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Two-step separation of Th, La and Ba using combined chromatographic columns

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Abstract
A combined chromatographic columns including LN resin and P204 resin were fabricated to separate Th^{4+}, La^{3+} and Ba^{2+} ions with a series of parallel experiments at different acidity and initial concentrations. The results show that LNS resin can selectively adsorb Th^{4+} ions but not La^{3+} and Ba^{2+} ions in 1 mol/L HNO_3, and the separation factor of Th/La and Th/Ba can reach up to 8.260×10^4 and 8.224×10^5, respectively. The La^{3+} and Ba^{2+} ions can be separated by P204 resin at pH = 3 with a separation factor of 2.191×10^4. The successful separation and enrichment of La in this separation system provides a possibility for the separation of Ac, which is of great significance to TAT.

Keywords P204 resin · LN resin · Separation · Chromatographic column

Introduction

Nuclear medicine has great application value in both diagnosis and treatment [1–3]. Radionuclides used for diagnosis or treatment are typically attached to selective biomolecules (peptides, antibody fragments, or intact antibodies) to deliver imaging or therapeutic doses to targets in vivo (tumors or other tissues). Targeted alpha therapy (TAT) is a rapidly developing field that alpha radionuclides are used selectively to deliver radiation doses capable of killing cells to tumor sites [4]. Low penetration range (50–90 μm) and high linear energy transfer of α-particles (tens to hundreds of keV/μm) are able to destroy cancer cells to the maximum with minimal damage to surrounding healthy tissue. ^{225}Ac ($t_{1/2} = 9.92$ d) has significant potential as a TAT nuclide because its favorable half-life and decay cascade including multiple short-lived alpha nuclides [5, 6]. For example, ^{225}Ac can not only be directly used for therapy by binding with biological targeting vectors, but also the nuclide ^{213}Bi ($t_{1/2} = 45.6$ min) produced by ^{225}Ac decay can be used for targeted therapy through chelation or complexation in vivo. Currently, the main source of ^{225}Ac is the decay of the parent body ^{229}Th ($t_{1/2} = 7340$ a), which was obtained early from a ^{233}U reactor as fuel [7]. However, the global supply of ^{225}Ac created by decay does not exceed 63 GBq (1.7 Ci) per year, which cannot meet the current demand for ^{225}Ac [8]. Therefore, it is urgent to explore a new method of mass production of ^{225}Ac.

Different groups, including Los Alamos National Laboratory (LANL), have investigated alternative methods for producing ^{225}Ac [9–11]. One of the most promising pathways is thought to be the $^{232}Th(p, x)$ nuclear reaction via the activation of ^{232}Th by a medium-energy (> 70 MeV) proton. Data obtained by LANL indicate that irradiation of ^{232}Th with a proton of 250 μA, 100 MeV energy for 10 days yields ^{225}Ac of 73.2 GBq (1.98 Ci) [9]. This amount exceeds the accumulation of ^{225}Ac prepared by ^{229}Th by research departments around the world. Although significant quantities of ^{225}Ac can be produced from the $^{232}Th(p, x)$ nuclear reaction, separating actinium from irradiated thorium targets is a challenge. The system of $^{232}Th(p, x)$ nuclear reaction mainly contains a large amount of Th, minor amounts of Ac and Ra, and trace amounts of other radioactive elements [9]. On the one hand, the concentration difference of thorium and actinium is several orders of magnitude, because thorium and actinium are the relationship between the decay parent and the second generation of the child. On the other hand, thorium, also an actinide element, has similar chemical properties to actinium. Therefore, it is necessary to seek

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an efficient and rapid method for the chemical separation and purification of Th, Ac and Ra, which not only provides a basis for the research and development of TAT, but also has great significance for the development of radionuclide drugs. The methods for separating Ac contain precipitation method [12], liquid–liquid extraction method [13, 14], extraction column [11, 15], ion exchange method [7], adsorption method [16] and so on. Compared to other separation techniques, chromatographic column separation has the high efficiency and speed of column separation [17]. Here, a combined chromatographic column was constructed to achieve Th separation and Ac enrichment by a two-step separation method [18]. Since actinium and radium are highly radioactive and difficult to obtain, Ac and Ra can be replaced by La and Ba with similar properties in the reported literature [19]. V. Ostapenko et al. found that commercial series LN resin had excellent separation effect on rare earth elements and actinides [20]. In addition, P204 resin can achieve the separation of Sr and Y, so it is speculated that it also has the separation effect on La and Ba of the same main group [21, 22].

This paper describes the separation of Th^{4+}, La^{3+} and Ba^{2+} ions using a combine chromatographic column with LN resin and P204 resin. The advantages of this separation are that the effect of concentration ratio on separation is investigated, which has not been seen in previous articles, meanwhile the three-ion separation can be accomplished in a simple process. The novelty of this paper mainly lies in the establishment of a set of separation system by which Ac can be conveniently and quickly separated and enriched from Th and Ra.

Experiment

Materials

The thorium standard solution was obtained from Beijing North Weiye Metrology Technology Research Institute. The single-element standard solution of lanthanum and barium was purchased from the National Nonferrous Metals and Electronic Materials Analysis Center. LN series resin was provided by TrisKem International. P204 resin was supplied by Lanzhou University. Other reagents were purchased from Tianjin Damao Chemical Reagent Factory. All reagents are analytical reagent grade and can be used directly without further purification. All tests were performed using deionized water unless otherwise noted.

Setting the chromatographic column

The resin is immersed in deionized water to remove impurities, ensuring that the volume of the deionized water is twice the volume of the resin. The pre-treated resin was prepared into a suspended water solution and transferred to the glass dropper with a length of 0.15 m and a diameter of 6 mm (Fig. 1). A microporous sheet of teflon is placed at the bottom of the glass dropper to avoid resin loss. The column was filled with wet packing method. Finally, another microporous sheet of teflon is placed on top of the packed resin, which prevents the resin from being dispersed. The feed rate should be controlled to ensure stable column pressure during the addition of the stock solution, which facilitates full contact between the solution and the resin.

Separation of Th^{4+} ions with LN resin

The solution with different concentration ratio (Th: La: Ba) was configured under the condition of 1 mol/L HNO_3. Firstly, 20 mL of 1 mol/L HNO_3 was used to flush the loaded LN column, then a mixture solution of Th^{4+}, La^{3+} and Ba^{2+} ions was poured into the separation funnel and passed through the LN column at a flow rate of 1 mL/min, meanwhile, the outflow was collected sequentially in 5 mL containers. The ion concentration in the outflow was measured by inductively coupled plasma emission spectrometer (ICP-OES, PQ9000, Germany). The distribution coefficient (K_d) and the separation factor (SF) were calculated according to the volume of outflow liquid and the ion concentration of outflow liquid to explore

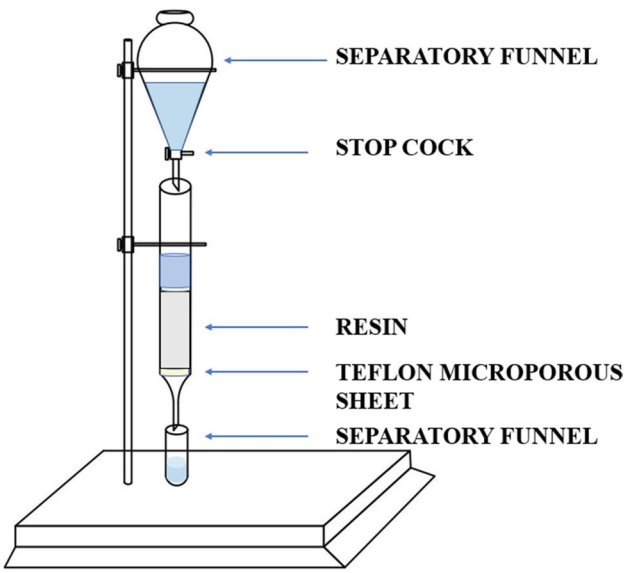

Fig. 1 Diagram of chromatographic column separation device

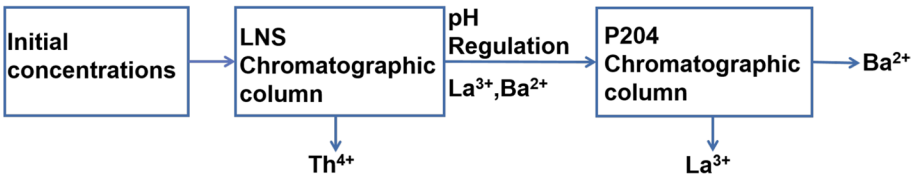

Fig. 2 Flow diagram

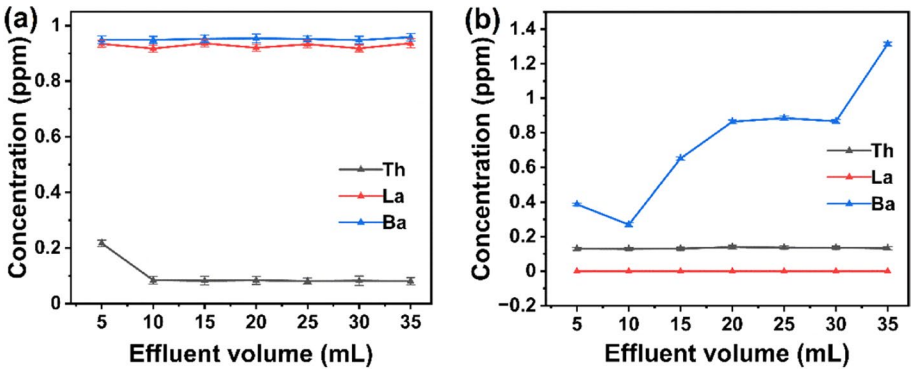

Fig. 3 Effect of LNA resin and P204 resin on separation of Th^{4+}, La^{3+} and Ba^{2+} ions **a** Th: La: Ba = 100 ppm: 0.951 ppm: 0.962 ppm, acidity: 1 mol/L HNO_3, m_{LNA} = 573 mg; **b** Th: La: Ba = 129 ppm: 1.150 ppm: 1.170 ppm, acidity: 1 mol/L HNO_3, m_{P204} = 474 mg

the separation performance of resin. The K_d and the SF were calculated according to the following equation:

$$K_d = \frac{(C_0 - C_e)}{C_e} \cdot \frac{V}{m} \quad (1)$$

$$\mathrm{SF} = \frac{K_d(M1)}{K_d(M2)} \quad (2)$$

where C_0, C_e represent the concentration of metal ions at initial and equilibrium conditions (mg/L), V emblems the volume of the solution (L), and m is the mass of adsorbent (g), respectively.

Separation of La^{3+} ions with P204 resin

The column containing P204 resin was rinsed with 20 mL of nitric acid solution with the same acidity as the feed solution. The mixed solution containing La^{3+} and Ba^{2+} ions breaks through the column, and the outflow was collected at intervals with the volume of 5 mL. ICP-OES was performed to test the ion concentration in the effluent. K_d and SF were calculated to optimize the optimal separation conditions. To further separate and enrich La^{3+} ions, 1 mol/L HNO_3 was used to elute La^{3+} ions adsorbed in the P204 column, still 5 mL fraction collected and La^{3+} ions concentration measured by ICP-OES.

The overall adsorption flow chart is shown in Fig. 2.

Table 1 The separation effect of P204 resin and LNA resin

	K_d^{Th}(L/g)	K_d^{La}(L/g)	K_d^{Ba}(L/g)	$SF_{Th/La}$	$SF_{Th/Ba}$
LNA	80.10	1.247×10^{-3}	4.680×10^{-4}	6.423×10^{4}	1.711×10^{5}
P204	71.17	–	7.472×10^{-2}	–	9.525×10^{2}

Table 2 Separation factors (SF) of Th^{4+}, La^{3+} and Ba^{2+} by different materials

Materials	$SF_{Th/La}$		Refs.
LNresin	6.423×10^{4}	$SF_{Th/Ba} = 1.711\times10^{5}$	–
Cyanex272	24.11	–	[23]
Cyanex302	142.8	–	[23]
TBP	0.48	–	[23]
Water-stable MOF	19.2	–	[24]
Amberlite IR-120	–	$SF_{La/Zr} = 2.1\times10^{3}$	[27]
AM3	–	$SF_{Ra/Th} = 325.3$	[28]

Results and discussion

Optimization of resin for separation of Th^{4+}, La^{3+} and Ba^{2+} ions

Figure 3 displays the separation effect of Th, La, and Ba on an ion chromatographic column containing LNA resin and P204 resin, respectively. As shown in Fig. 3a, LNA resin only adsorbed Th^{4+} ions in 1 mol/L HNO_3, while La^{3+} and Ba^{2+} ions were adsorbed hardly. The calculated K_d and SF values of LNA resin and P204 resin for Th, La and Ba are listed in Table 1. Among them, $SF_{Th/La}$ and $SF_{Th/Ba}$ reach up

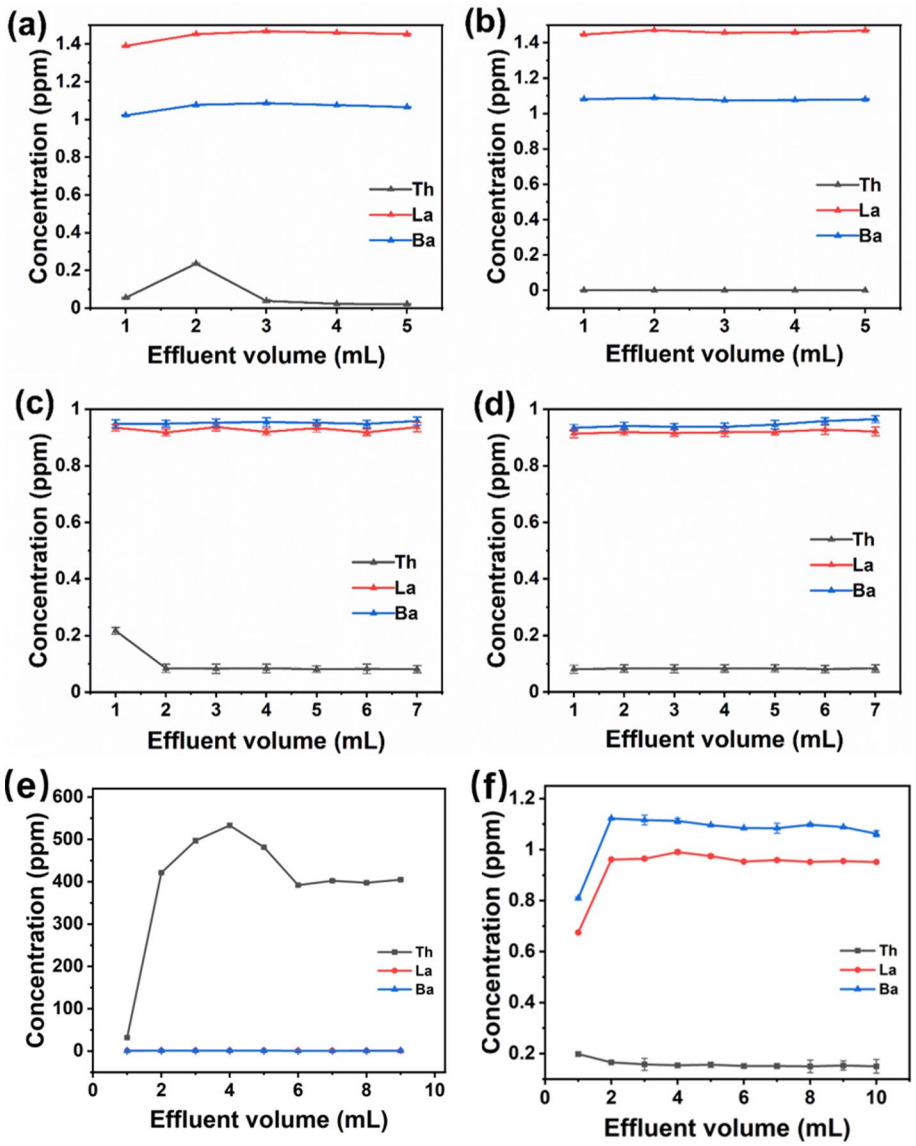

Fig. 4 Effect of different concentrations on LN resin for separation of Th^{4+}, La^{3+} and Ba^{2+} ions. **a** LNA resin, **b** LNS resin (Th: La: Ba = 28.50 ppm: 1.44 ppm: 1.05 ppm,acidity: 1 mol/L HNO_3, m_{LN} = 573 mg); **c** LNA resin, **d** LNS resin (Th: La: Ba = 100 ppm: 0.897 ppm: 0.931 ppm,acidity: 1 mol/L HNO_3, m_{LN} = 573 mg); **e** LNA resin, **f** LNS resin (Th: La: Ba = 500 ppm: 1.00 ppm: 1.10 ppm, acidity: 1,mol/L HNO_3, m_{LN} = 573 mg)

Table 3 The separation effect of LN resin at different concentration

	K_d^{Th}(L/g)	K_d^{La}(L/g)	K_d^{Ba}(L/g)	$SF_{Th/La}$	$SF_{Th/Ba}$
(a)	62.13	1.520×10^{-4}	3.139×10^{-4}	4.088×10^{5}	1.979×10^{5}
(b)	82.88	1.215×10^{-4}	2.928×10^{-4}	6.821×10^{5}	2.831×10^{5}
(c)	80.10	1.247×10^{-3}	4.680×10^{-4}	6.423×10^{4}	1.711×10^{5}
(d)	80.31	1.045×10^{-3}	3.384×10^{-4}	7.685×10^{4}	2.373×10^{5}
(e)	0.1539	1.158×10^{-2}	9.471×10^{-3}	13.29	16.25
(f)	281.6	3.409×10^{-3}	3.424×10^{-4}	8.260×10^{4}	8.224×10^{5}

to 6.423×10^{4} and 1.711×10^{5}, respectively, which is superior to some previous reports (Table 2) [23, 24]. As displayed in Fig. 3b, P204 resin not only can adsorb obviously Th^{4+} and La^{3+} ions, but also can trap a small amount of Ba^{2+} ions, implying that P204 cannot realize the separation of the three elements. P204 is a kind of acidic phospholipid extraction agent, and usually present in the form of dimer (H_2L_2) in non-polar solvent[25] which can form complex though cation exchange, chelation with high valence states ions is similar, so it is difficult to separate Th^{4+} and La^{3+} ions. For LN resin, which is used for actinide lanthanide separation has a similar mechanism The adsorption mechanism of P204 and LN resin in the acid environment is generally described as Eqs. (3) and (4) [20, 26], respectively. The difference of the adsorption effect of the two resins on metal ions can be attributed to the different complexing ability of different cations and ligands under different acidity conditions.

Therefore, LN resin was selected to separate Th^{4+} ions from mixed solution, and subsequent experiments were performed in 1 mol/L HNO_3 to explore the separation effect of LN resin on Th^{4+}, La^{3+}, and Ba^{2+} under different initial concentration ratios.

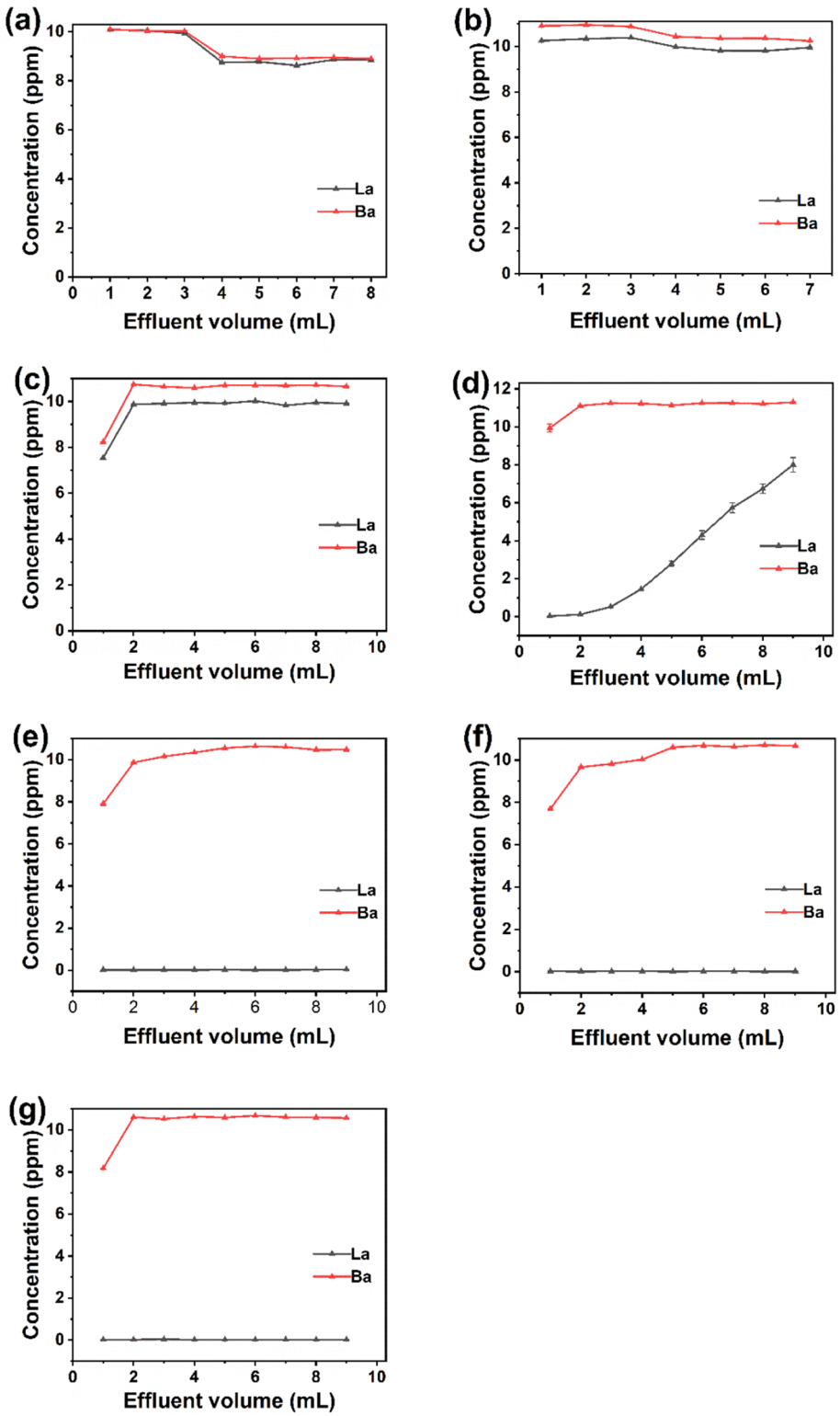

Fig. 5 Effect of different acidity to P204 resin for separation of La^{3+} and Ba^{2+}ions: **a** 1 mol/L HNO_3, **b** 4 mol/L HNO_3, **c** pH = 0.1, **d** pH = 1, **e** pH = 3, **f** pH = 4, **g** pH = 5 (La: Ba = 10.0 ppm: 10.0 ppm, m_{P204} = 474 mg)

$$M^{n+} + nH_2L_2 = \left[M(HL)_{(2n-z)}L_z\right]_0 + zH^+ \quad (3)$$

$$M^{n+} + n\overline{(HY)_2} \leftrightarrow \overline{M\left(HY_2\right)_n} + nH^+ \quad (4)$$

Table 4 The separation performance of P204 resin for La^{3+} and Ba^{2+} ions

	K_d^{La}(L/g)	K_d^{Ba}(L/g)	$SF_{La/Ba}$
1 mol/L HNO_3	1.493×10^{-3}	3.970×10^{-3}	0.3761
4 mol/L HNO_3	6.685×10^{-3}	5.686×10^{-3}	1.176
pH = 0.1	3.841×10^{-3}	2.650×10^{-3}	1.449
pH = 1	1.313	9.056×10^{-3}	1.450×10^{2}
pH = 3	110.3	5.034×10^{-3}	2.191×10^{4}
pH = 4	111.6	5.794×10^{-3}	1.926×10^{4}
pH = 5	110.3	5.627×10^{-3}	1.960×10^{4}

Optimization of concentration on LN resin for separation of Th^{4+}, La^{3+} and Ba^{2+} ions

In the ^{232}Th(p, x) nuclear reaction, only a trace amount of Ac can be obtained after irradiation, leading to a difference of several orders of magnitude in the content of Th and Ac. In order to enrich and separate trace amounts of Ac and Ra in a large amount of Th, it is necessary to explore the influence of the initial concentration ratio of Th: La: Ba on the separation of three metal ions.

In the actual separation system, the content of thorium is very different from that of actinide, so it is particularly important to study the separation of thorium and actinide under different initial concentration ratios. LNA resin and LNS resin of LN series were selected to investigate the separation performance for Th^{4+}, La^{3+} and Ba^{2+} ions by adjusting the initial concentration ratio (Fig. 4). The particle sizes of LNA resin and LNS resin are 100–150 μm and 50–100 μm, respectively. When the initial concentration ratio of Th^{4+}: La^{3+}: Ba^{2+} is 28.50 ppm: 1.44 ppm: 1.05 ppm, LNA resin and LNS resin only adsorb Th^{4+} ions, but not La^{3+} and Ba^{2+} ions (Fig. 4a, b). The calculated $SF_{Th/La}$ and $SF_{Th/Ba}$ values of LNA resin and LNS resin are higher than 10^4, suggesting that Th(IV) separation can be successfully achieved (Table 3). It is worth noting that the SF values calculated by LNS resin separation are slightly higher than those of LNA resin, which means that LNS resin possesses better separation performance for Th^{4+}, La^{3+} and Ba^{2+} ions. With the increase of Th^{4+} proportion (Th: La: Ba = 100 ppm: 0.897 ppm: 0.931 ppm), the SF values by LNA resin and LNS resin were all over 10^4, indicating that Th(IV) separation could still be achieved well (Fig. 4c, d and Table 3). As displayed in Fig. 4e, when concentration ratio of Th: La: Ba is 500 ppm: 1.00 ppm: 1.10 ppm, When the initial solution of Th, La and Ba with high concentration difference still flowed at the original flow rate, the LNA resin with large particle size had certain adsorption effect on Th(IV) but could not completely adsorb it. In addition, it still had no adsorption effect on La(III) and Ba(II). Whereas, the LNS resin was still able to completely adsorb Th^{4+}, but not La^{3+} and Ba^{2+} ions at all (Fig. 4f). The $SF_{Th/La}$ and $SF_{Th/Ba}$ values by LNS resin can reach up to 8.260×10^4 and 8.224×10^5, respectively, which be superior to the $SF_{Th/La}$ (13.29) and $SF_{Th/Ba}$ (16.25) values by LNA resin. The above results indicate that the resin particle size has obvious effect, the smaller the resin particle size, the better the separation performance of Th^{4+}, La^{3+} and Ba^{2+} ions.

Optimization of acidity on P204 resin for separation of La^{3+} and Ba^{2+} ions

P204 was selected as the resin for the separation of La^{3+} and Ba^{2+} ions due to its strong adsorption capacity for La^{3+} ions in previous experiments. Meanwhile, in order to better reflect the adsorption performance of P204 resin on La^{3+} ions, the initial concentration of La^{3+} and Ba^{2+} ions were increased to 10 ppm under the condition that the ratio of La^{3+} and Ba^{2+} ions was kept at 1:1. Figure 5 shows the separation performance of P204 resin for La^{3+} and Ba^{2+} ions under different acidity. The separation factors calculated by P204 resin disposing of La^{3+} and Ba^{2+} ions at different acidity are listed in Table 4. It is clearly observed that P204 resin has a comparatively higher $SF_{La/Ba}$ at pH = 3–5, which is attributed to the high pH promoting ion exchange, suggesting that P204 resin can successfully achieve the separation of La^{3+} and Ba^{2+} ions.

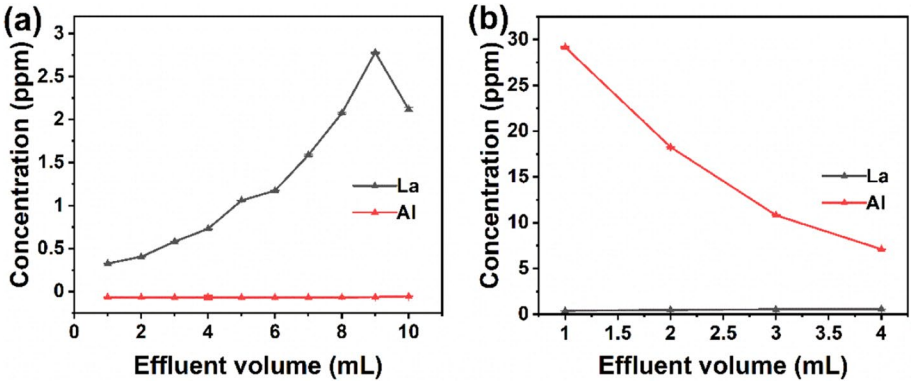

Fig. 6 **a** Adsorption of La^{3+} and Al^{3+} by P204 resin (La: Al = 10.0 ppm: 10.0 ppm, m_{P204} = 474 mg, pH = 3); **b** Desorption of La^{3+} and Al^{3+} ions at 1 mol/L HNO_3

Effect of Al^{3+} on the adsorption of La^{3+} by P204 resin

It is worth noting that a small amount of aluminum is produced by fission during the $^{232}Th(p, x)$ nuclear reaction. Al will interfere with the separation of Ac due to the same valence, while the interference with Th and Ra can be ignored [29].Therefore, it is necessary to separately explore the adsorption properties of P204 on La^{3+} ions in the presence of Al^{3+} ions. As shown in Fig. 6a, the P204 resin can still completely adsorb La^{3+} ions in the presence of Al^{3+} ions, but unfortunately some Al^{3+} ions are also retained in P204 resin, which is not conducive to the enrichment and purification of La^{3+} ions. This is precisely because of the adsorption mechanism of P204 resin which has a certain degree of adsorption of trivalent elements under this acidity condition. Nonetheless, La^{3+} ions can be quickly eluted and Al^{3+} ions are still adsorbed on the resin using 1 mol/L HNO_3 as the eluent. In conclusion, La^{3+} ions can be successfully enriched with LN resin and P204 resin by step separation method.

Conclusions

Combined columns were assembled using LN resin and P204 resin for Th(IV) separation and La(III) enrichment, respectively. The results show that LNS resin can selectively adsorb Th^{4+} ions at the initial concentration ratio of Th: La: Ba = 500 ppm: 1.00 ppm: 1.10 ppm with high separation ratio of $SF_{Th/La}$ and $SF_{Th/Ba}$ of 8.260×10^4 and 8.224×10^5, respectively. In addition, P204 resin could only absorb La^{3+} ions at pH = 3, leading to $SF_{La/Ba}$ up to 2.191×10^4. Even with the interference of Al^{3+} ion, P204 resin can still enrich La^{3+} ion by elution. Although both La^{3+} and Al^{3+} ions were simultaneously retained on the P204 resin, La^{3+} ions were subsequently eluted with 1 mol/L HNO_3 to achieve La(III) separation and enrichment. La^{3+} ions obtained by combined LNS/P204 resin chromatographic column have high radionuclide purity, which provides a possibility for the separation of high purity radiopharmaceutical nuclide Ac from the activation of ^{232}Th by a medium-energy (> 70 MeV) proton.

Declarations

Conflict of interest The authors declare that they have no conflict of interest.

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