Adsorption characteristics of thorium on silica-based anion exchange resins (Postprint)

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Abstract

To isolate and separate thorium from nitric acid solutions, three silica-based anion exchange resins were synthesized. Batch experiments were carried out to investigate adsorption behavior of thorium in nitric acid solutions. Adsorption at different concentrations of nitric acid and thorium, influence of contact time and coexisting metal ions, and effect of NO3- were investigated in detail. It was found that at high HNO3 concentrations, the resins exhibited higher adsorption capacity and better affinity towards thorium. The adsorption kinetics could be described by the pseudo-second order model equation, while the adsorption isotherms were well correlated by the Langmuir model. The maximum capacity towards thorium species on SiPyR-N4 was evaluated at 27–28 mg/g-resin. The thermodynamic parameters indicated the adsorption was an exothermic reaction. The presence of NO3- was found to promote the retention of the thorium species.

Full Text

Preamble

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Adsorption Characteristics of Thorium on Silica-Based Anion Exchange Resins

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Abstract: To isolate and separate thorium from nitric acid solutions, three silica-based anion exchange resins were synthesized. Batch experiments were carried out to investigate the adsorption behavior of thorium in nitric acid solutions, examining the effects of nitric acid concentration, thorium concentration, contact time, coexisting metal ions, and nitrate ions. The resins exhibited higher adsorption capacity and better affinity towards thorium at high HNO_3 concentrations. The adsorption kinetics followed a pseudo-second-order model, while the adsorption isotherms were well described by the Langmuir model. The maximum capacity for thorium species on SiPyR-N4 was evaluated at 27–28 mg/g-resin. Thermodynamic parameters indicated that the adsorption was an exothermic reaction, and the presence of NO_3^- was found to promote the retention of thorium species.

Keywords: Thorium, Silica, Anion exchange resin, Adsorption
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Introduction

Almost all nuclear reactors currently operate using uranium-based fuel with very limited fuel recycling, which is not sustainable given the world's increasing energy demands. Thorium is 3–4 times more abundant than uranium, prompting significant research into its use in CANDU reactors, light water reactors (LWRs), and molten salt reactors. The thorium fuel cycle offers advantages including smaller initial fuel inventory and reduced radioactive waste formation. India has been particularly active in developing thorium-based technology, motivated by limited uranium reserves and large thorium deposits. However, practical use of thorium as a nuclear fuel requires effective reprocessing methods.

Similar to the UREX and PUREX processes, the THOREX process was developed for the thorium fuel cycle, utilizing tri-n-butyl phosphate (TBP) to extract thorium and uranium. However, THOREX has significant drawbacks including large waste volumes and the need for large-scale extraction equipment, necessitating the development of advanced fuel cycle processes with more compact equipment and reduced radioactive waste for efficient utilization of thorium-based fuels.

Ion exchange technology has been applied to spent nuclear fuel reprocessing for over 70 years due to its simple operation, organic solvent-free nature, and compact equipment requirements. Conventional organic ion exchangers suffer from poor radiation resistance and slow adsorption rates, which led to the development of silica-based ion exchangers featuring fast retention kinetics, excellent radiation resistance, and low pressure drop in packed columns. These materials have demonstrated superior performance in recovering uranium, plutonium, and neptunium from spent nuclear fuels, and show strong affinity for specific fission products such as palladium, ruthenium, and technetium. They can also be used to adsorb and separate rare earth elements. The silica-based anion exchangers include the weak-base anion exchange resin (SiPyR-N3), strong-base anion exchange resin (SiPyR-N4), and resin with complex functional groups (AR-01). The structures of these functional groups are shown in Fig. 1 [Figure 1: see original paper].

The feasibility of adsorbing thorium with anion exchangers in hydrochloric, nitric, and sulfuric acid media was investigated decades ago. Commercial anion exchangers such as Dowex-1 and Dowex-2 exhibit good adsorption and separation properties for thorium. However, silica-based anion exchange resins have not been extensively applied to thorium recovery. This paper reports our investigation of thorium adsorption behavior from nitric acid solutions using silica-based anion exchangers, with the aim of enabling large-scale applications in the thorium fuel cycle for power generation.

Experimental

A. Materials

Silica particles with a mean pore size of 600 nm and particle size of 37–74 µm were used as the base material for resin synthesis. Two monomers were provided by J&K Scientific Ltd.: DVB (divinyl benzene) as an m- and p-mixture at 55% purity, mixed with EVB (ethyl-vinyl benzene) and DEB (diethyl benzene) and stabilized with TBC (tert-butyl-catechol), with the polymerization retarder washed by sodium hydroxide solution; and 4VP (4-vinylpyridine) purified to 95% purity through vacuum distillation and stabilized with TBC. Acetophenone and diethyl-o-phthalate (analytical reagent grade, Sinopharm Chemical Reagent Co., Ltd.) were used as solvents. AIBN (α,α‘-azobisisobutyronitrile, chemical pure grade, Tianjin Guangfu Fine Chemical Research Institute) and ACCN (1,1’-azobiscyclohexane-1-carbonitrile, 98% purity, Sigma-Aldrich Co., Ltd) served as initiators. $Th(NO_3)_4$ was obtained from Aladdin Chemistry Co., Ltd. Other nitrates and HNO_3 (all analytical grade) were from Sinopharm Chemical Reagent Co., Ltd. Ruthenium(III) nitrosyl nitrate (1.5 wt% Ru in dilute HNO_3) was from Sigma-Aldrich Co., Ltd. De-ionized water (18 MΩ) was used in all experiments.

B. Synthesis of the Resins

After washing with hydrochloric acid and vacuum drying, the silica particles were placed in a rotary evaporator flask connected to a vacuum pump. A mixture of monomers (85 wt.% 4VP and 15 wt.% DVB), initiators (AIBN and ACCN), and diluents (acetophenone and diethyl-o-phthalate) was drawn into the flask, which was rotated slowly to ensure complete filling of the silica pores. Under nitrogen protection, the flask was gradually heated to 363 K in a silicone-oil bath and rotated at this temperature for 20 hours to polymerize the monomers. The synthesized SiPyR-N3 resin was washed sequentially with water and acetone several times.

To investigate pore size effects on resin properties, silica particles with a mean pore size of 50 nm were also used to synthesize SiPyR-N3. SiPyR-N4 was obtained by converting SiPyR-N3 to quaternary ammonium through reaction with dimethylsulphate. AR-01 was synthesized similarly.

The synthesized resins were characterized by FTIR analysis (Shimadzu IRAffinity-1 with ATR attachment), TG-DTA analysis on a thermal gravimetry analyzer (Shimadzu T-60) under oxygen atmosphere (heated from 30 °C to 550 °C at 1 °C/min), and scanning electron microscopy (Sirion 200). Surface area and pore size were measured by Accelerated Surface Area and Porosimetry System (ASAP 2020). Exchange capacity was determined by titration.

C. Batch Experiments

Batch experiments of thorium adsorption were conducted in a thermostatic air shaker to study the effects of acidity and initial thorium concentration, adsorption kinetics, and retention performance compared with coexisting metal ions. Resins were preconditioned to the NO_3^- form at different concentrations. Each resin type was packed into a sand core funnel connected with a dropping funnel, and nitric acid was added dropwise. Samples were then transferred to a sand core funnel to evacuate the solution and vacuum-dried.

The equilibrium adsorption capacity (Q , mmol/g) and distribution coefficient (K_d, cm^3/g) were calculated by:

$Q\ = (C_0 - C\ )V/(M \cdot m)$
K_d = $(C_0 - C\ )V/(C\ \cdot m)$

where C_0 and C denote the initial and equilibrium concentrations in the aqueous phase (mg/L), respectively; M is the molar mass of Th; m is the mass of resin (g); and V is the volume of aqueous phase in batch experiments (cm^3).

1. Influence of Nitric Acid Concentration In nuclear fuel reprocessing, spent fuel is dissolved in nitric acid. Since thorium adsorption behavior onto anion exchange resins may change significantly with HNO_3 concentration, this effect requires investigation. Industrial nuclear fuel reprocessing typically uses 3–6 M HNO_3. Therefore, batch experiments were carried out in HNO_3 solutions ranging from 0.1–9 M. The initial thorium concentration was 10 mM and contact time was 2 hours.

2. Adsorption Kinetics To investigate adsorption kinetics, experiments were performed with contact times from 5 minutes to 2 hours, ensuring equilibrium was attained. The HNO_3 concentration was 3 M and initial thorium concentration was 10 mM.

3. Adsorption Isotherm To determine thorium adsorption capacity, isotherms were obtained through batch experiments with initial thorium concentrations ranging from 1 mM to 40 mM, where saturation should be attained. The HNO_3 concentration was 3 M and contact time was 2 hours.

4. Influence of Nitrates To understand the adsorption mechanism, nitrate salts were added to investigate their effect on thorium retention. Previous studies have shown that nitrate salts enhance uranium uptake onto anion exchange resins with effectiveness in the order: $Al(NO_3)_3 > Ca(NO_3)_2 > LiNO_3 > NH_4NO_3 > HNO_3$, and that NH_4NO_3 and $Mg(NO_3)_2$ can effectively improve thorium adsorption. Therefore, $NaNO_3$ and $Mg(NO_3)_2$ were used to study their influence, with concentrations limited to below 3 M due to solubility constraints. The initial thorium concentration was 10 mM and contact time was 2 hours.

5. Adsorption Thermodynamics To investigate temperature effects, batch experiments were conducted at 25 °C, 30 °C, 35 °C, 40 °C, and 45 °C, with a contact time of 2 hours, HNO_3 concentration of 9 M, and initial Th concentration of 10 mM.

6. Separation of Thorium from Accompanying Metals To apply silica-based anion exchange resins in the thorium fuel cycle, it is necessary to investigate separation of thorium from coexisting metal ions in nitric acid solutions, including representative fission products such as ruthenium, palladium, strontium, zirconium, and rare earth metals. HNO_3 concentration ranged from 0.1 M to 6 M to optimize separation conditions. The initial metal ion concentration was 10 mM and contact time was 2 hours.

Throughout all batch experiments, the air shaker was maintained at 298 K, the phase ratio was 0.1 g resin to 5 mL solution, and samples were shaken at 120 r/min. After adsorption, phase separation was achieved using membrane filters with 0.45 µm mean pore size. Thorium concentrations in the aqueous phase before and after adsorption were determined using ICP-AES spectrometry (Shimadzu ICP-7510).

D. Desorption Experiment

Under optimal thorium adsorption conditions, resins at adsorption equilibrium were dried, and the amount of thorium adsorbed was calculated from concentration differences. The resins and eluent were mixed at a phase ratio of 0.1 g resin to 5 mL eluent, at 298 K with shaking at 120 r/min. The thorium concentration was detected by ICP-AES and the elution rate was calculated.

Results and Discussion

A. Characterization of the Silica-Based Anion Exchange Resins

SEM images of the SiPyR-N4 resin (Fig. 2 [Figure 2: see original paper]) reveal an obvious porous structure, confirmed by BET analysis. SEM images of SiPyR-N3 (Fig. 3 Figure 3: see original paper) and AR-01 (Fig. 3(b)) show similar porous structures, though SiPyR-N3 has much smaller pores. In the synthesis procedure, SiPyR-N3 was fabricated using silica particles with a mean pore size of 50 nm, significantly smaller than the 600 nm particles used for the other resins under otherwise identical conditions. FTIR, TG-DTA, titration, and BET analyses revealed that the resins have characteristics similar to those previously reported. Main physical and chemical parameters are listed in Table 1 [15–17].

B. Batch Adsorption Experiments

1. Effect of HNO_3 Concentration Th^{4+} hydrolysis may occur at low nitric acid concentrations, which would interfere with the adsorption process. Previous reports indicate significant Th^{4+} hydrolysis at $pH > 2$, as described in Eq. (3) [22]. In this work, HNO_3 concentrations of 0.1–9 M were used, where thorium was expected to exist as Th^{4+} or $[Th(NO_3)\ ]^{4-n}$ ligand complexes.

As shown in Fig. 4 [Figure 4: see original paper], HNO_3 concentration strongly affects thorium adsorption. At low acidity, the resins adsorb little thorium because most thorium ions exist as cations (Th^{4+}, $[Th(NO_3)]^{3+}$, $[Th(NO_3)_2]^{2+}$, $[Th(NO_3)_3]^{+}$) with no affinity for anion exchange resins. With increasing NO_3^- concentration, thorium ions acquire more ligands and form $[Th(NO_3)_5]^-$ or $[Th(NO_3)_6]^{2-}$. Spectrophotometric studies have shown these complexes can be adsorbed onto anion exchange resins [23], and batch experiments at higher acidity confirmed this, with optimal adsorption occurring in 9 M HNO_3. For practical application in nuclear fuel cycle processes, subsequent adsorption kinetics and isotherm experiments used 3 M HNO_3.

2. Adsorption Kinetics As shown in Fig. 5 Figure 5: see original paper, adsorption reaches equilibrium quickly, indicating notable adsorption kinetics. The pseudo-first-order kinetics model (Eq. (1)) and pseudo-second-order kinetics model (Eq. (2)) were used to evaluate the adsorption mechanism:

$$q\ = q\ (1–e^{-\ 1}\ )$$
$$q\ = (k_2q\ ^2t)/(1 + k_2q\ t)$$

where q (mg/g) is the adsorption quantity at any time before equilibrium, q (mg/g) is the equilibrium adsorption quantity, and k_1 and k_2 are rate constants.

Figures 5(a) and 5(b) show fitting curves for both models, with parameters listed in Table 2 . The correlation coefficients (R^2) indicate that the pseudo-second-order model fits thorium adsorption better, suggesting that thorium is adsorbed in anionic form through a chemical reaction process. SiPyR-N3 had the slowest adsorption rate, requiring about 30 minutes to reach equilibrium, while SiPyR-N4 and AR-01 needed only about 10 minutes. This difference is attributed to SiPyR-N3's smaller pore size, demonstrating that larger resin pores promote thorium anion flow and diffusion, resulting in faster adsorption.

3. Adsorption Isotherm Results are shown in Fig. 6(a) [Figure 6: see original paper]. The Langmuir equation was used as a theoretical isotherm model to calculate maximum capacity. Assuming monolayer adsorption on a homogeneous surface [24], the model is described as:

$$C\ /Q\ \ = C\ /Q\ \ \ + 1/(K\ Q\ \ )$$

where C (mmol/L) is the equilibrium thorium concentration in the aqueous phase, Q (mmol/g) and Q (mmol/g) are the equilibrium and saturation adsorption quantities, respectively, and K (L/mmol) is the Langmuir constant related to adsorption energy.

Figure 6(b) shows linear Langmuir adsorption isotherms for the silica-based anion exchange resins, with key parameters given in Table 3 . The R^2 values indicate that thorium adsorption follows the Langmuir model, suggesting monolayer adsorption controlled by homogeneous active sites. SiPyR-N4 exhibits the highest thorium adsorption capacity at 27–28 mg/g-resin.

4. Influence of Nitrates Figure 7 [Figure 7: see original paper] shows thorium adsorption behavior in 3 M and 6 M HNO_3 solutions with varying $NaNO_3$ concentrations. $NaNO_3$ clearly promotes thorium adsorption. Figure 8 [Figure 8: see original paper] shows thorium adsorption in 0.1 M HNO_3 with different $Mg(NO_3)_2$ concentrations, with HNO_3 concentration effects from Fig. 4 shown as dotted lines for comparison. The distribution coefficient increases with $Mg(NO_3)_2$ concentration, indicating effective promotion of thorium adsorption. At the same NO_3^- concentration, H^+ presence reduces thorium retention, consistent with literature results [23].

This phenomenon can be explained by the following dynamic equilibrium:

$$[Th(NO_3)_6]^{2-}\quad [HTh(NO_3)_6]^{-}\quad [H_2Th(NO_3)_6]$$
$$Th(NO_3)_4\quad [Th(NO_3)_5]^{-}\quad [Th(NO_3)_6]^{2-}$$

In solutions with constant H^+ concentration, adding $NaNO_3$ or $Mg(NO_3)_2$ increases NO_3^- concentration, thereby increasing thorium anion concentration as described in Eq. (8), promoting the anion exchange process. Thus, NO_3^- promotes thorium adsorption while H^+ decreases retention. While thorium adsorption is better in 9 M HNO_3, adding nitrates such as $NaNO_3$ or $Mg(NO_3)_2$ can achieve improved adsorption at relatively low acidity (3–6 M).

5. Adsorption Thermodynamics Standard ΔH^0 and ΔS^0 values for thorium anion exchange reactions were calculated from slopes and intercepts of the linear Van't Hoff equation (Eq. (9)), with results shown in Fig. 9 [Figure 9: see original paper]:

$$\ln K\_d = -\Delta H^0/(RT) + \Delta S^0/R$$

The Gibbs free energy ΔG^0 was calculated using Eq. (10):

$$\Delta G^0 = \Delta H^0 - T\Delta S^0$$

where R is the gas constant (8.314 J/(mol • K)), K_d is the distribution coefficient, and T is temperature (K). Thermodynamic parameters are given in Table 4 . Negative ΔG values indicate spontaneous adsorption processes, while negative enthalpy changes confirm that thorium adsorption is exothermic.

6. Adsorption of Thorium from Accompanying Metal Ions As shown in Fig. 10 [Figure 10: see original paper], among the eight metal ions tested, palladium exhibits the strongest affinity for the anion exchange resins, consistent with literature [13], making its presence detrimental to thorium adsorption. Therefore, palladium should be removed from solutions using SiPyR-N3 before thorium separation.

Figure 11 [Figure 11: see original paper] shows thorium adsorption in the presence of accompanying metal ions (excluding palladium) using SiPyR-N4. The resin demonstrates stronger thorium adsorption than other metal ions at high HNO_3 concentration, with AR-01 showing similar behavior. This indicates that thorium can be separated from accompanying metals at high nitric acid concentrations (6–9 M). Therefore, it is feasible to separate thorium from accompanying metal ions in HNO_3 solutions by first removing palladium with SiPyR-N3, then recovering thorium with SiPyR-N4 or AR-01.

C. Desorption Experiment

Using 0.1 M nitric acid as eluent, nearly all adsorbed thorium could be eluted within 5 minutes, with recovery approaching 100%. Thorium elution was quite easy, as reported in the literature [22].

Conclusion

Three types of macro-porous silica-based anion exchange resins were synthesized and applied for the first time to adsorb thorium from nitric acid solutions. The following conclusions can be drawn:

(1) The adsorption process follows monolayer adsorption on a homogeneous surface, obeying the pseudo-second-order kinetic equation.

(2) Among the three resins, SiPyR-N4 exhibits the best kinetics and greatest adsorption capacity.

(3) Thorium shows better affinity to the resins at higher nitric acid concentrations.

(4) NO_3^- promotes thorium adsorption while H^+ significantly decreases thorium retention.

(5) Thorium adsorption is an exothermic reaction.

(6) Using SiPyR-N3 and SiPyR-N4 sequentially, thorium can be separated from accompanying metals.

(7) Adsorbed thorium can be easily eluted with 0.1 M nitric acid solution.

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Note: Figure translations are in progress. See original paper for figures.