol·g⁻¹); C_0 and *C* are the initial concentration and the concentration of metal ion in solution when the contact time is *t* (mmol·ml⁻¹); *V* is the volume (ml); and *W* is the weight of the chelating resin (in grams).

Isothermal adsorption

The isothermal adsorption was investigated also by batch studies. A typical procedure empolys a series of 50-mL test tubes. Each test tube was filled with 10 mL of metal ion solution of varying concentrations and adjusted to pH 2.0 and desired temperature. A known amount of resin (about 0.05 g) was added into each test tube and agitated intermittently for the desired time periods, up to a maximum of about 12 h. The



Scheme 2

TABLE I						
The Elemental	Analysis	Results	of	PS-BMT ^a		

Elemental			Reactio	n time/ł	ı	
analysis (%)	1	3	7	10	12	14
CI	10.82	7.54	5.66	4.84	4.90	4.87
S			_	19.51	19.52	19.60
Ν	—	—	—	5.80	5.75	5.80

 a —, no data analyzed. The standard deviations for the data are 0.04–0.08, 0.04–0.09, and 0.04–0.09 for content of Cl, S, and N, respectively.

adsorption capacities were calculated also using eq. (1), where *C* is the equilibrium concentration of metal ion in solution.

The regeneration of resin

To assess the reusability of PS-BMT resin, consecutive adsorption–desorption cycles were repeated five times using the same resin. The solution of 2% thiourea in 0.1 mol·L⁻¹ HCl was employed as the desorption medium. The Hg²⁺-loaded PS-BMT resin was placed in this medium, and then the mixture was stirred for 1 h at room temperature (a primary experiment showed that the Hg²⁺ ions adsorbed on resin were almost desorbed completely when the contact time of Hg²⁺-loaded PS-BMT resin with this medium was about 40 min). The final concentration of Hg²⁺ in the aqueous phase was determined using AAS. The desorption ratio was calculated from the amount of Hg²⁺ adsorbed on the resin and the final concentration of Hg²⁺ in the desorption medium.



RESULTS AND DISCUSSION

Synthesis of PS-BMT resin

Some attempts were made to synthesize the target resin in the presence of base (KOH or NaOH) using quaternary ammonium salts $[(n-Bu)_4^+Br^-]$ as phase transfer catalysts and benzene/water as solvents, but the experimental results showed that no desired product was obtained. This was probably because BMT could form double-negative ion **A** (see Scheme 2) in basic solution and then form a double-ion pair (**B**) with a positive ion of the phase transfer catalyst. The hydrophilicity of **B** made it too difficult to enter the hydrophobic organic phase (here benzene) and further enter the hydrophobic polymeric matrix.

The reaction between PS-Cl and BMT obeyed the $S_N 2$ mechanism. Theoretically, the nonprotonic polar solvent was beneficial to the $S_N 2$ reaction. So acetone was selected as solvent to synthesize the PS-BMT. As a nonprotonic polar solvent, acetone can swell PS-Cl thoroughly, which would make the double-negative ion of BMT diffuse easily into the PS-Cl matrix and reach the active sites.



Figure 1 FTIR spectra of BMT, PS-Cl, and PS-BMT.





The optimum reaction time was determined by monitoring the changes of contents of chlorine or sulfur (or nitrogen) with the reaction time. The data in Table I show that the contents of Cl and S (or N) did not increase significantly when the reaction time was over 10 h, so 10 h was regarded as the optimum reaction time for synthesizing PS-BMT.

According to the content of chlorine and sulfur (or nitrogen) measured by elemental analysis, it can be calculated that only approximately 67% of chlorine was converted into functional group BMT. The content of functional group BMT was about 2.03 $mmol \cdot g^{-1}$ (2.07 $mmol \cdot g^{-1}$ according to N content), while 47% of BMT was in the form of monosubstitution (PS-BMT-1) and 53% was in the form of doublesubstitution (PS-BMT-2). A possible reason for this was that when BMT entered the matrix of PS-Cl, it reacted first with C-Cl to form structure PS-BMT-1 and further with C-Cl to form structure PS-BMT-2. The existence of crosslinked structure PS-BMT-2 affected seriously the diffusion of other double-negative ions of BMT and their arrival to the inner active sites. So in PS-BMT there was still part of the residual chlorine, which didn't participate in the reaction with BMT. The elemental analysis also showed that the molar ratio between S and N in resin PS-BMT was near 3:2, which was basically coincident with the ratio in the BMT monomer, indicating that the whole five-membered heterocycle of BMT was not destroyed during the period of substitute reaction.

As a conjugate system, BMT had three kinds of sympathetic structures designated (1), (2), and (3) described in Scheme 3. The absorption peak at 3432 cm⁻¹ in IR



From FTIR of PS-BMT in Figure 1, it can be noted that the characteristic peak of C-Cl at 675 cm⁻¹ weakened apparently after the reaction of PS-Cl with BMT, and a series of new peaks involved in BMT appeared at 3448 and 1381 cm⁻¹, indicating that the BMT functional group was introduced successfully into the polymeric matrix. Among these new peaks, 3448 cm⁻¹ was assigned the absorption of *N*-H in PS-BMT-1' (in Scheme 4) and broadening of the peak near 1611 cm⁻¹ showed the existence of $\nu_{\rm C} = N$ in the conjugate system, which demonstrated the presence of PS-BMT-1.

Adsorption kinetics

The adsorption kinetics at different temperatures is shown in Figure 2. The contact time and other conditions were selected on the basis of preliminary experimental results in Figure 2 that demonstrated that the equilibrium was established in 10 h. Equilibration for longer times, between 10 and 13 h, gave practically the same uptake. Therefore, the contact period was 12 h in all equilibrium tests.

It can be seen from Figure 2 that temperature had a significant effect on the adsorption capacities, that is, the adsorption capacities increased with increasing temperature. A possible explanation for the phenomenon above was that the resin was swollen more com-



Figure 2 The adsorption kinetics of PS-BMT for Hg^{2+} .



Figure 3 The relationship between $-\ln(1-Q/Q_0)$ and *t*.

TABLE IIThe Constants of Adsorption k and Correlation Coefficient R2					
Temperature (°C)	Constant rate of adsorption k (s ⁻¹)	Correlate coefficients R^2			
27	9.40×10^{-5}	0.9638			
35	$1.14 imes 10^{-4}$	0.9803			
45	1.43×10^{-4}	0.9386			

pletely at higher temperatures, which made mercury ions diffuse more easily into the resin.

Figure 3 was obtained by treating the data in Figure 2 with the G. E. Boyd equation, $-\ln(1-Q/Q_0) = kt$, where Q is the adsorption amount when the contact time is t; Q_0 is the saturated adsorption capacity; k is the constant rate of adsorption; and t is the adsorption time. Both the excellent linear relationship between $-\ln(1-Q/Q_0)$ and t shown in Figure 3 and $R^2 > 0.93$ shown in Table II indicate that the adsorption process of PS-BMT for Hg²⁺ could be precisely described by the G. E. Boyd equation, meaning that this adsorption obeyed the mechanism of liquid film diffusion control.²⁹

According to the Arrhenius equation, $\ln k = -E_a /RT + \ln A$, where *k* is the constant of adsorption, plotting $\ln k$ against 1/T, Figure 4 was obtained. The apparent activation energy of adsorption E_a calculated from the linear slope was 18.56 kJ·mol⁻¹. This low activation energy compared to that of the typical chemical reaction of 65–250 kJ·mol⁻¹ implied that the adsorption of PS-BMT for Hg²⁺ was a facile procedure.

Isothermal adsorption

The adsorption isotherms of the mercuric ions at pH 2.0 are presented in Figure 5 for three different temperatures. The isotherms revealed that the adsorption capacities increased with equilibrium concentration. This was Brunauer I-type adsorption and had typical



Figure 4 The relationship between $\ln k$ and 1/T.



Figure 5 Adsorption isotherms of PS-BMT for Hg^{2+} .

Langmuir characteristics. While treating the data in Figure 5 with Langmuir (eq. 2) and Freundlich (eq.3) equations, respectively, Figures 6 and 7 were obtained,

$$CQ^{-1} = CQ_0^{-1} + (Q_0 b)^{-1}$$
⁽²⁾

$$\lg Q = n^{-1} \lg C + \lg k, \tag{3}$$

where the parameter of the equations are the following: Q is the adsorption capacity; C is the equilibrium concentration; Q_0 is the saturated adsorption capacity; b is an empirical parameter; n is the Freundlich constant; and k is the binding energy constant reflecting the affinity of the resin to metal ions.

All parameters in the Langmuir and Freundlich equations are listed in Table III. The correlation coefficients showed that, in general, the Langmuir model fitted the results much better than the Freundlich model, indicating that all adsorption processes could be described by the Langmuir formula. The R_F^2 = 0.9402 at 304 K showed that the adsorption process was also described approximately by the Freundlich equation at a lower temperature.

Figure 8 gives the curve of $\lg D$ versus 1/T, where *D* is the distribution ratio, D = Q/C, and *Q* and *C* are



Figure 6 The Langmuir isotherms of PS-BMT for Hg^{2+} .



Figure 7 The Freundlich isotherms of PS-BMT for Hg^{2+} .

adsorption capacity and the concentration of free metal ion at the equilibrium adsorption, respectively. The result revealed that the distribution ratio increased with increasing temperature. This means that the adsorption process was an endothermal process. From Figure 8, it could be concluded that the linear slope was -855.47 and the correlation coefficient was 0.9962. According to $lgD = -\Delta H/(2.303RT) + \Delta S/R$, ΔH and ΔS could be calculated as 16.38 kJ·mol⁻¹ and 37.36 J·mol⁻¹·K⁻¹. That ΔH is positive justified that the adsorption of PS-BMT for Hg²⁺ was a endothermal process, which was fitted to the results obtained from the adsorption kinetics. According to $\Delta G = \Delta H - T \Delta S$, ΔG at 298 K could be calculated as -5.99 kJ·mol⁻¹. That ΔG is negative implied that the adsorption of PS-BMT for Hg^{2+} was a spontaneous process.

Effect of pH on the adsorption of Hg²⁺

In general, the pH value was an important factor affecting the adsorption of chelating resin for metal ions, especially those containing functional groups like an amino group, which was easily protonized in the case of lower pH. In this paper, the adsorption property of PS-BMT was investigated at pH $1.0\sim5.0$ (experiment showed that further increase of pH would result in the hydrolysis and precipitation of Hg²⁺). It could be observed from Figure 9 that the pH of the solution had only a slight effect on the adsorption of PS-BMT, and PS-BMT resin maintained good adsorption for Hg²⁺ among a wide range of pH values. A possible explanation for this was that the main contributors for chelating Hg²⁺ in PS-BMT were sul-



Figure 8 lgD versus 1/T plots for Hg²⁺ adsorption on PS-BMT.

fur and nitrogen atoms that could not be protonized under this experimental condition.

The regeneration of resin

Such solutions as 0.1 mmol·L⁻¹ HNO₃, 0.5 mmol·L⁻¹ HCl, and 2% thiourea in 0.1 mol·L⁻¹ HCl were chosen to be eluent in the desorption of mercury ion adsorbed in resin. The elution procedure was similar to the regeneration of resin described above (0.1 g of resin with 0.62 mmol $Hg^{2+}g^{-1}$, 10 mL of eluent, room temperature, stirring 1 h). The experimental result showed that elution values of the above-mentioned solutions were 92.8, 89.5, and 98.4%, respectively, indicating that the third solution was the most effective. This was because thiourea was also a S- and N-containing compound, which can coordinate effectively with Hg²⁺ and capture it from PS-BMT resin after adsorption. Therefore, solution of 2% thiourea in 0.1 mol·L⁻¹ HCl was chosen to use in the experiments of regeneration of resin.

The dynamic desorption of Hg^{2+} was also investigated by the following procedure: 0.2 g of dry Hg^{2+} loaded PS-BMT resin (with 0.62 mmol $Hg^{2+} \cdot g^{-1}$) packed in a glass column ($\Phi = 5$ mm) was eluted by 20 mL of solution of 2% thiourea in 0.1 mol·L⁻¹ HCl at a flow rate of 20 mL·h⁻¹. The concentration of Hg^{2+} was determined at an interval of 2 mL of the collected eluent solution. The result showed that leakage of eluent occurred when about 14 mL of total of eluent was col-

 TABLE III

 The Parameters in the Langmuir and Freundlich Equations

Temperature (K)	Qo	$b \times 10^{-3}$	$R_{\rm L}^2$	n^{-1}	k	$R_{\rm F}^2$
304	0.4238	1.573	0.9963	0.1083	0.3836	0.9402
312	0.5292	1.453	0.9990	0.1498	1.0473	0.8652
323	0.6945	0.600	0.9779	0.2468	4.2746	0.8426
341	1.0107	0.380	0.9215	0.3361	22.1003	0.8770

lected, and about 98.8% Hg²⁺ was recovered at this time. It should be noted that the elution value 98.8% of dynamic desorption is near 98.4% of the above-mentioned static desorption. So the static desorption method was used to assess the reusability of the resin.

The results of five adsorption–desorption cycles are shown in Table IV. Interesting results could be drawn from Table IV. That is, the adsorption of PS-BMT resin had an evident increase after three cycles of adsorption–desorption. Even after five cycles the adsorption capacity of resin was higher than that in the first adsorption. A proper interpretation for the phenomenon above was that after several adsorption–desorption cycles, some pore canals that caved during the process of vacuum dryness were reopened, so the functional groups could be used fully. This showed that this resin was suitable for repeated use for more than five adsorption–desorption cycles without considerable change in adsorption capacity.

CONCLUSION

We prepared a novel chelating resin with functional group containing S and N atoms using chloromethylated polystyrene and BMT as materials. The content of the functional group was determined by elementary analysis of 2.07 mmol BMT g^{-1} resin, 47% of which were in the form of monosubstitution (PS-BMT-1) and 53% in the form of double substitutions (PS-BMT-2). The process of adsorption for Hg²⁺ was controlled by liquid film diffusion. Increasing the temperature was beneficial to adsorption. The Langmuir model was much better than the Freundlich model for describing the isothermal process. The adsorption activation energy (E_a), ΔG , ΔH , and ΔS values calculated were 18.56 $kJ \cdot mol^{-1}$, -5.99 $kJ \cdot mol^{-1}$, 16.38 $kJ \cdot mol^{-1}$, and 37.36 $J \cdot mol^{-1} \cdot K^{-1}$, respectively. The chelating resin could be easily regenerated by 2% thiourea in 0.1 mol· L^{-1} HCl with higher effectiveness. Five adsorption-desorption cycles demonstrated that this resin was suitable for repeated use without considerable change in adsorption capacity.



0.6

Figure 9 The effect of pH on adsorption of PS-BMT for Hg^{2+} .

TABLE IV Desorption and Regeneration Data^a

-	Ŭ				
Reuse times	1	2	3	4	5
Adsorption (mmol \cdot g ⁻¹)	0.62	0.65	0.70	0.69	0.66

^a The relative standard deviations (RSD) for the data are 0.38-1.32%.

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