

# Metal Sorption Properties of Sulfur-Chlorinated Jojoba Wax Bound to Polystyrene Beads

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## SYNOPSIS

A new solid extractant (designated PS-DETA-JS) in which sulfur-chlorinated jojoba wax is bound via an amine spacer group to polystyrene beads was synthesized. The absorption of mercury cations from acidic solutions and of chromate anions from saline solutions onto PS-DETA-JS was investigated. The sorption of mercury ions by the solid extractant was compared with that by liquid-sulfurized jojoba wax impregnated inside macroporous resins. The static and dynamic properties of dichromate sorption from 2–20 g/L NaCl solutions at pH 4.1 were studied. Selective sorption of Cr(VI) was obtained at low chromate concentrations (~ 6 ppm) in saline aqueous solutions. Complete regeneration of the PS-DETA-JS resin was achieved after the reduction of Cr(VI) to Cr(III) and the elution of the Cr(III) with 1N HCl. © 1997 John Wiley & Sons, Inc.

## INTRODUCTION

Sulfurized jojoba wax serves as an excellent extractant for the extraction of mercury ions from nitric acid both in liquid-liquid and in solid-liquid extraction processes<sup>1–3</sup> (Scheme 1). However, the application of solvent-impregnated polystyrene or polyacrylate resins in which sulfurized jojoba was absorbed suffers from one of the main disadvantages of solvent extraction—loss of the organic extractant into the aqueous phase.<sup>4–6</sup> An additional problem is that solvent extraction processes are limited by phase separation difficulties at pH values exceeding 1 in the aqueous phase.<sup>1</sup>

The concept of tailoring metal-specific ligands covalently bonded to a polymer support in the form of beads or membranes is one of the approaches used to solve these problems.<sup>7,8</sup> The attachment of appropriate multidentate ligands<sup>9,10</sup> to an insoluble polymer matrix is a common technique for the preparation of metal-selective ion-exchange resins.<sup>11,12</sup> In most cases, the synthesis

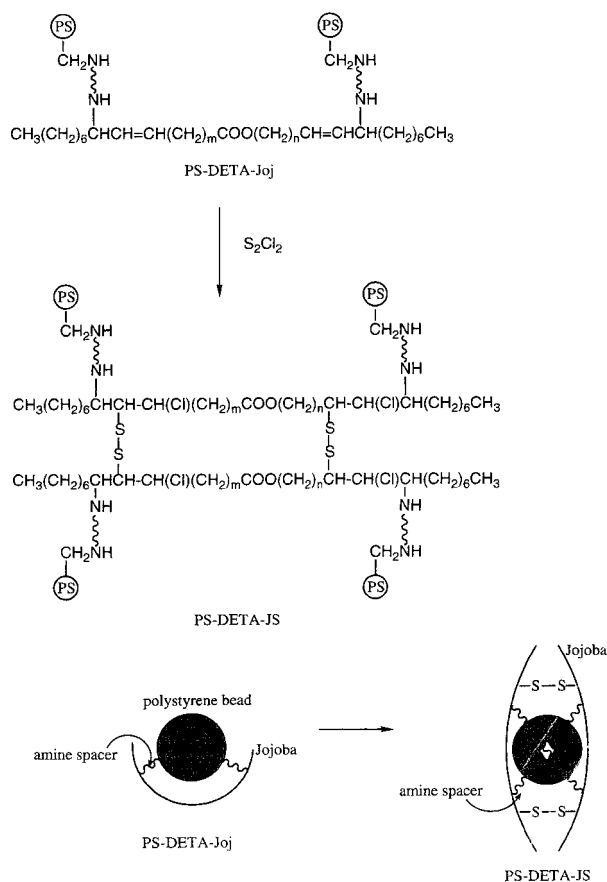
involves the addition of suitable functional groups or ligands to a crosslinked polystyrene matrix.<sup>13–15</sup> A variety of synthetic resins with donor N and S atoms in their functional groups were found to be promising exchangers for selective preconcentration and the separation of noble metals;<sup>16,17</sup> for example, the commercial Sraffion NMR and Monivex resins are isothiouranium sorbents of this type. The excellent extraction properties of compounds with thio and mercapto functional groups for heavy metal cations (Hg, Pd, Cd, etc.) have long been recognized.<sup>18,19</sup>

Recently, we prepared a series of solid extractants comprising metal-specific ligands based on functionalized jojoba wax bound to polystyrene beads.<sup>20,21</sup> One of the synthesized products, designated PS-DETA-JS, consisted of sulfur-chlorinated jojoba wax chemically bound to polystyrene via a low-molecular-weight polyethyleneimine spacer group, i.e., diethylenetriamine (DETA).

The polyethyleneimine itself acts as an effective complexant of various transition metal ions<sup>22</sup>: chelating acrylic resins having oligoethyleneimine moieties can be used for the selective removal of heavy metal ions from waste water.<sup>23,24</sup> Oligoethylenimines attached to polystyrene matrices readily form complexes with a number of

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**Scheme 3**

beads (PS-DETA) containing 5.6% nitrogen gave an anion-exchange capacity of 4.50 mEq/g.

### Synthesis of PS-DETA-JS Polymer

Jojoba wax in the form of the diallylbromide derivative (J-2Br) dissolved in dioxan was bound to the PS-DETA resin to give a polymer designated PS-DETA-Joj that contained 40% (w/w) of bound jojoba. The double bonds in the bound jojoba were then reacted with sulfur monochloride in nitrobenzene (Scheme 3). The final product, PS-DETA-JS, contained 6.1% chlorine and 5.0% sulfur.<sup>28</sup>

### Determination of Total Anion-Exchange Capacity

Before the measurement of the anion-exchange capacity, all resins were equilibrated alternately several times in 0.1N HCl and 0.1N NaOH. Each dry sample (0.2 g) in the OH<sup>-</sup> form was placed in a 100 cm<sup>3</sup> volumetric flask, to which was added 50 cm<sup>3</sup> of 0.1N HCl. The suspension was then

placed in a shaker for 24 h. Thereafter, 5 cm<sup>3</sup> of the solution was back-titrated with 0.05N NaOH. The capacity of the anion-exchange resin was calculated from the results of the titration as  $Q_{H^+}$  in milliequivalents per gram.

### Determination of the Sorption Properties of PS-DETA-JS Polymer

#### Mercury Ion Extraction (Batch Experiments)

Air-dried resin samples (100 mg) of the PS-DETA-JS polymer were equilibrated by mechanical shaking with 2–150 cm<sup>3</sup> of 1,000–5 ppm Hg(II) [added as Hg(NO<sub>3</sub>)<sub>2</sub>]. The pH was adjusted to 1 with conc. HNO<sub>3</sub> or with 50 cm<sup>3</sup> of 5–50 ppm Hg(II) in 0–1N HNO<sub>3</sub>. After 24 h of shaking at room temperature, aliquots were taken from the aqueous phase and analyzed by ICP emission spectroscopy. The absorption capacity of the polymer was calculated as follows:  $Q = V(C_1 - C_2)/W$ , where  $Q$  is the absorption capacity of the polymer (in milligrams per gram),  $W$  is the weight of polymer (in grams),  $V$  is the volume of the solution (in cubic centimeters), and  $C_1$  and  $C_2$  are the concentrations of the mercury ions before and after the absorption processes, respectively (in milligrams per cubic centimeter).

#### Regeneration of Loaded Mercury Ions from PS-DETA-JS

A 10% Na<sub>2</sub>SO<sub>3</sub> solution was used for the elution of the loaded mercury ions from the PS-DETA-JS polymer. Then, the samples were regenerated with 0.1N NaOH before reuse. Three cycles of loading and elution were performed with each polymer sample, with no deterioration of efficacy.

#### Dichromate Extraction (Batch Experiments)

Air-dried resin samples (100 mg) were equilibrated by mechanical shaking with three aqueous solutions: 0.001–0.1N K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>, 0.001–0.1N K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> in 0–20 g/cm<sup>3</sup> NaCl, and 0.0004N K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> in 18 g/cm<sup>3</sup> NaCl + 2 g/cm<sup>3</sup> CaCl<sub>2</sub>. After 24 h of shaking at room temperature, the concentration of Cr(VI) in the aqueous solution was measured by ICP, and the concentration of Cl<sup>-</sup> anions was determined by argentometric titration with a 6.0404.100 (DB) combined electrode (Metrohm). From the difference in anion concentrations, the absorption capacities of the polymer for dichromate anions  $Q_{Cr}$  and chloride  $Q_{Cl}$  (in milliequivalents per gram) anions were calculated.

The selectivity constant of the polymer for dichromate anions was calculated as follows:  $K_s = Q_{Cr} * C_{Cl} / C_{Cr} * Q_{Cl}$ , where  $C_{Cr}$  and  $C_{Cl}$  are the equilibrium concentrations of dichromate and chloride anions in aqueous solution, respectively.

### Dichromate Extraction and Recovery by Column Technology

A glass column with an internal diameter of 0.5 cm was filled with 1 g of PS-DETA-JS polymer and suspended and preequilibrated in 0.1N HCl. The height of the polymer beads was 5 cm. The solution containing 6 ppm Cr(VI) (added as  $K_2Cr_2O_7$ ) in the presence of 18 g/L NaCl and 2 g/L  $CaCl_2$  at pH 4.1 was passed through the column at 50 bed volumes (BV)/h. Fractions of the eluate were collected at fixed intervals (every 10 BV), and the concentrations of Cr(VI) were determined by ICP emission spectroscopy.

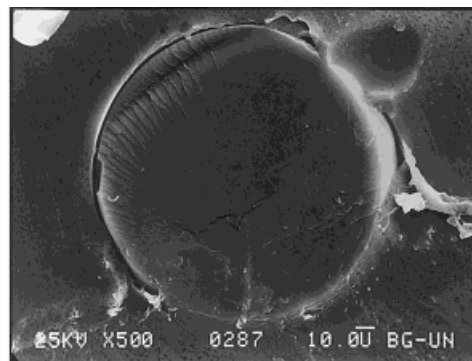
The regeneration of loaded Cr(VI) ions on the polymer was performed in two stages. First, a 10%  $NaHSO_3$  solution was passed through the column at 5 BV/h, and thereafter, the Cr(III) ions were eluted from the polymer with 1N HCl at 10 BV/h.

The elution of dichromate anions from the anion-exchange resins was performed with 1N NaOH. The loading and elution experiments were performed three times with each polymer sample, with the same efficacy.

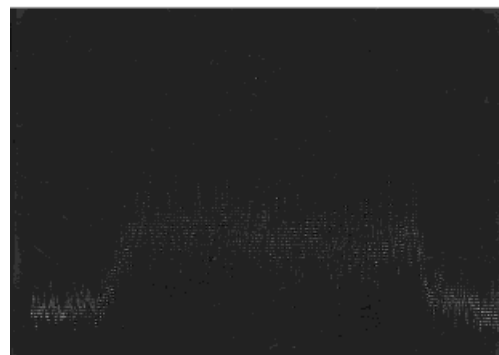
## RESULTS AND DISCUSSION

### Synthesis and Structure of PS-DETA-JS Polymer

The PS-DETA-JS polymer was obtained by a sequence of synthetic steps. First, the polystyrene aminated with DETA (PS-DETA) was reacted with the dibromoallyl derivative of jojoba wax (J-2Br), via a nucleophilic substitution reaction, to form C—N covalent bonds. Then, the double bonds in the bound jojoba wax were sulfur chlorinated (Scheme 3). The polymer contained 40% of bound jojoba wax (0.4 g of jojoba per gram product) linked by —S—S— bridges (1.56 mmol of S/g) and had an anion-exchange capacity of 0.5 meq/g because of the presence of free amino groups.<sup>28</sup> SEM studies revealed that the distribution of the chlorine atoms in the polymer was homogeneous, as may be seen from the line-scanning profile (LSP) (Fig. 1).



(a)



(b)

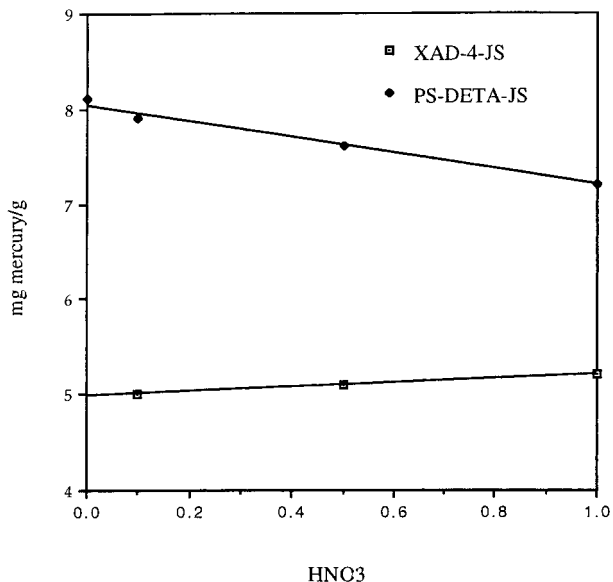
**Figure 1** (a) Cross-section of PS-DETA-JS polymer. (b) LSP-C1 in the cross-section of the PS-DETA-JS bead.

### Sorption of Mercury Ions

For purposes of comparison, results of similar experiments performed previously with macroporous resins impregnated with liquid-sulfurized jojoba wax are presented.

#### Effect of Acid Concentration

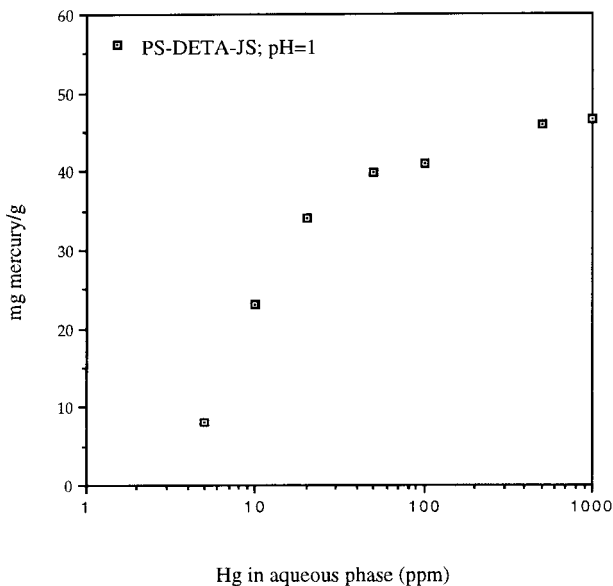
The influence of pH on the sorption of mercury metal ions (5 ppm) by PS-DETA-JS was determined over a range of acidity (0–1N  $HNO_3$ ). The experimental results are presented in Figure 2, together with the results of mercury extraction by sulfurized jojoba wax impregnated into an XAD-4 macroporous polystyrene matrix (XAD-4-JS).<sup>3</sup> The uptake of mercury by PS-DETA-JS decreased slightly as the concentration of nitric acid increased, while that by the XAD-4-JS polymer was not affected by the acidity. The PS-DETA-JS extractant exhibited 150–160% higher mercury capacities than did the XAD-4-JS-impregnated polymer at low mercury concentrations in acid and neutral media.



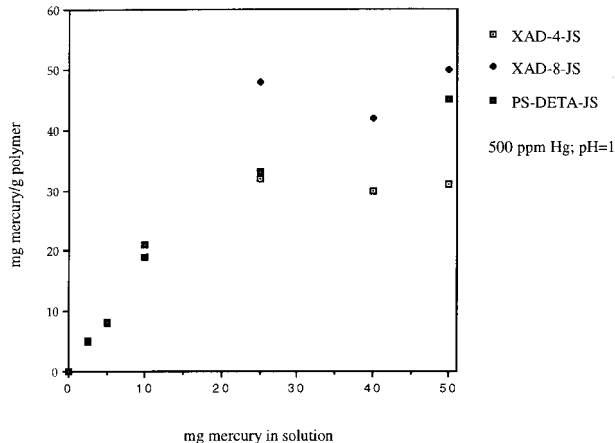
**Figure 2** Milligrams of mercury adsorbed per gram of XAD-4-JS and PS-DETA-JS polymers as a function of HNO<sub>3</sub> concentration in the aqueous phase.

**Effect of Mercury Ion Concentration**

The influence of mercury concentration in the aqueous phase on mercury adsorption by PS-DETA-JS at pH 1 is shown in Figure 3. It can be seen from the adsorption isotherm that the mercury capacity of the polymer exhibits an almost linear increase as the original concentration of



**Figure 3** Milligrams of mercury adsorbed per gram of PS-DETA-JS polymer at pH 1 as a function of the concentration of mercury ions in the aqueous phase.



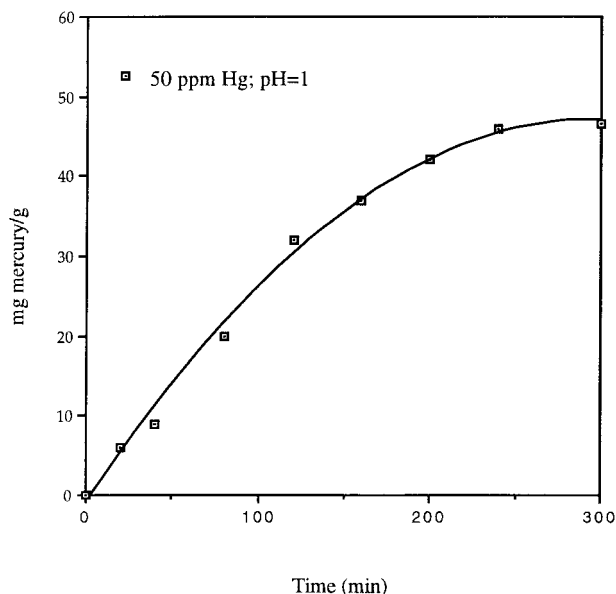
**Figure 4** Milligrams of mercury adsorbed per gram of XAD-4-JS, XAD-8-JS, and PS-DETA-JS polymers from different volumes of 500 ppm Hg aqueous solution at pH 1 as a function of milligrams of mercury in solution.

mercury ions in the aqueous phase is increased, up to ~100 ppm of Hg. At higher concentrations, only a slight increase in mercury uptake was observed.

**Effect of the Polymer Matrix**

A series of experiments were conducted to determine the amount of mercury adsorbed as a function of the concentration of mercury in the aqueous solution. A known amount of PS-DETA-JS polymer was equilibrated with a 500-ppm mercury solution at pH 1 (pH adjusted with nitric acid). Figure 4 shows the mercury extraction by three different types of solid extractants: PS-DETA-JS polymer (0.40 g of JS/g of resin) and two impregnated resins, XAD-4-JS (0.88 g of JS/g of resin) and XAD-8-JS (1.00 g of JS/g of resin).<sup>3</sup> Similar absorption behavior was obtained for all three polymers. PS-DETA-JS was capable of absorbing about 46 mg of Hg per gram of polymer; the corresponding values for XAD-4-JS and XAD-8-JS were about 34 and 50 mg of Hg per gram of resin, respectively.<sup>3</sup>

Although the solvent-impregnated resins contain roughly twice as much sulfurized jojoba (0.88–1.00 g) and S (3.3–3.6 mmol) per gram of resin versus PS-DETA-JS, the two types of polymer exhibit similar sorption capacities. It thus seems that the donor character of amino groups contributes to mercury sorption and that the extraction behavior of the solid extractant in which sulfur-chlorinated jojoba is chemically bound is similar to that of the sulfurized jojoba extractant



**Figure 5** Milligrams of mercury adsorbed per gram of PS-DETA-JS polymer from 50 ppm Hg aqueous solution at pH 1 as a function of time.

physically absorbed inside the macroporous resin. The advantage of the solid extractant is that the extraction and regeneration cycles may be repeated at least three times.

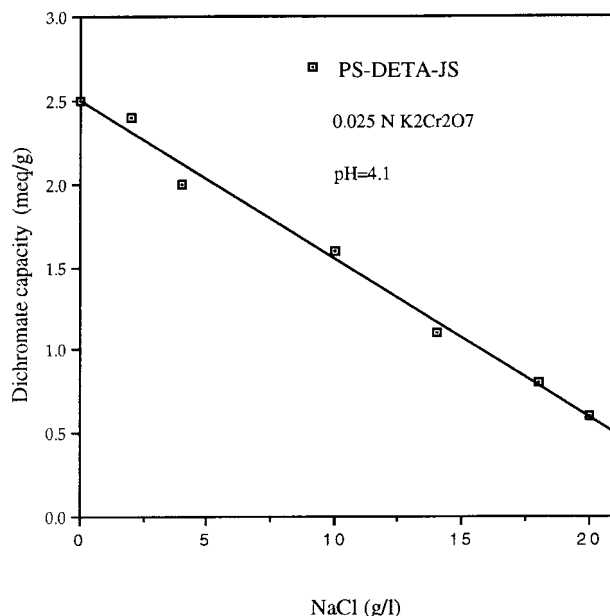
#### Kinetics of Mercury Sorption

The kinetics of mercury adsorption from a 50-ppm Hg solution was followed for the PS-DETA-JS polymer at pH 1 (Fig. 5). Equilibrium was achieved within 4 h with PS-DETA-JS, while 2 h was sufficient for the solvent-impregnated resins.<sup>3</sup> This could be explained by the fact that adsorption in the PS-DETA-JS polymer takes place inside the gel matrix, and the rate of the diffusion of mercury ions into the polymer is therefore slow in comparison to the diffusion of ions inside the solvent-impregnated resins. The elution of loaded mercury ions from PS-DETA-JS with 10%  $\text{Na}_2\text{SO}_3$  solution gave almost 100% recovery of Hg(II).

#### Sorption of Dichromate Anions from Solutions Containing High Salt Concentrations

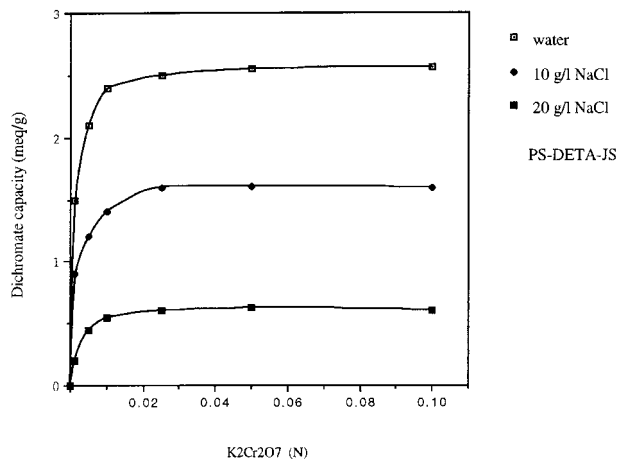
##### Batch Experiments

The equilibrium sorption of dichromate anions on PS-DETA-JS was measured in NaCl media of different concentrations. The Cr(VI) sorption capacities of PS-DETA-JS decreased as the concentration of  $\text{Cl}^-$  anion in the aqueous phase increased



**Figure 6** Dichromate capacity of PS-DETA-JS polymer as a function of NaCl concentration in a 0.025N  $\text{K}_2\text{Cr}_2\text{O}_7$  aqueous solution at pH 4.1.

(Fig. 6). From the sorption isotherms plotted in Figure 7, it is evident that the chromate capacity increased sharply at first, with an increase in the concentration of chromate anions in the aqueous phase, and then leveled out. The polymer reached optimal capacities at considerably low Cr(VI) concentrations of about 0.01N, both in water and in 10 and 20% NaCl solutions. Thus, PS-DETA-JS has good sorption selectivity for Cr(VI) ions at low chromate and high chloride concentrations in the aqueous phase.



**Figure 7** Dichromate capacity of PS-DETA-JS polymer as a function of  $\text{K}_2\text{Cr}_2\text{O}_7$  concentration in aqueous and saline solutions.

**Table I Extraction of Cr(VI) from Concentrated Chloride Solutions**

Resin	Polymer Matrix	Functional Group	$Q_{Cl^-}$ (mEq/g)	Dichromate Capacity (mEq/g)			$K_s^d$
				$Q^a$	$Q^b$	$Q^c$	
Dowex 1-X8	PS <sup>e</sup>	S.B. <sup>e</sup>	3.00	4.20	1.45	>0.1	29
IRA-93	PS	W.B. <sup>e</sup>	3.02	1.30	0.45	>0.1	28
Lewatite 304/88	AC <sup>e</sup>	W.B.	4.00	1.25	0.30	>0.1	21
PS-DETA <sup>e</sup>	PS	W.B.	4.50	4.25	0.80	>0.1	19
PS-DETA-Joj-SC1	PS <sup>e</sup>	W.B.-JS	0.55	2.50	1.60	0.60	5000

<sup>a</sup> 0.025N  $K_2Cr_2O_7$ .

<sup>b</sup> 0.025N  $K_2Cr_2O_7$  in 10 g/L NaCl.

<sup>c</sup> 0.0004N  $K_2Cr_2O_7$  in 18 g/L NaCl + 2 g/L  $CaCl_2$  at pH 4.1.

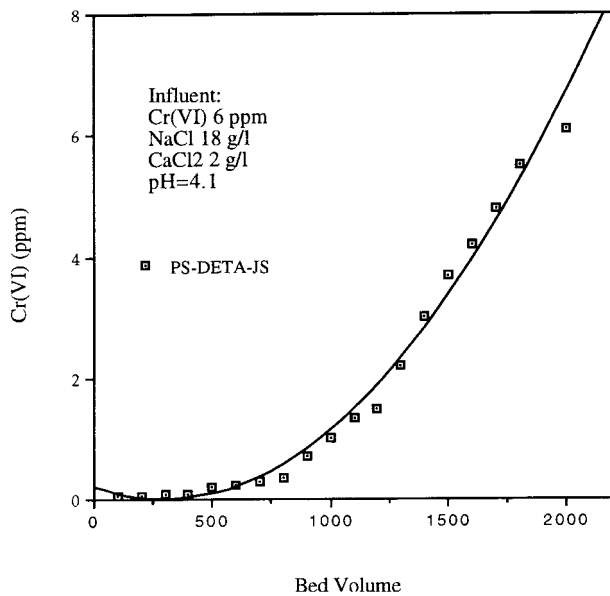
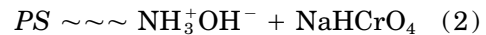
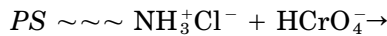
<sup>d</sup>  $K_s$  based on extraction results from solution c.

<sup>e</sup> DETA, diethylenetriamine; PS, polystyrene; AC, polyacrylate; S.B., strong base; W.B., weak base.

In order to compare the absorption of chromate anions by different polymers in the presence of competitive chloride anions, the following series of experiments was performed. The extraction of dichromate anions by commercial anion-exchange resins and our polymers from aqueous solutions or mixed ion solutions with a high  $Cl^-$  concentration at pH 4.1 was tested (Table I). The results indicate that the chromate selectivity for both the anion exchangers and the PS-DETA-JS solid extractant in the presence of competing chloride anions depends on the concentration of  $Cl^-$  anions in the aqueous phase. The extraction behavior of the commercial anion-exchange resins toward chromate anions was in agreement with previously reported results.<sup>26,27</sup> However, both weak- and strong-base resins failed to extract chromate from mixed solutions in which the concentration of Cr(VI) was low (~ 6 ppm) and that of  $Cl^-$  anions was about 20,000 times higher (20 g/L). Under the same experimental conditions, the PS-DETA-JS polymer selectively extracted Cr(VI) ions, and the calculated dichromate capacity is 0.6 mEq/g. The chromate selectivity of the PS-DETA-JS solid extractant under slightly acidic conditions (pH 4.1) and a high concentration of  $Cl^-$  anions was found to be higher than that of the other tested anion-exchange resins ( $K_s = 5,000$  vs. 20–30; Table I). All of our chromate sorption experiments were performed with aqueous solutions at pH 4.1–4.5, at which value the predominant species of hexavalent chromium Cr(VI) is  $HCrO_4^-$ , whereas both  $HCrO_4^-$  and  $Cr_2O_7^{2-}$  exist in the exchange phase.<sup>27</sup>

The selective anion-exchange reaction of base anion exchangers (all of the resins in Table I, except PS-DETA-JS) toward chromate anions was

considerably reduced in the presence of a high concentration of competitive  $Cl^-$  anions (20 g/L). However, the chromate anions loaded on these resins were completely eluted with 1N NaOH. The sorption [eq. (1)] and desorption [eq. (2)] anion-exchange reactions can be represented by the following equations:



**Figure 8** Cr(VI) concentration in the aqueous phase as a function of bed volume.

**Table II EDS Analysis of the External Surface of PS-DETA-JS Polymer after Mercury Extraction**

Average Bead Diameter ( $\mu\text{m}$ )	Elements (%) <sup>a</sup>			Molar Fraction <sup>b</sup> of Elements			S <sup>c</sup>	mmol S/g <sup>d</sup>
	S	Cl	Hg	S	Cl	Hg	Hg	mmol Hg/g
40–60	31.8	27.8	40.4	0.99	0.78	0.20	5.0	—
140–160	25.8	25.1	49.1	0.81	0.71	0.24	3.4	—
General	33.4	26.8	39.8	1.04	0.75	0.20	5.2	6.9

<sup>a</sup> Relative percentage of elements on the surface of PS-DETA-JS beads (EDS analyses, SEM).

<sup>b</sup> Calculated value; relative element percentage per atomic weight of element.

<sup>c</sup> Calculated value based on EDS analyses; ratio between relative molar fraction of S and Hg.

<sup>d</sup> Calculated on the basis of elemental analyses ratio between 1.56 mmol of S per gram of product and capacity of 0.225 mmol of Hg per gram of product.

The PS-DETA-JS solid extractant has weak base anion-exchange groups (0.55 mEq/g), in addition to the organic chelating ligands of the sulfur-chlorinated jojoba. The extraction behavior of the ligands differs from that of the anion-exchange groups: the —S—S— bridges can interact with extracted chromate anions, and as a result, the formation of stable chelates will occur in the polymer phase. Evidence to support this supposition comes from the fact that loaded chromate ions were not eluted with a basic solution, as they were with the other anion-exchange resins. The regeneration of the solid extractant was achieved only after the reduction of Cr(VI) to Cr(III) on the polymer by 10% NaHSO<sub>3</sub> solution, as indicated by a change in the color of the polymer from brown to blue-green. The Cr(III) ions were completely eluted with 1N HCl.

#### Fixed-Bed Column Run

The uptake of the chromate in the presence of Cl<sup>-</sup> anions (added as NaCl and CaCl<sub>2</sub>) was tested in a conventional fixed-bed column run. Complete loading of PS-DETA-JS under dynamic conditions

at 10 BV/h was obtained after 2,000 BV. The breakthrough of 0.5 mg/L of Cr(VI) was observed after 1,000 BV (Fig. 8).

#### SEM Studies

PS-DETA-JS beads were inspected by the SEM technique in order to obtain qualitative identification analyses for S, Cl, Hg, and Cr (EDS analyses) on the external surfaces, in addition to elemental analyses and capacity determination. The results are summarized in Tables II and III.

The ratio between the sulfur functional groups and the adsorbed species (mercury or chromate) was calculated on the basis of the experimental results. The ratio on the surface of the beads evaluated from EDS analyses and the ratio in the bulk volume of a bead were obtained from the ratio between elemental S analyses and the ion-exchange capacity of adsorbed species (Tables II and III).

A representative sample of PS-DETA-JS polymer consisted of two main fractions of beads with average bead diameters of 40–60  $\mu\text{m}$  and 140–

**Table III EDS Analysis of the External Surface of PS-DETA-JS Polymer after Chromate Extraction**

Average Bead Diameter ( $\mu\text{m}$ )	Elements (%) <sup>a</sup>			Molar Fraction <sup>b</sup> of Elements			S <sup>c</sup>	mmol S/g <sup>d</sup>
	S	Cl	Cr	S	Cl	Cr	Cr	mmol Cr/g
40–60	29.8	32.1	38.1	0.93	0.90	0.73	1.3	—
140–160	26.2	27.1	46.7	0.82	0.76	0.90	0.9	—
General	26.1	33.8	40.1	0.82	10.95	0.77	1.1	1.9

<sup>a</sup> Relative percentage of elements on the surface of PS-DETA-JS beads (EDS analyses, SEM).

<sup>b</sup> Calculated value; relative element percentage per atomic weight of element.

<sup>c</sup> Calculated value based on EDS analyses; ratio between relative molar fraction of S and Cr.

<sup>d</sup> Calculated value based on element analyses; ratio between 1.56 mmol of S per gram of product and capacity of 0.83 mmol of Cr per gram of product.



160  $\mu\text{m}$ . A lower ratio between sulfur atoms and adsorbed species (mercury or chromate) was found for the surface of the beads with the higher diameter. This ratio is lower on the surface than in the bulk volume of the beads (5.2 versus 6.9 for mercury extraction and 1.1 vs. 1.9 for chromate extraction). These somewhat higher concentrations of adsorbed species on the bead surface vs. inside the bead could be the result of some diffusion limitations of the ionic species into the bulk polymer matrix. The ratio between the adsorbed mercury ions and the sulfur atoms present in the polymer was found to be 1:5–7. Higher ratios of 1 : 2 were obtained for the sorption of chromate ions with the same matrix.

## CONCLUSIONS

The solid polystyrene gel-type matrix with chemically bound jojoba wax functionalized by sulfur chlorination was active in the absorption of mercury (II) cations and chromate (VI) anions for several runs without a loss of activity. In the case of Cr(VI) sorption, there was high selectivity in the presence of high concentrations of  $\text{Cl}^-$ , in contrast to base anion exchangers that do not absorb Cr(VI) and thus show no selectivity.

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