Adsorption Behavior and Separation of Vanadium(V), Molybdenum(VI), and Rhenium(VII) Ions on Crosslinked Polymers Containing Acrylic Acid Derivative Moieties

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ABSTRACT: The radical polymerization of [2-(acryloyloxy)ethyl]trimethylammonium methyl sulfate and [3-(methacryloylamino)propyl]trimethylammonium chloride by using ammonium peroxydisulfate as initiator and *N,N*^{\prime}-methylene-bis-acrylamide as crosslinker agent at 70°C for 17 h was carried out. The crosslinker agent in the feed ranged between 2 and 6 mol %. The resins were completely insoluble in water and characterized by elemental analysis and FTIR spectroscopy. The metal ion binding capacity of these resins with $V(V)$, $Mo(VI)$, and $Re(VII)$ were investigated using the batch equilibrium type. The pH, but not the crosslinking percentage in the feed, influenced the retention capacity of the resin, particularly for V(V) and Re(VII). Thus, at pH 1 only $\lt 15\%$ of V(V) was retained, but at pH 3 higher than 97% of V(V) was adsorbed. The resin–ion equilibrium was achieved between 15 min and 2 h depending on the resin, ion, and crosslinking degree. According to the pH, it is possible separate Re(VII) and Mo(VI) from V(V) at pH 1.0. To reuse the resins, the ions were stripped by using HCl, H_2SO_4 , and Na_2CO_3 at different concentrations. © 1999 John Wiley & Sons, Inc. J Appl Polym Sci 73: 369–376, 1999

Key words: metal ion chelating resin; batch equilibrium; vanadium; molybdenum; rhenium

INTRODUCTION

Associated with the copper mines important metals vanadium(V), molybdenum(VI), and rhenium- (VII) have a high commercial value. The latter two are used in steel blends, manufacturing of electrodes, etc., and to manufacture thermoelectric couples, catalysts of oxidation, and hydrogenation reactions.

Of the two most common forms, vanadium(IV) and vanadium (V) , the latter is the one active species prevailing in contact with atmospheric oxygen. The rate of oxidation depends on the acidity of the environment. For instance, at $pH < 2.4$,

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vanadium(IV) is more stable also in the presence of oxygen.¹

The chemistry of Mo in aqueous phase is more complex due to the multiple oxidation states and different species that can be formed from each one. The Mo(VI) is the more stable. Mo(VI) species dissolved in aqueous media exist as the monomeric $MoO₄²⁻$ in basic solutions, but various polymerized species, such as $Mo_{7}O_{24}^{6-}$, $HMo_{7}O_{24}^{5-}$, and $H_2Mo_7O_{24}^{4-}$, are successively formed with decreasing \bar{p} H solutions,^{2,3} the equilibrium constants of which are reported in literature. 4.5

On the other hand, Re shows only one species in the VII oxidation state, ReO_4^- . To separate these different metal ions from an aqueous solution, one of the employed techniques is the solvent extraction. $6-10$ However, it has many disadvantages, such as the large loss of organic reagent, the high input of extractant, the high cost of the chemicals, etc. One alternative is the use of the

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Resin	MBA $\pmod{\%}$	Dry Weight (g)	Yield $(\%)$	Wet Weight (g)	Swelling Ratio
1.4	4	9.0	92	527	58.5
1.5	5	9.2	94	395	42.9
1.6	6	9.3	95	119	12.7
2.2	$\overline{2}$	10.0	93	1100	110.0
2.4	4	10.2	94	780	76.5
2.6	6	10.8	98	269	24.9

Table I Polymerization of [2-(Acryloxy)ethyl]trimethylammonium Methyl Sulfate and [3- (Methacryloylamino)propyl]trimethylammonium Chloride with Ammonium Peroxidisulfate (0.5 mol %) at 60°C for 17 h

chelating resins containing the ion exchanger or ligand group at the main or side chain. $11-18$

Jyo et al.¹⁹ recently reported the behavior of a macromolecularstyrene–divinylbenzenecopolymerbased methylene phosphonic resin in the adsorption and desorption of Mo(VI). The chelating resin Chelex 100 was used for the separation and preconcentration of V from complex matrices.^{20–21}

This article reports the synthesis of water-insoluble resins by radical polymerization of [2- (acryloyloxy)ethyl]trimethylammonium methyl sulfate and [3-(methacryloylamino)propyl]trimethylammonium chloride and their binding ability for $V(V)$, $Mo(VI)$, and $Re(VII)$ by batch equilibrium procedure.

EXPERIMENTAL

Reagents

[2-(Acryloyloxy)ethyl]trimethylammonium methyl sulfate (AOTM), [3-(methacryloylamino)propyl]trimethylammonium chloride (MATA), N,N'-methylene-bis-acrylamide (MBA), and ammonium peroxy-

Figure 1 Thermograms of the resins (a) 1.6 and (b) 2.6. Heating rate, 20° C/min under N₂.

disulfate (APS) were purchased from Aldrich (Milwaukee, WI) and used as received.

Reagent grade metal ions were used and solutions were prepared by dissolving appropriate weights of the metal oxides (V_2O_5, MO_3, Re_2O_7) in deionized water. Hydrochloric acid and sodium hydroxide standard solutions were used to adjust the pH.

Preparation of the Resins

Resins were synthesized by radical polymerization of AOTM and MATA monomers at 70°C by using APS and MBA as initiator and crosslinker agents, respectively. After 17 h, the resins were filtered, washed with deionized water, and dried to constant weight. The dried resins were ground and sieved and the $180-250$ - μ m portion was used in all the sorption experiments (see Table I).

Recovery of Metal Ions from Aqueous Solution by Batch Method

The recovery of V(V), Mo(VI), and Re(VII) was carried out by means of a batch equilibrium procedure at different pH. Dry resin (50 mg) was stirred with 10 mL of an aqueous solution of V [858 ppm in $V(V)$], Mo [1000 ppm in Mo(VI)], and Re $[1154$ ppm in Re(VII)], at 25° C for 1 h.

The Mo(VII), V(VI) in eluate were determined by atomic absorption spectrometry, whereas Re- (VII) was determined colorimetrically.

Maximum Adsorption Capacity of the Resins

In a flask were placed 0.5 g of the resin and 50 mL of an unbuffered metal ion solution $[1 \text{ g/L in } V(V)]$ and $Mo(VI)$ and 1.15 g/L in $Re(VII)$]. The mixture was shaken at 25°C; after 2 h it was filtered, washed with water, and the filtered solution transferred into a calibrate flask. The process was repeated three times, the volume made up to 250 mL, and then the amounts of V(V), Mo(VI), and Re(VII) adsorbed into the resins were calculated from the determination of these metal ions in the supernatant.

Rate of V(V), Mo(VI), and Re(VII) Uptake

A resin (50 mg) and aqueous solution of metal ion at pH 3.0 (10 mL) were taken into a flask. After the determined time, the metal ions were analyzed in the filtrates. From the concentration decrease of metal ion in the solution, metal ion uptake was calculated at the sampled time.

Separation of the Metal Ions from Binary Mixtures at pH 1

The following binary mixtures were evaluated as V(V)–Mo(VI), V(V)–Re(VII), and Mo(VI)–Re(VII). An aqueous solution of 5 mL of each metal ion [pH 1, 1 g/L in $V(V)$ and Mo(VI); pH 1, 1 g/L in $V(V)$ and 1.15 g/L in Re(VII); and pH 1, 1 g/L in Mo(VI) and 1.15 g/L in Re(VII)] was mixed for 1 h at 25° C with 0.5 g of dry resins. Then, the resin was filtered and washed repeatedly with deionized water. All the metal ions were analyzed in the filtrate as previously described.

Recovery of the Resin

Elution assays were carried out with the metal ion–loaded resin at maximum capacity by placing 50 mg of loaded resin with 10 mL of an aqueous solution of eluent (HCl, $HNO₃, H₂SO₄, Na₂CO₃$) of different concentrations for 1 h at 25°C. The resin was separated by filtration and the metal ion was analyzed in the supernatants as previously described.

Apparatus

V(V) and Mo(VI) were analyzed on a Perkin– Elmer 1100 atomic absorption spectrophotometer and Re(VII) was analyzed with a CADAS 100 spectrophotometer. The pH was measured with a digital Extech microcomputer pH meter. The FTIR spectra were recorded on a Magna Nicolet 550 spectrophotometer. Thermal stability analy-

sis was performed on a Polymer Laboratory STA-625 system with a heating rate of 10°C/min under N_2 .

RESULTS AND DISCUSSION

Synthesis and Characterization of the Resins

The copolymer beads of crosslinked resins CAOTM and CMATA were prepared with MBA (2–6 mol %) as crosslinking reagent and APS as initiator. All the resins were completely insoluble in water and in common organic solvents.

Figure 2 Sorption $(\%)$ of $V(V)$ ($\Box \Box$), Mo(VI) $(-\blacksquare-)$, and Re(VII) $(-\blacksquare-)$ at pH = 1.0 by the CAOTM (1.4, 1.5, and 1.6) and CMATA (2.2, 2.4, and 2.6) resins.

Figure 3 Time course of vanadium(V) uptake. Conditions: 50 mg of each resin; 10 mL of solution containing 1000 ppm in V; pH = 3.0; temperature: 25° C; resin 1.4 (- \bullet —); 1.5 (- \bullet —); and 1.6 (- \bullet —).

Resin CAOTM: $A = -CO_2CH_2CH_2N(CH_3)_3(OSO_3CH_3)$ and R = H; resin CMATA: $A = -COMH(CH₂)₃$ - $N(CH₃)₃Cl₋$ and $R = -CH₃$; D = $-COMHCH₂NHCO-$

The crosslinked resins from [2-(acryloyloxy) ethyl]trimethylammonium methyl sulfate, CAOTM, correspond to the resins 1.4, 1.5, and 1.6, and those from [3-(methacryloylamino)propyl]trimethylammonium chloride, CMATA, correspond to the resins 2.2, 2.4, and 2.6, respectively (see

Table III Dependence of the Metal Ion Sorption (%) with the Contact Time

				Time (min)	
Resin	Metal Ion ^a	15	30	60	120
1.4	V(V)	98	95	99	99
1.5	V(V)	95	99	99	99
$1.6\,$	V(V)	72	84	91	99
2.2	V(V)	99	98	99	99
2.4	V(V)	99	99	99	99
2.6	V(V)	99	99	99	99
1.4	Re(VII)	81	87	87	88
1.5	Re(VII)	84	87	84	84
$1.6\,$	Re(VII)	87	88	85	85
$2.2\,$	Re(VII)	75	75	76	79
2.4	Re(VII)	78	83	86	86
$2.6\,$	Re(VII)	79	83	82	83

Temperature 25°C; sample: 0.5 g; pH 3.0.

^a Metal ion concentration: 10 mL of an aqueous solution ion containing 1000 ppm in V(V) or Re(VII).

Figure 4 Maximum load capacity (MLC) of the CAOTM and CMATA resins for Re(VII). Conditions: 0.5 g of each resin; 100 mL of solution containing 1.15 g/L in rhenium; $pH = 3.0$; temperature: 25°C; time: 2 h. Resin 1.6 (\longrightarrow) and 2.6 (\longrightarrow).

Table I). The yield was higher than 91%; for the resin 2.6 the yield was 98%.

The FTIR spectra show, among the most characteristic absorption bands, signals at 3457 cm^{-1} (N—H, secondary amide), at 1738 cm^{-1} (C=O, carboxylic acid), at 1670 cm^{-1} (C=0, amide), at 1483 cm⁻¹ (C-N), at 1202 cm⁻¹ (sulfonic ester), and at 967 cm^{-1} (S-O).

The swelling ratio was calculated by the following equation:

Swelling ratio $=$ weight of wet resin (g)
weight of dry resin (g)

In both CAOTM and CMATA resins, the swelling ratio was higher for the lowest crosslinking degree employed, 58.5 and 110, respectively (See Table I). Both resins show a similar thermal behavior up to about 400°C, losing close to 70% of their weight. Therefore, the CMATA resin continues the weight loss, whereas CAOTM remains almost constant (see Fig. 1).

Batchwise Metal Ion Uptake by Selected Resins

Metal ion uptake was evaluated by batch equilibrium procedure. Detailed conditions appear in the experimental section. The particle size fraction employed was $180-250 \mu m$. The metal ion binding properties of the CAOTM and CMATA resins were evaluated in the pH range between 1 and 9 (see Table II).

The strongest resin–metal ion interaction is observed for $V(V)$ for both resins from $pH = 3.0$. The two resins adsorb lower than 13% V(V) at pH 1.0. One of them, resin 1.4, adsorbs V(V) but higher $Re(VII)$ (58%) and Mo(VI) (71%) at pH 1.0. In general, there is not an important effect of the crosslinking degree on the metal–ion sorption. Only the CMATA resin shows a decrease of the retention

Resin	Metal Ion	Metal Ion in the Feed (mmol)	Metal Ion Adsorbed for Each Contact (mg)				
			1st	2nd	3rd	4st	MLC (mmol)
1.4	V(V)	3.9	99	05	41		3.7(95)
	Mo(VI)	3.1	99	99	97		3.1(100)
$1.5\,$	V(V)	4.6	99	93	41		4.6(94)
	Mo(VI)	3.1	99	99	98		3.1(100)
1.6	V(V)	3.9	99	72	49		3.2(82)
	Mo(VI)	3.1	99	99	64		2.7(87)
	Re(VII)	1.8	100	82	65		1.3(72)
2.2	V(V)	5.9	98	99	99	31	5.5(93)
	Mo(VI)	3.1	98	99	98		3.1(100)
2.4	V(V)	4.9	99	99	99	49	4.9(100)
	Mo(VI)	3.1	99	99	99		3.1(100)
2.6	V(V)	4.9	88	88	88	98	4.9(100)
	Mo(VI)	3.1	99	99	99		3.1(100)
	Re(VII)	1.8	94	73	60		1.2(67)

Table IV Maximum Load Capacity (MLC) for Each Metal Ion After Three or Four Contacts

Amount of sample: 0.5; temp.: 25°C; time: 2 h (each contact). Values within parentheses represent MLC percent.

Figure 5 FTIR spectra of the resin 1.6 (a) for V(V) (b), $Mo(VI)$ (c), $Re(VII)$ (d), and resin 2.6 (e) for (V) (f), Mo(VI) (g), and Re(VII) (h).

of Re(VII) $(22%)$ when 6 mol % of MBA was employed (see Fig. 2). It can suggest a control diffusion of the metal ion to the ion exchange sites.

The retention behavior can be explained by considering the sorption mechanism as an anionic exchange where the most relevant species like VO_4^{2-} , MoO_4^{2-} , and ReO^{4-} are involved with the chloride and methylsulfate ion exchanger groups. V and Mo are more easily retained than Re due to their high charge. According to these results, it would be possible to separate $Mo(VI)$ from $V(V)$ at $pH = 1.0$ by the resin 1.5 and Mo(VI) from V(V) and Re(VII) by the resin 2.6.

Figure 3 and Table III show the time course of the V(V), Mo(VI), and Re(VII) uptake by CAOTM resin. Although the three resins exhibit the similar time course, within 30 min, the metal ion uptake by CAOTM consists of rapid process, attributed to the metal ion uptake by the ion exchange group placed near the surfaces of microgels.

The high swelling brings the rapid diffusion of metal ions even in the bulk of the microgels, thus resin 1.4 exhibits faster uptake than resin 1.6. All the resins showed a fast uptake of Mo(VI), achieving 99% after 15 min.

Evaluation of the maximum load capacity (MLC) for the considered metal ions was carried out at $pH = 3.0$ (see Fig. 4 and Table IV). Almost all of the resins showed a very high MLC (between 3.1 and 4.9 mmol) except for the resin 2.6 for Re(VII), which was 67%. The 1.6 resin, which has the lowest swelling ratio, shows the lowest MLC values with respect to resins 1.4 and 1.5. About 40% of Re(VII) is adsorbed by the resins 1.6 and 2.6 during the first contact, and only 27% during the third contact (see Fig. 4). The loaded resins showed changes at the FTIR spectra and the thermal properties showed changes with respect to the unloaded resins. Thus, the FTIR spectra show the characteristic bands of $[VO₄]^{3-}$ $(823-831 \text{ cm}^{-1}), \text{ [MoO}_4]^2$ ⁻ $(904-905 \text{ cm}^{-1}), \text{ and}$ $[{\rm ReO}_4]^-$ (906–909 cm⁻¹) [see Fig. 5(a-h)]. The presence of these absorption signals would support the ion exchange process with chloride or methylsulfate, but it is not possible to disregard completely the involvement of the coordination through ligand groups like amino groups. The presence of V(VI), Mo(VII), and Re(VII) strongly increases the thermal stability of the resins loaded at maximum capacity, particularly at higher temperatures (e.g., 400°C). Thus, at 400°C the resins 1.6 and 2.6 lose 67.5 and 71.5% of their weight, respectively, but those loaded with Mo(VI) lose only 35.7 and 19.7% of their weight, respectively (see Table V).

The selectivity sorption from binary mixtures was then studied. The resin was suspended for 1 h in an aqueous solution containing equal

	Weight Loss Percent at Different Temperatures (°C)								
Resin	100	200	300	400	500				
$1.6\,$	4.3	4.0	11.2	67.3	75.0				
$1.6 + V(V)$	2.3	10.1	16.0	31.5	45.3				
$1.6 + Mo(VI)$	0.0	$1.5\,$	6.1	35.7	44.1				
$1.6 + \text{Re(VII)}$	2.7	4.1	11.6	32.5	44.2				
2.6	5.1	10.0	16.3	71.5	94.5				
$2.6 + V(V)$	3.7	6.5	10.5	30.3	38.8				
$2.6 + Mo(VI)$	$1.1\,$	$1.5\,$	2.8	19.7	29.9				
$2.6 + Re(VII)$	0.5	$1.0\,$	$3.0\,$	33.8	41.9				

Table V Thermal Decomposition Behavior of the Unloaded and Loaded Resins at Heating Rate 10°C min2**¹ Under Nitrogen**

amounts of two kinds of metal ions for each metal ion mixture: V(V)–Mo(VI), V(V)–Re(VII), and Mo- (VI)–Re(VII). The adsorptivity was determined by observing the concentration of two ions in the aqueous phase. The resins 1.4 and 1.6 show an excellent selectivity for Mo(VI) with respect to $V(V)$ and for $Re(VII)$ with respect to $V(V)$, but not between Mo(VI) and Re(VII). On the other hand, the resin 2.4 shows an excellent sorption selectivity for $Mo(VI)$ with respect to $V(V)$, which is similar to the resins 1.4 and 1.6. The resin 2.6 shows a very good sorption selectivity for Mo(VI) with respect to V(V) and Re(VII), respectively (see Table VI).

Elution Assays by Acid and Basic Media

From a practical point of view, besides the high performances in the adsorption of metal ions, the adsorbed metal ions should be eluted rapidly and quantitatively. Thus, the elution of V(V), Mo(VI), and Re(VII) was examined. Table VII gives results for batchwise elution of these ions adsorbed on the resin CAOTM (1.4, 1.5, and 1.6) and the resin CMATA (2.4, 2.5, and 2.6) with hydrochloric acid, sulfuric acid, and sodium carbonate solutions of three different concentrations. In general,

in acidic mediums, Mo(VI) and Re(VII) are better eluted at higher concentrations (2 and 3*M*) of HCl and H_2SO_4 , the elution being quantitative for some resins.

Mo(VI) forms more stable complexes at basic medium, and therefore, this metal ion is highly eluted in comparison with the elution in acid media.

These results suggest a possible separation of metal ions depending on the eluent used. Thus, it should be possible to recover the loaded resin 1.4 by treatment with $1M$ H₂SO₄ [100% elution of V(V)] and with $0.50M$ Na_2CO_3 [97% elution of Mo(VI)]. In a similar way, it should be possible to separate $V(V)$ from the Mo(VI) in the 1.6-loaded resin.

CONCLUSIONS

From CAOTM and CMATA crosslinked with MBA, chelating resins with methylsulfate or chloride ion exchanger groups were derived. The adsorption ability for ions was influenced by the swelling ratio. The resins showed high efficiency to adsorb $V(V)$, Mo(VI), and Re(VII) ($>75\%$) at pH $=$ 3.0 and an excellent selectivity from either individual metal ion solutions or binary mixtures.

Table VI Retention (%) of the Different Metal Ions at pH 1 from Binary Mixtures

Resin	Binary Mixtures									
	V(V)	Mo(VI)	V(V)	Re(VII)	Mo(VI)	Re(VII)				
1.4		94	6	94	60	40				
1.6		93	8	92	62	38				
2.4		98	43	57	74	26				
2.6	16	84	30	70	82	18				

		Eluent								
Resin		HC1			H_2SO_4			Na ₂ CO ₃		
	Resin + Metal Ion	1M	2M	3M	1M	2M	3M	0.25M	$0.50M\,$	1M
1.4	$1.4 + V(V)$	58	80	78	100	98	100	55	89	74
1.5	$1.5 + V(V)$	16	74	77	49	84	89			
1.6	$1.6 + V(V)$	60	76	79	96	96	95	50	63	80
2.2	$2.2 + V(V)$	57	79	76	93	85	96	59	68	73
2.4	$2.2 + V(V)$	66	88	86	94	100	100	66	76	82
$2.6\,$	$2.6 + V(V)$	72	86	82	100	100	100	65	79	79
1.4	$1.4 + Mo(VI)$	48	63	65	9	45	64	84	97	97
1.5	$1.5 + Mo(VI)$									
1.6	$1.6 + Mo(VI)$	36	63	64	$\sqrt{3}$	20	49	100	100	100
2.2	$2.2 + Mo(VI)$									
2.4	$2.4 + Mo(VI)$	68	68	64	67	78	72	74	93	90
2.6	$2.6 + Mo(VI)$	64	67	68	70	70	72	83	83	77
1.4	$1.4 + Re(VII)$									
1.5	$1.5 + Re(VII)$									
1.6	$1.6 + Re(VII)$	51	57		56	54		60	64	60
2.2	$2.2 + Re(VII)$									
2.4	$2.4 + Re(VII)$									
2.6	$2.6 + Re(VII)$	55	53		50	54		36	65	64

Table VII Elution of the Metal Ions by HCl, H₂SO₄, and Na₂CO₃ at Different Concentrations

It is also possible to separate selectively metal ions changing the eluent medium. The resin– metal ion equilibrium was achieved rapidly ≤ 1 h), considering that it occurs in a heterogeneous phase. The process is relatively slow for the resin containing methylsulfate as anion in respect to that containing the chloride group.

The metal ion–loaded resins showed a higher thermal stability than those unloaded resins, particularly between 400 and 500°C, due to the effect of a crosslinking point of the metal ion on the resin.

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