Amberlite XAD-2 impregnated Cyanex272: sorption desorption studies of thorium(IV)

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Summary. Amberlite XAD-2 impregnated with Cyanex272 was packed in a column and studied for separation and recovery of Th(IV). The effect of various parameters influencing sorption of Th(IV) viz. acid concentration, aqueous phase flow rates, nature and concentration of eluents and other ions were systematically studied. Under optimized conditions Th(IV) was quantitatively sorbed from 0.001 M HNO₃ and recovered using 10 cm³ of 4.0 M HCl at 0.6 cm³ min⁻¹ flow rate. Batch studies using impregnated resin were evaluated for using Langmuir and Freundlich adsorption isotherms. The sorption capacity of the impregnated resin for Th(IV) was 8.48 mmol g⁻¹. The limit of detection for Th(IV) was 0.67 µg dm⁻³ and a reusability for more than 65 cycles was observed. The method developed was applied for isolation of Th(IV) from monazite sand. It was reproducible with a relative standard deviation (R.S.D.) of 0.8%.

1. Introduction

Solvent extraction has been commonly used for separation and determination of thorium owing to its industrial importance. However, solid phase extraction has emerged as an alternative to liquid-liquid extraction as it minimizes the large scale use of organic solvents and thus reduces the wastes and the problems associated with their disposal. Solid polymeric resin based specific ion collectors have made significant contribution for the detection of trace elements and several methods for preconcentration and determination of thorium exist [1]. Amberlite XAD series resins [2–7], Merrifield's chloromethylated resin [8], and silica gel [9] were chemically modified for separation and determination of thorium from soil, sediment [3, 8], and nuclear spent fuel sample [6, 7]. Solid phase extraction methodologies involving impregnation based upon physical interaction of a ligand on a solid support is a simple and easy process [10–16] and avoids the complexities involved in the chemical reaction for synthesis of ligand [5, 7, 8] and/or its binding to the solid support [2–7, 9]. Impregnating a liquid extractant on a solid support for recovery of valuable metals is

interesting [14, 15] as it not only combines the high distribution coefficients amenable with liquid extractants but also the multistage characteristic of chromatographic process. High sorption capacity and good enrichment factor was reported using Amberlite XAD-2 impregnated with Cyanex302 {bis(2,4,4-trimethylpentyl)monothiophosphinic acid} [15]. In the present work, XAD-2 was impregnated with Cyanex272 {bis(2,4,4-trimethylpentyl)phosphinic acid} to understand the sorption desorption characteristics of Th(IV) and facilitate its recovery from associated elements and monazite sand. Amberlite XAD-2 was selected as a solid support due to its characteristic physical properties, good mechanical stability, and structural rigidity (mean pore diameter 90 Å, 20–60 mesh, and average surface area 330 m² g⁻¹). The sorption desorption conditions were optimized by a column method, while a batch mode was used to study Langmuir and Freundlich adsorption isotherms.

2. Experimental

2.1 Instrumentation

A glass column of 150 mm length and 10 mm internal diameter was used for the packing of the solid phase. A wrist action flask shaker EXPO from General Trading Corporation, India was used for equilibration in batch method. Digispec-110D visible spectrophotometer was used for metal detection. A flame atomic absorption spectrophotometer (FAAS), AAnalyst200 (Perkin Elmer) and an inductively coupled plasma atomic emission spectrometer (ICP-AES), JY JOBIN YVON HORIBA ULTIMA 2 were also used.

2.2 Chemicals and reagents

A stock solution containing 2.5 mg cm⁻³ Th(IV) was prepared from Th(SO₄)₂·9H₂O (Loba Chemie). It was standardized complexometrically [16] and a working solution containing 20 µg cm⁻³ of Th(IV) was prepared by appropriate dilution. An aqueous solution of Arsenazo(III) (0.1% w/v) (S.d. Fine Chemicals) was used for spectrophotometric determination of Th(IV). Amberlite XAD-2 was purchased from Supelco, Sigma Aldrich Co. Cyanex272 was obtained as a gift sample from Cytec Canada. All chemicals and reagents used were of A.R. grade.

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2.3 Preparation of Cyanex272 impregnated Amberlite XAD-2 resin

Amberlite XAD-2 resin (100 mg) and Cyanex272 (100 mg) in 10 cm³ methanol were mixed together till it resulted in almost complete evaporation of methanol and was then kept overnight at room temperature. The impregnated resin was uniformly packed in a glass column (150 mm length and 10 mm internal diameter) with glass wool as support.

2.4 General procedure for sorption of Th(IV)

The column was preconditioned with 10 bed volumes of 0.001 M HNO₃. A 10.0 cm³ sample solution containing 2.0 µg cm⁻³ Th(IV) in 0.001 M HNO₃ was sorbed on the column at a flow rate of 1.0 cm³ min⁻¹ and then recovered with 10.0 cm³ of 4.0 M HCl at a flow rate of 0.6 cm³ min⁻¹. Th(IV) was determined spectrophotometrically using 0.1% Arsenazo(III) at 660 nm [17].

2.5 Procedure for the analysis of monazite sand

A 0.1 g sample of monazite sand was digested in concentrated H₂SO₄ at 250 °C [18] and after repeated extraction with 1 : 10 H₂SO₄ was finally diluted to a suitable volume. A known aliquot of sample was passed through the column as per general procedure.

3. Results and discussion

3.1 Column mode

Optimization for preconcentration and recovery of Th(IV)

Influence of amount of Cyanex272

Amberlite XAD-2 (100 mg) was impregnated with varying amounts of Cyanex272 (25–100 mg) to study sorption of Th(IV). Using Th(IV) (20 µg) in 0.001 M HNO₃ and passing through the column packed with different amounts of Cyanex272 at 1.0 cm³ min⁻¹ flow rate indicated that 100 mg of Cyanex272 was necessary to achieve quantitative sorption (99.3 ± 0.8) % of Th(IV). Lower sorption of Th(IV) using Cyanex272 less than 100 mg for impregnation is possibly due to incomplete pore filling of the resin and thus reducing the accessibility for complexation of Th(IV).

Effect of aqueous phase acidity and flow rate

Sorption of Th(IV) was studied at different aqueous phase acidities (Fig. 1). Th(IV) sorption was quantitative from 0.001 M HNO₃ and 0.001 M HCl, presumably due to sorption on Cyanex272 as a cationic species. Further studies were carried out using 0.001 M HNO₃ as the aqueous phase. Aqueous phase flow rate was optimized by passing a sample solution (10.0 cm³) containing 2.0 µg cm⁻³ Th(IV) in 0.001 M HNO₃ through the column at (0.5–5.0) cm³ min⁻¹ flow rate. Sorption of Th(IV) was quantitative with a flow rate of 1.0 cm³ min⁻¹. While it was (98.7 ± 0.2) % and (96.3 ± 0.2) %, using sample flow rates 2.0 cm³ min⁻¹ and 5.0 cm³ min⁻¹ respectively due to its insufficient contact with the impregnated resin. Thus, a flow rate of 1.0 cm³ min⁻¹ was used in all studies.

Fig. 1. Effect of aqueous phase acidity on sorption of Th(IV). Experimental condition: column packed with 100 mg Amberlite XAD-2 impregnated with 100 mg Cyanex272. Aqueous phase condition = 20 µg Th(IV) in 10.0 cm³, at 1.0 cm³ min⁻¹ flow rate.

Breakthrough volume

Different sample volumes (10–1000) cm³ containing 20 µg Th(IV) were passed through the column packed with the impregnated resin at the optimum flow rate. Using these sample solutions not more than 0.006 ± 0.006 µg cm⁻³ Th(IV) was detected in the aqueous solution upto 1000 cm³. Therefore, the preconcentration factor of 100 was achieved by using 10.0 cm³ of 4.0 M HCl as eluting agent.

Influence of various eluting agents on desorption of Th(IV)

Recovery of Th(IV) was studied using different eluents (Table 1). Higher concentrations of HNO₃ and H₂SO₄ were not studied as preliminary experiments indicated leaching of the extractant from the impregnated resin. Recovery of Th(IV) was quantitative (99.3 ± 0.8) % with 4.0 M HCl and 2.0 M H₂SO₄. However, 4.0 M HCl was selected as an eluent. Different volumes (5.0–20.0) cm³ and flow rates (0.2–1.0) cm³ min⁻¹ of 4.0 M HCl were studied. Using 5.0 cm³ of HCl at flow rate 0.6 cm³ min⁻¹, Th(IV) recovery was 91.5 ± 0.3% and with 10.0 cm³ of 4.0 M HCl at 0.8 cm³ min⁻¹ flow rate its recovery was of 97.3 ± 0.2%. Eluent selected for further studies was 10.0 cm³, 4.0 M HCl at 0.6 cm³ min⁻¹ flow rate.

Column bed reusability

The stability and potential reusability of the column was assessed by monitoring the sorption of Th(IV) through a number of sorption desorption cycles under the optimum conditions. The sorption of Th(IV) was (99.8 ± 0.3) % and it remained unchanged even after continuous usage upto 65 cycles (Fig. 2) indicating a very high reusability and reliability of the resin.

Effect of diverse ions on sorption of Th(IV)

The influence of alkali, alkaline earths, transition metals and rare earth elements on the sorption of Th(IV) was studied under its optimum conditions. The tolerance limit was set as the amount of foreign ion required to cause ±2.0%

Table 1. Influence of eluting agents (aqueous phase condition = 20 µg Th(IV) in 10.0 cm³ of 0.001 M HNO₃, at 1.0 cm³ min⁻¹ flow rate, elution condition = 10.0 cm³, at 0.6 cm³ min⁻¹ flow rate).

Conc. [M] → Eluents ↓	0.01	0.1	0.5	1.0 % Recovery ^{±a}	2.0	3.0	4.0
HCl	10.4 ± 0.3	45.4 ± 0.6	56.3 ± 0.4	66.0 ± 0.2	90.7 ± 0.3	96.4 ± 0.2	99.5 ± 0.2
HNO ₃	9.4 ± 0.2	23.8 ± 0.5	50.4 ± 0.3	73.9 ± 0.2	—	—	—
H ₂ SO ₄	47.6 ± 0.3	72.2 ± 0.2	90.9 ± 0.2	96.3 ± 0.3	99.2 ± 0.3	—	—
HClO ₄	7.2 ± 0.2	38.7 ± 0.4	67.6 ± 0.6	78.1 ± 0.2	88.0 ± 0.3	89.4 ± 0.2	92.9 ± 0.5
CH ₃ COOH	1.4 ± 0.2	3.3 ± 0.2	7.7 ± 0.3	18.0 ± 0.6	27.9 ± 0.7	35.6 ± 0.4	44.4 ± 0.3
H ₂ C ₂ O ₄	60.3 ± 0.3	81.9 ± 0.4	87.5 ± 0.5	92.8 ± 0.3	—	—	—
(NH ₄) ₂ CO ₃	3.4 ± 0.2	15.9 ± 0.5	63.4 ± 0.2	56.8 ± 0.4	—	—	—

a = % R.S.D. (N = 3).

Fig. 2. Column bed reusability. Experimental condition: column packed with 100 mg Amberlite XAD-2 impregnated with 100 mg Cyanex272. Aqueous phase condition = 20 µg Th(IV) in 10.0 cm³ of 0.001 M HNO₃, at 1.0 cm³ min⁻¹ flow rate. Elution condition = 10.0 cm³ of 4.0 M HCl, at 0.6 cm³ min⁻¹ flow rate.

change in the recovery of Th(IV). The tolerances for alkali and alkaline earth elements using 20 µg of Th(IV) were 100 µg for sodium(I), 250 µg for potassium(I) and magnesium(II), and 500 µg for calcium(II). Iron(III), nickel(II), lanthanum(III), and uranium(VI) showed strong interferences as they were tolerated in the ratios 1 : 1, 1 : 1, 1 : 1.5, and 1 : 1.5 respectively. Certain elements like lead(II), yttrium(III), neodymium(III), samarium(III), and dysprosium(III) had good tolerances with tolerance ratios 1 : 5, 1 : 7.5, 1 : 12.5, 1 : 5, and 1 : 5, respectively. Various anions like Cl⁻, NO₃⁻, SO₄²⁻ showed high tolerance limits for Th(IV). Binary mixtures were proposed for separation of Th(IV) from other elements. The resulting separation factors ($\beta = D_{\text{Th}} : D_{\text{M}}$) were good (Table 2). As the other element remained in the aqueous phase and was determined quantitatively.

3.2 Batch mode

Sorption capacity of impregnated resin

A 20.0 cm³ solution containing (1.0–10.0) mg Th(IV) in 0.001 M HNO₃ was shaken for a predetermined time of 20 min. in a stoppered bottle on a mechanical shaker. After phase separation, Th(IV) was determined in the aqueous phase spectrophotometrically. The sorption capacity was defined as the maximum amount of Th(IV) adsorbed on the

Table 2. Separation of Th(IV) from binary mixtures (aqueous phase condition = 20 µg Th(IV) in 10.0 cm³ of 0.001 M HNO₃, at 1.0 cm³ min⁻¹ flow rate, elution condition = 10.0 cm³ of 4.0 M HCl, at 0.6 cm³ min⁻¹ flow rate).

Metal ion	Amount of metal added (µg)	Metal un-extracted (%) ^{±a}	Recovery of Th(IV) (%) ^{±a}	Separation factor (β) $\beta = D_{\text{Th}} : D_{\text{M}}$
Na(I)	100	99.8 ± 0.1	98.9 ± 0.2	55.9 × 10 ³
K(I)	250	99.0 ± 1.7	99.0 ± 0.2	10.7 × 10 ³
Ca(II)	500	99.8 ± 0.2	99.4 ± 0.4	71.2 × 10 ³
Mg(II)	250	99.1 ± 0.7	99.2 ± 0.2	14.7 × 10 ³
Fe(III)	20	87.9 ± 2.0	98.8 ± 0.4	11.9 × 10 ²
Co(II)	100	98.9 ± 0.1	99.6 ± 0.2	22.7 × 10 ³
Ni(II)	20	99.0 ± 0.2	99.0 ± 0.2	90.6 × 10 ²
Pb(II)	100	99.0 ± 0.1	99.4 ± 0.3	16.7 × 10 ³
Y(III)	150	99.2 ± 0.3	99.1 ± 0.3	17.6 × 10 ³
La(III)	30	98.5 ± 0.2	98.8 ± 0.3	94.0 × 10 ²
Ce(IV)	60	98.7 ± 0.3	99.4 ± 0.3	10.4 × 10 ³
Pr(III)	40	99.6 ± 0.2	99.2 ± 0.2	33.3 × 10 ³
Nd(III)	250	99.0 ± 0.2	99.0 ± 0.2	18.4 × 10 ³
Sm(III)	100	99.4 ± 0.3	99.2 ± 0.2	21.9 × 10 ³
Gd(III)	40	99.7 ± 0.1	98.7 ± 0.2	44.4 × 10 ³
Dy(III)	100	99.5 ± 0.2	99.3 ± 0.2	26.3 × 10 ³
U(VI)	30	83.1 ± 0.3	99.3 ± 0.2	69.6 × 10 ¹

a: = % R.S.D. (N = 3).

impregnated resin at equilibrium, under its optimum conditions for sorption. Amberlite XAD-2 resin impregnated with Cyanex272 had a sorption capacity of 8.48 mmol g⁻¹.

Adsorption isotherms for Th(IV) on impregnated resin

100 mg Amberlite XAD-2 impregnated with 100 mg Cyanex272 and 20.0 cm³ of 0.001 M HNO₃ solutions containing (50–500) mg dm⁻³ of Th(IV) were shaken in a stoppered bottle on a mechanical shaker for 20 min. The modified support was filtered and the residual solution was analyzed for Th(IV) spectrophotometrically. The experimental data for adsorption isotherm was treated for Langmuir and Freundlich adsorption isotherm models. The rearranged Langmuir equation [19] is as follows:

$$\frac{C_e}{q_e} = \frac{1}{q_0 b} + \frac{C_e}{q_0} \quad (1)$$

C_e is the equilibrium concentration of Th(IV) (mg dm⁻³), q_e is its amount adsorbed at equilibrium and q_0 and b are Lang-

Fig. 3. Langmuir adsorption isotherm. Experimental condition: 100 mg Amberlite XAD-2 impregnated with 100 mg Cyanex272. Aqueous phase condition = (50–500) mg dm⁻³ Th(IV) in 20.0 cm³ of 0.001 M HNO₃, shaking time = 20 min.

Fig. 4. Freundlich adsorption isotherm. Experimental condition: 100 mg Amberlite XAD-2 impregnated with 100 mg Cyanex272 aqueous phase condition = (50–500) mg dm⁻³ Th(IV) in 20.0 cm³ of 0.001 M HNO₃, shaking time = 20 min.

muir constants related to adsorption capacity and energy of adsorption, respectively.

While, Freundlich equation [19] is represented as follows:

$$\log(x/m) = \log K_f + \log C_e(1/n) \quad (2)$$

x/m is the amount of metal adsorbed per 100 mg impregnated resin, K_f the Freundlich constant is the measure of adsorption capacity, and $1/n$ is adsorption intensity. The plots of C_e/q_e vs. C_e and $\log(x/m)$ vs. $\log C_e$ indicated that the adsorption of Th(IV) on Cyanex272 impregnated on Amberlite XAD-2 resin obeyed Langmuir as well as Freundlich adsorption isotherm models (Figs. 3 and 4). The correlation coefficient (R^2) for the linear regression fit of the Langmuir plot as well as Freundlich plot were 0.986 and 0.998 respectively while the values for q_0 , b , K_f , and n were 3.93, 0.16, 1.50 and 5.93 respectively.

Precision and detection limit

Th(IV) determination using the same column in six replicate determinations had a relative standard deviation (R.S.D.) of 0.8%. The limit of detection (LOD) evaluated, as three

Table 3. Separation of Th(IV) from synthetic mixtures (aqueous phase condition = 20 µg Th(IV) in 10.0 cm³ of 0.001 M HNO₃, at 1.0 cm³ min⁻¹ flow rate, elution condition = 10.0 cm³ of 4.0 M HCl, at 0.6 cm³ min⁻¹ flow rate).

Composition of synthetic mixture	Recovery of Th(IV) (%) ^{±a}
Ca(II); 10 µg, Pb(IV); 10 µg, Th(IV); 10 µg	99.3 ± 0.4
Ca(II); 2.5 µg, Ce(IV); 12.5 µg, Sm(III); 17.5 µg, Gd(III); 17.5 µg, Nd(III); 10.0 µg, Th(IV); 20 µg	98.6 ± 0.1
Ca(II); 20 µg, La(III); 7.5 µg, Ce(IV); 17.5 µg, Nd(III) 5.0 µg, Th(IV); 20 µg	98.8 ± 0.4
Na(I); 10 µg, Sr(II); 30 µg, Th(IV); 20 µg	99.3 ± 0.2
Ba(II); 95 µg, K(I); 1.0 µg, Na(I); 1.0 µg, Sr(II); 5.0 µg, Ca(II); 2.5 µg, La(III); 22.5 µg, Ce(IV); 10 µg, Nd(III); 2.5 µg, Th(IV); 20 µg	99.1 ± 0.3

a: = % R.S.D. ($N = 3$).

times the standard deviation of the blank signal [19] was 0.67 µg dm⁻³.

4. Analytical applications

4.1 Separation of Th(IV) from synthetic mixtures

The method developed was applied to separate and recover Th(IV) from synthetic mixtures containing elements commonly associated with it. The recovery of Th(IV) was quantitative with a R.S.D. not more than 0.8% (Table 3). The other elements were not sorbed under the optimum conditions for sorption of Th(IV) and were quantitatively determined in the aqueous phase by FAAS or ICP-AES.

4.2 Recovery of Th(IV) from monazite sand

The applicability of the method developed was checked using an aliquot of sample prepared as described in experimental section 2.5. The amount of Th(IV) detected in monazite sand was 114.98 ± 0.28 mg g⁻¹ as against 115.91 mg present in it. This result using sample solution was cross-checked by ICP-AES analysis and corresponded to a (99.2 ± 0.3) % recovery for Th(IV) and had R.S.D. within 0.8%.

5. Conclusion

The separation and determination of Th(IV) using Amberlite XAD-2 resin impregnated with Cyanex272 is a simple, selective, and specific method to isolate traces of Th(IV) from associated elements. The sorption capacity for Th(IV) was superior [4–8, 10, 12], reusability good and LOD low [4–10, 15] in comparison to other solid phase extraction methods. The method developed was successfully applied for recovery of Th(IV) from monazite sand.

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