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Synthesis of Extraction Resin Containing *N,N,N′,N′*-Tetraisobutyl Diglycolamide and its Application for Separation of Sr(II) from Rb(I)

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Abstract: A novel Levextrel resin containing *N,N,N′,N′*-tetraisobutyl diglycolamide (TiBDGA) was synthesized by suspension polymerization of styrene and divinylbenzene and its performance for separation of Sr(II) from Rb(I) was investigated. The effects of crosslink degree, stirring speed, and the ratio of porogenic-agents on the resin synthesized were examined. The optimum TiBDGA resin with 40 wt.% divinylbenzene as cross linking agent and 20 wt.% of *n*-octanol as porogenic-agents provided excellent adsorption capacity of 22.5 mg/g. The distribution coefficient (K_d) of Sr(II) in HNO_3 media onto the resin depended heavily on the acidity in the aqueous solution. The maximum K_d for Sr(II) (7300 mL/g) was observed when the HNO_3 concentration in aqueous phase reached 2 mol/L. The difference in the Sr(II) and Rb(I) distribution coefficients of several orders of magnitude implied that ^{89}Sr may be separated from ^{86}Rb with a radionuclide purity greater than 99%. And the maximum static adsorption capacity of Sr(II) on the resin was 22.5 mg/g.

Keywords: Levextrel resin, *N,N,N′,N′*-tetraisobutyl diglycolamide, purification, strontium, suspension polymerization

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INTRODUCTION

^{89}Sr is a virtually pure β-emitter, with a half-life of 50.6 days and a mean emission energy of 0.583 MeV. It is widely used for palliation of pain in patients suffering from of bone metastases (1). Through the ^{89}Y (*n, p*) ^{89}Sr reaction, high-specific activity as well as carrier-free ^{89}Sr can be obtained. In the production of ^{89}Sr, pelletized yttrium oxide of natural isotope composition is used as a target and irradiated in a fast-flux reactor with the fast neutron flux of more than $1 \times 10^{15}\,cm^{-2}\,s^{-1}$ for 60–100 days. The yield of ^{89}Sr is about 10–15 Ci/kg ^{89}Y (2–4). Irradiation is followed by reprocessing, including dissolution of targets in nitric acid and purification of ^{89}Sr. The purification of ^{89}Sr is carried out by means of the following steps: first crystallization of bulk yttrium nitrate, then extraction separation, and finally chromatography purification. The purpose of final purification procedure is the elimination of micro impurity isotopes such as ^{86}Rb which is the dominate impurity (2) (Its radioactivity is that of about 1% of ^{89}Sr). Since ^{86}Rb is a γ-emitter whose γ-ray is severely harmful to health of patients, it must be removed thoroughly. Therefore, it is very important to separate ^{89}Sr from ^{86}Rb in the production of ^{89}Sr.

In the present, ion-exchange and extraction chromatography are usually employed to separate ^{89}Sr from ^{86}Rb (3–5). In terms of the former, due to the significant difference of selectivity coefficient between bivalent strontium and univalent rubidium toward cation exchange resin, it is feasible to separate ^{89}Sr from ^{86}Rb thoroughly. However, in order to elute ^{89}Sr, high concentration hydrochloric acid of 4–5 mol/L as elution agent is necessary because of its strong adsorption on the cation exchange resin. This is very disadvantageous for the subsequent adjustment of acidity of desorbed solution containing ^{89}Sr due to the criterion of pH 4–7.5 of $^{89}SrCl_2$ injection. Usually, the adjustment of acidity is carried out by evaporating the solution up to moist salts and diluting with the hydrochloric acid of pH 4–5, which causes some unsatisfied results. On the one hand, there will be a large loss of ^{89}Sr (about 10–20%) in the process of boiling-off high concentration hydrochloric acid; on the other hand, hydrochloride gas is corrosive and harmful. In terms of the extraction chromatography, a kind of commercial resin named as Sr. Resin™ is often used for the separation of ^{89}Sr from ^{86}Rb. Sr. Resin™ contains the crown ether compound of bis(t-butylcyclohexano)-l8-crown-6 which is a specific extractant for strontium (5–7). It is very effective for strontium separation (8–10). ^{89}Sr can be eluted with dilute hydrochloric acid. The final acidity adjusting is much easier and simpler than that of ion-exchange method. However, bis(t-butylcyclohexano)-l8-crown-6 is quite difficult to be synthesized and very expensive, which leads to the higher

production cost of ^{89}Sr. Therefore, there are growing interests to pursue new methods for the final purification of ^{89}Sr.

In recent years, it has been found that some diglycolamides including *N,N,N′,N′*-tetraoctyl diglycolamide (TODGA) and *N,N,N′,N′*-tetraisobutyl diglycolamide (TiBDGA) can directly extract strontium from acidic solution. The strontium extracted can be stripped effectively by HNO_3 with low concentration (11–14). Compared with bis(t-butylcyclohexano)-l8-crown-6, diglycolamides have several advantages for use as extractants for strontium. One advantage is that the raw materials of these diglycolamides are much cheaper than those of crown ethers. Another advantage is that their synthesis process is easy. So a series of studies have been carried out by using various diglycolamides in the separation and purification of strontium (15,16). At present, the extraction resins containing diglycolamides have also been developed. Zhang et al. improve MAREC (Minor Actinides Rocovery from HLW by Extraction Chromatography) process utilizing TODGA/SiO_2-P polymeric materials to remove Sr(II) from simulated high level liquid waste by extraction chromatography (17–20). Ansari et al. have used TODGA as the stationary phase in the extraction chromatographic separation of actinides and other metal ions from pure nitric acid as well as from simulated high-level waste (21). Horwitz et al. prepared and characterized two extraction resins adsorbing TODGA and *N,N,N′N′* tetrakis-2-ethylhexyldiglycolamide (TEHDGA), and identified their potential applications (22). The above extraction resins are prepared by the direct adsorption of the extractants on the polymeric supporters. This kind of extraction resin is usually called solvent impregnated resin. Another method for preparation of extraction resin by the polymerization of styrene and divinylbenzene in the presence of the extractant has been proposed by Kroebel and Meyer (23). The resin prepared by the above method is called Levextrel resin. The extractant of Levextrel resin can be firmly fixed in the macropores of the polymer and is not liable to lose.

In our previous work, Sr(II) can be extracted effectively with TiBDGA from nitric acid solution by using n-octanol–kerosene as diluent, and be stripped effectively employing 0.01 mol/L nitric acid solution (15). At the same time, alkali metal ions such as Na(I), K(I), and Rb(I) are practically not extracted (distribution ratio $<10^{-2}$). Theoretically, the efficient separation of Sr(II) from Rb(I) can be achieved using Levextrel resin containing TiBDGA. To the best of our knowledge, the synthesis of TiBDGA Levextrel resin and its performance have not been reported. In the present paper, we report on the synthesis of the Levextrel resin containing TiBDGA by suspension polymerization of styrene and divinylbenzene and its performance for the separation of ^{89}Sr from ^{86}Rb.

EXPERIMENTAL

Reagents

TiBDGA was synthesized in our laboratory and the details of the synthesis procedure are described elsewhere (24). Its chemical structure is shown in Fig. 1. The product obtained was characterized by IR, MS, ^{1}HNMR, and elemental analysis (purity >98%). Sulfonated kerosene was treated by reference to literature (25): Kerosene (Chengdu Kelong chemical, industrial grade) was pretreated with concentrated sulfuric acid to remove unsaturated hydrocarbon. Styrene (Kanto chemical, 99%) and divinylbenzene (Chengdu Kelong chemical, 80%) were pretreated with 5% sodium hydroxide solution to remove inhibitors and benzoyl peroxide (Acros organics, 99%) were used as the initiators of polymerization. Polyvinyl alcohol (Chengdu Changzheng chemical, degree of hyrolysis; 88 mol%), $CaCO_3$ and Sodium dodecyl-sulfonate (Chengdu Kelong chemical, 99%) were used as dispersant agents. ^{89}Sr and ^{86}Rb were provided by the China Institute of Academy of Engineering Physics with radiochemical purity 99.3%. The other chemicals such as nitric acid and n-octanol used were of analytical grade and without further treatment.

Synthesis of TiBDGA Resin

The procedures for synthesizing resin by suspension-polymerization are shown in Fig. 2. The suspension-polymerization reaction was carried out in a 100-mL three-neck round bottom flask. To the reactor were added 6.4 g styrene, 1.6 g–6.4 g divinylbenzene, 0.05 g benzoyl peroxide, 3.2 g TiBDGA and different ratio of pretreated kerosene, polyvinyl alcohol as porogenic-agents followed by addition of 1 wt% monomer amount of polyvinyl alcohol, $CaCO_3$ and sodium dodecyl-sulfonate in 30-mL deionized water as dispersion agents. The suspension-polymerzation reaction was conducted at 80°C for 24 h at different stirring speed. The

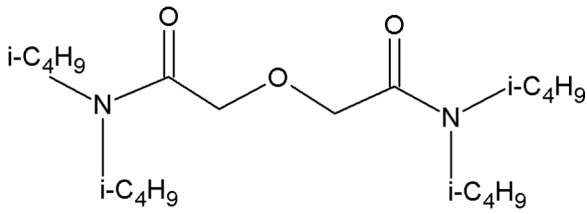

Figure 1. Structure formula of *N,N,N′,N′*-tetraisobutyl diglycolamide.

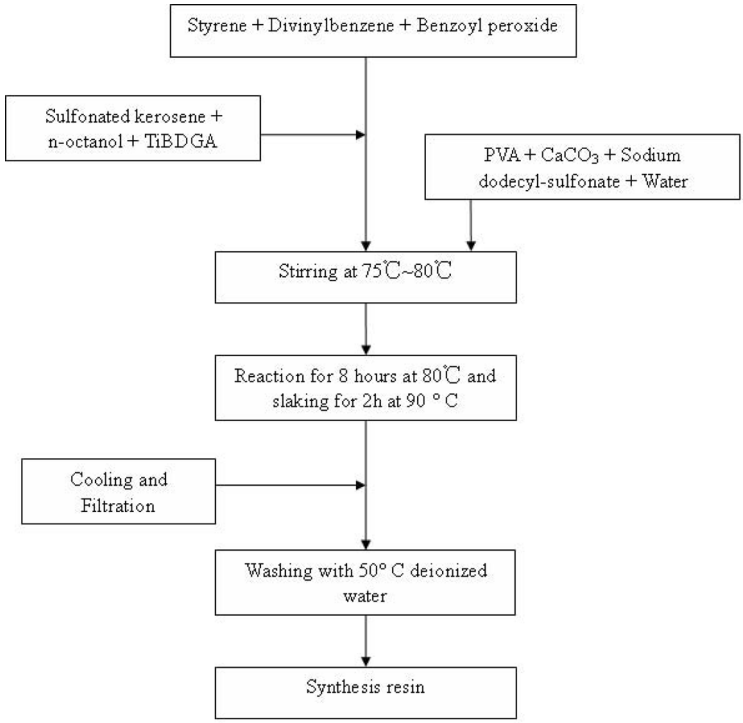

Figure 2. The procedures for the suspension-polymerization method for resin synthesis.

extraction resins synthesized after the copolymerization reaction were washed with methanol or acetone and were filtered to eliminate remaining solvent. The particle size and its distribution were measured by the Scanning Electronic Microscope (SEM, Hitachi S-3000H) and the surface area of resin particles was investigated by Surface Area and Porosity Analyzer (Tristar 3000).

Adsorption Experiments

Adsorption experiments were performed in batch mode, a weighed amount of TiBDGA extraction resin (typically 0.1 g) was combined in a glass vial with a measured volume (typically 10 mL) of an aqueous solution containing 5×10^{-3} mol/L $Sr(NO_3)_2$ or $RbNO_3$ with trace amount of the radionuclides ^{89}Sr or ^{86}Rb. The resultant mixture was maintained in a water bath at 25°C and shaken mechanically at 150 rpm. Then the

solution was separated from the extraction resin by filtration and sampled for analysis. Suitable aliquots of the aqueous phase were taken before and after equilibration for assaying radiometrically. Assay of ^{86}Rb was carried out by gamma counting in FT-603 well type NaI(Tl) scintillation counter (Beijing Nuclear Instruments Plant, China). ^{89}Sr solutions were loaded in the stainless discs of 20 mm in diameter and 1 mm in thickness. Afterwards ^{89}Sr aqueous samples were dried by infrared radiator at 105°C. Beta counting for ^{89}Sr was carried out by using FJ-2603 low-background α, β measurement apparatus (Xian Nuclear Instruments Plant, China). The capacity of adsorption Q (mg/g) and the distribution coefficient (K_d) were determined by Eq. (1) and Eq. (2), respectively,

$$Q\ (\mathrm{mg/g}) = \frac{\rho_0 - \rho_c}{m} \times V \tag{1}$$

$$K\mathrm{d}\ (\mathrm{mL/g}) = \frac{\rho_0 - \rho_c}{\rho_c} \times \frac{V}{m} \tag{2}$$

Where ρ_0, ρ_e mean the initial concentration (activity) and equilibrium concentration (mg/mL) of the metal ions in the solution phase; V indicates the volume of aqueous solution (mL), and m is the weight of the dry extraction resin (g).

Column Operation Procedures

The separation experiment for $^{89}Sr/^{86}Rb$ was carried out using a Pyrex-glass column with 10 mm inner diameter and 55 mm in length. The extraction resin was transferred to the column in slurry state. The column and the solutions were kept at a constant temperature of 25°C throughout. Prior to the chromatographic operation, the column was treated with 2 mol/L HNO_3 solution. Then the sample solution containing $Sr(NO_3)_2$ and $RbNO_3$ with a trace amount of the radionuclides ^{89}Sr and ^{86}Rb was added to the column at a constant flow rate of ~2–3 drops per min (~0.15 mL/min) by constant-current pump. Subsequently, the column was washed at a flow rate of ~3–5 drops per min (~0.25 mL/min) with 2 mol/L HNO_3 solution to elute ^{86}Rb. The eluates were collected by an auto-fractional collector in 0.5 mL aliquots. The gamma-activity of ^{86}Rb in each fraction was counted by FT-603 well type NaI(Tl) scintillation counter. Finally, Desorption of ^{89}Sr was carried out at a flow rate of ~3–5 drops per min by diluent HNO_3. The eluates from the column were collected by stainless discs and then dried by infrared radiator. The beta-activity of ^{89}Sr in the effluents was determined by FJ-2603 low-background α, β measurement apparatus.

RESULTS AND DISCUSSION

Synthesis of TiBDGA Resin

Effect of Stirring Speed

The resin particle size depends on the size of the drops of the polymerization solution. The drop of the solution was created by stirring the solution in the suspension polymerization. So the shear rate caused by stirring is the key factor for controlling the size and shape of the resin. The average grain diameter of styrene-divinylbenzene copolymer at different stirring speeds is shown in Fig. 3.

In the range of 150–350 rpm, the resin diameter decreased drastically. At a stirring speed of 150 rpm, the average size of the resin was 300 μm. At 350 rpm, the diameter of product fell to 110 μm. This inverse trend could be explained by the enhancement of particle dispersion caused by increase of stirring speed. Therefore, the desired particle size of TiBDGA Levextrel resin can be obtained by regulating stirring speed. Generally, the particle size of the resin required for laboratory use is in the range of 150–170 μm. We chose the stirring speed of 300 rpm for the synthesis of TiBDGA Levextrel resin in our laboratory at which the resin with grain diameter of 170 μm could be achieved.

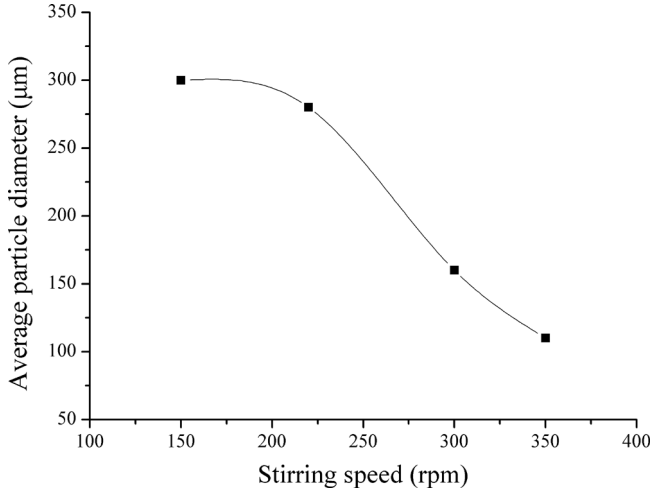

Figure 3. Effect of average bead diameter of styrene–divinyl benzene copolymer as a function of stirring speed.

Effect of Cross-Linking Degree

The degree of cross linking (the weight ratio of divinylbenzene in the gross of monomer) has a great impact on the performances of the resin. For example, with increase of cross-linking degree, the moisture content and degree of swell decreased. However, mechanical strength and the resistance of the chemicals were improved (26). The effect of the cross linking degree on the adsorption capacity was shown in Table 1. The adsorption capacity of the resin increased with the increase of the cross linking degree, the adsorption capacity reached 16.5 mg/g in the cross linking degree of 40%. Further increasing of cross linking degree leads to decrease of adsorption capacity. So the adequate ratio of divinylbenzene in the gross of monomer is suggested to be 40% for the synthesis of the resin.

Effect of Porogenic-Agents

The porogenic-agent could only increase the surface area of the extraction resin and did not influence the particle size. He and Huang indicated that inert solvents or linear polymers, which do not engage in polymerization, create pores in polymer resin during synthesis of macroporous resin (27).

An organic solvent such as toluene, paraffin oil, and alcohols can swell the polymer and thus create micropores in the polymer. *n*-Octanol, which usually employed as a diluent in the extraction of Sr(II) with TiBDGA from acidic solution (15), was added to create the micropores in the synthesis of TiBDGA Levextrel resin. As shown in Table 2, the surface area of the extraction resins increased with the rise of ratio of n-octanol as well as the adsorption capacity of resins were simultaneously improved. When the ratio of n-octanol was 5 wt.% of the copolymer, the

Table 1. The effect of cross linking degree (the weight ratio of divinylbenzene in the gross of monomer) of resin to adsorption capacity

Cross linking degree of resin	Adsorption capacity (mg/g dry resin)
20%	8.5
30%	10.2
40%	16.5
50%	14.7

Table 2. The variation of the surface area of resins in various amounts of n-octanol at cross linking degree of 40%

Ratio of n-octanol in copolymer (wt.%)	Surface area of levextrel resin (m^2/g)	Adsorption capacity for Sr(II) (mg/g)
5%	44.3	16.7
10%	61.6	18.9
20%	77.3	22.5
30%	92.4	22.6

surface area was 44.3 m^2/g and the adsorption capacity for Sr(II) was 16.7 mg/g. The resin surface area increased from 61.6 m^2/g to 77.3 m^2/g when the ratio of n-octanol increased from 10 wt.% to 20 wt.%. At the same time, the adsorption capacity of Sr(II) on the resin increased from 18.9 mg/g to 22.5 mg/g. However, no further increase of Sr(II) capacity was observed when the ratio of n-octanol exceeded 20 wt.%. So it is concluded that the ratio of 20 wt.% n-octanol is the optimum porogenic-agent for resin synthesis.

The Appearance of Resin

The appearance and the particle size of synthesized TiBDGA Levextrel resin were determined by SEM. The TiBDGA Levextrel resin obtained has the similar structure as macroporous ion exchanger (Figs. 4a, b). The resin matrix withdrawing the extraction agent using organic solvent (methanol or acetone) is shown in Fig. 4d. Many micropores distributed in the surface of microspheres evenly. SEM analysis revealed that the structure of TiBDGA Levextrel resin is the same as that of Levextrel resins containing TBP and DEHPA (28).

The Performance of Synthesized Resin

In order to get optimum conditions for obtaining the higher K_d value for Sr(II) on TiBDGA Levextrel resin, batch adsorption experiments were carried out by varying concentration of HNO_3 solution. Fig. 5 shows distribution coefficient of Sr(II) in HNO_3 media onto TiBDGA Levextrel resin as a function of the acidity in the aqueous solution after contact for 24 h at $25 \pm 1°C$. It is obvious that K_d of Sr(II) increased rapidly from 0.2 ml/g to 7300 ml/g with the increase of acid concentration from 0.01 mol/L HNO_3 to 2 mol/L HNO_3. Further increase of acidity gave

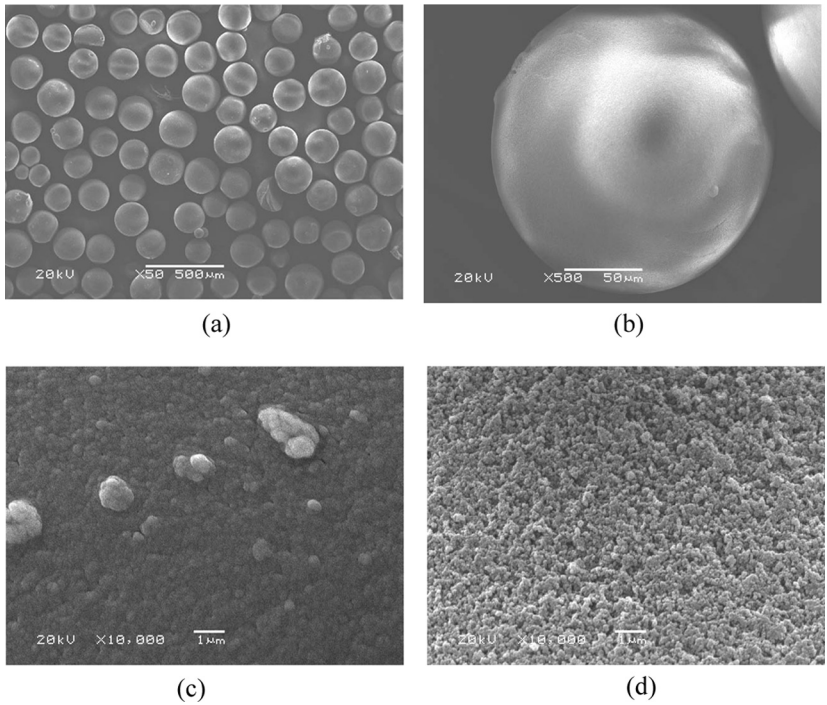

Figure 4. SEM photographs of synthesized extraction resin: (Fig. 4a) × 50; (Fig. 4b) × 500; (Fig. 4c) × 10,000; (Fig. 4d) × 10,000.

rise to the decrease of K_d. The results show a good agreement with the extraction behavior of Sr(II) with TiBDGA in n-octanol from HNO_3 (15). Thus, 2 mol/L HNO_3 was chosen as optimum condition for further retention of Sr(II) by TiBDGA Levextrel resin. In the meantime, the distribution coefficients of Rb(I) were less than 10^{-2} ml/g in the range of acidity investigated, suggesting that alkali metal ions Rb(I) were practically not adsorbed. Therefore, the separation of strontium from rubidium can be carried out by Levextrel resin containing TiBDGA.

Figure 6 shows the distribution coefficient of Sr(II) on resin at uptake acid concentration as a function of time at $25 \pm 1°C$. In the initial 4-hour period, K_d of Sr(II) increased quickly with the absorption time corresponding to Sr^{2+}/TiBDGA extraction process on the outer surface of resin particles. Then, the rate of increase of K_d became slower in the time period from >5 h to 16 h, corresponding to the extraction process in the deeper layers of the swelling resin particles, after extracting the most of TiBDGA-molecules of the outer surface with Sr(II). Finally,

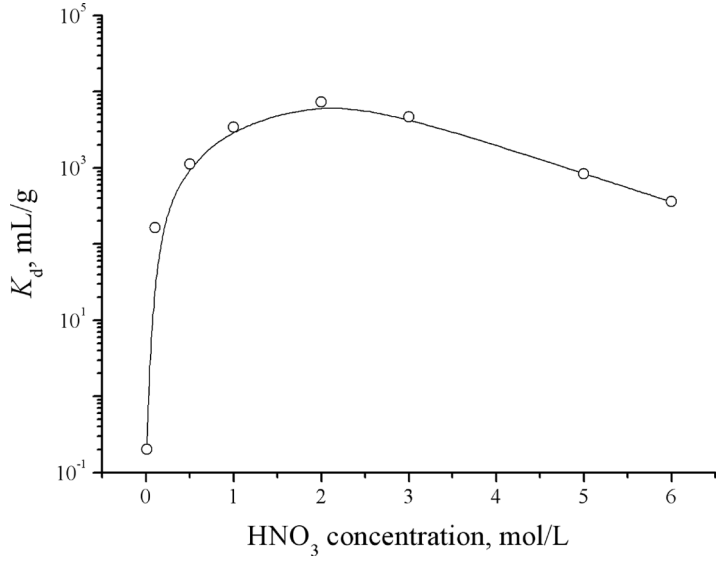

Figure 5. Distribution coefficient of Sr(II) in HNO_3 media onto TiBDGA levextrel resin as a function of the acid concentration in the aqueous solution. Aqueous phase: 5×10^{-3} mol/L $Sr(NO_3)_2$ with trace amount of the radionuclides ^{89}Sr.

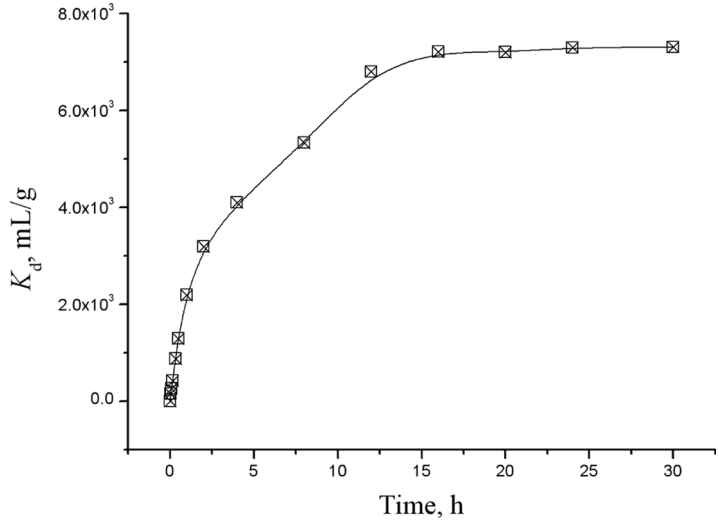

Figure 6. Distribution coefficient of Sr(II) on resin at uptake acid concentration as a function of time. Aqueous phase: $HNO_3 = 2$ mol/L, 5×10^{-3} mol/L $Sr(NO_3)_2$ with trace amount of the radionuclides ^{89}Sr.

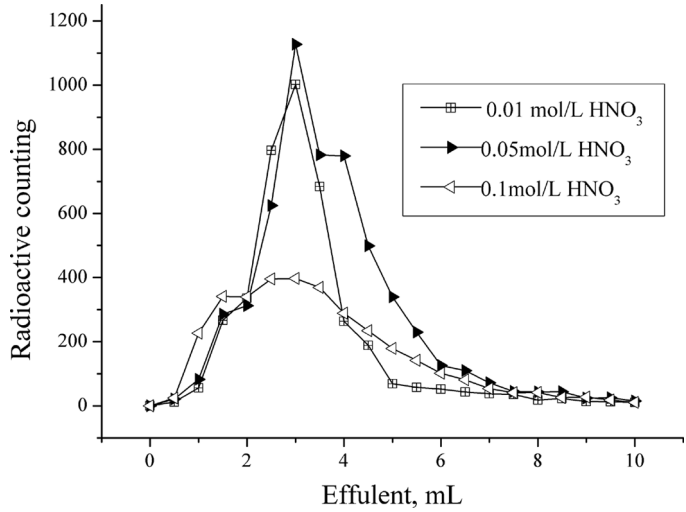

Figure 7. Sr(II) elution profiles for various nitric acid solution of 0.01 mol/L, 0.05 mol/L, and 0.1 mol/L.

no appreciable increase in K_d is noticed (only a little increase) after 16 h until attaining the equilibrium as indicated by the plateau, where the maximum K_d value (7300 ml/g) was obtained at 24 h.

The separation of ^{89}Sr and ^{86}Rb was executed by elution experiment using the TiBDGA Levextrel resin packed column. Rb(I) was effectively removed from the column by washing the column with 2 mol/L HNO_3 solution. The stripping of Sr(II) from the column was carried out by using various concentrations of HNO_3 solution (0.01 mol/L, 0.05 mol/L, and 0.1 mol/L). The Sr(II) elution profiles are shown in Fig. 7. The elution curve of 0.01 mol/L HNO_3 has a good coincidence with the normal probability curve without the tailing peak. The results suggest a feasible stripping agent of HNO_3 at a concentration of 0.01 mol/L. The beta spectrum indicated that efficient strip of Sr(II) can be achieved at a 5 mL-elution-volumes.

CONCLUSIONS

A novel Levextrel resin containing *N,N,N′,N′*-tetraisobutyl diglycolamide (TiBDGA) was synthesized by suspension polymerization of styrene and divinylbenzene. The resin with high adsorption performance was obtained when the cross linking degree was 40% in the present of 20 wt.% *n*-octanol at a stirring speed of 300 rpm. The maximum static

adsorption capacity of Levextrel resin containing TiBDGA for Sr(II) was 22.5 mg/g. The difference in the Sr(II) and Rb(I) distribution coefficients of several orders of magnitude implied that ^{89}Sr can be separated from ^{86}Rb with a radionuclide purity greater than 99%, which meets the criterion of medical $^{89}SrCl_2$ solution. The results here show that the TiBDGA Levextrel resin has an excellent adsorptive characteristic for Sr(II) and can be used in the final purification in the high-specific activity ^{89}Sr production.

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