Design of Microchannel Suitable for Packing with Anion Exchange Resins: Uranium Separation from Seawater Containing a Large Amount of Cesium

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Abstract

We present a resin-packed microchannel that can reduce the radiation exposure risk and secondary radioactive wastes during uranium (U) separation by downscaling the separation using a microchip. Two types of microchips were designed to densely pack the microchannels with resins. The microchannels had almost the same cross-sectional area but different outer circumferences. A satisfactory separation performance could be obtained by arranging more than ca. 10 resins along the depth and width of the microchannels. The resin-packed microchannel is an effective separation technique for determining U concentration via inductively coupled plasma mass spectrometry owing to its ability to avoid the contamination of the equipment by cesium and reduce the matrix effect. The size of the separation site was scaled down to <1/5000 compared to commonly used counterparts. The radiation exposure risk and secondary radioactive wastes can be reduced by 10- and 800-fold, respectively, using the resin-packed microchannel.
**Introduction**

Uranium (U) has a long half-live and is one of the important nuclides for the evaluation of radioactive wastes containing various elements. The concentrated solution of a low-level radioactive liquid waste generated at a research facility often contains high concentrations of matrix elements because a large amount of sodium (Na) is used for neutralization. The accident at the Fukushima Daiichi Nuclear Power Plant (referred as to 1F) and its decommissioning have been generated radioactive wastes (1F samples) containing high concentrations of matrix samples comprising contaminated seawater, and soil. For example, the retained water in a nuclear reactor contains U, other actinides, cesium (Cs-137), and elements contained in seawater. It contains a large amount of $\gamma$-ray-emitting nuclide Cs-137 ($1.4 \times 10^9$ Bq·L$^{-1}$) compared to the target U-238 (5.7 Bq·L$^{-1}$).

The concentration of U can be determined via atomic spectrometric techniques, such as inductively coupled plasma optical emission spectrometry (ICP-OES) and inductively coupled plasma mass spectrometry (ICP-MS). The direct injection of a sample containing high matrix elements (e.g., Na and K) that are easily ionized into ICP apparatuses suppresses and destabilizes the target signal due to the matrix effect. Additionally, injection of samples containing high concentrations of Cs-137 causes the radioactive contamination of the equipment. Therefore, separation of U from various matrix elements is necessary to avoid the matrix effect and the radioactive contamination. Ion exchange and extraction chromatography were reported as conventional bulk-scale methods of U separation from interfered Cs-137 and matrix metal ions. However, these methods are disadvantageous as they are manually operated, have long separation periods, and generate high volumes of waste solutions, increasing
the risk of radiation exposure for analysis workers while generating a large amount of secondary radioactive wastes (e.g., consumable supplies and solutions).

To address the limitations of conventional bulk-scale methods, scaled-down analysis using a microchip has been proposed as an effective solution. A microchip can offer several advantages, such as small volume (µl) of sample and waste solution, accelerated operation, and suitability for automation. Various microchip-based analytical methods have been developed\textsuperscript{9-13}. G.E. Collins \textit{et al.}\textsuperscript{14} reported electrophoresis using a microchip for U separation. Since UV–visible absorption-based detection was adopted, the limit of detection was limited to approximately 100 ppb in this method. G. Hellé \textit{et al.}\textsuperscript{15} reported U analysis combined with solvent extraction using a microchip and an online ICP-MS determination. Solvent extraction with a microchip is a versatile separation method but offers poor control over the flow rates of water and organic solvents, resulting in a relatively low recovery ratio of U (extraction, 72 %; stripping, 42 %; overall, 30 %). Moreover, resin-packed microchannels can accurately separate small amounts of target samples and be coupled with analytical detectors. On-chip chromatographic separation of biomolecules and organic dyes has been performed using the particle-packed column.\textsuperscript{16,17} Applications for separation of hazardous metal ions are limited due to the lack of suitable experimental tools. Therefore, the method of U separation using a microchip is in the basic research stage.\textsuperscript{18}

In this study, we focus on a resin-packed microchannel, which facilitates control over the solution and sensitive U determination via ICP-MS. We integrated an anion-exchange resin to a microchannel based on the results of the study on the separation of U from 1F fuel debris via anion exchange.\textsuperscript{19} A commercially available anion exchange resin (average particle size, 11 µm) that can be packed into a microchannel was used.
The microchannel must be densely packed with resins to efficiently separate traces of the target from highly concentrated matrix elements. As a strategy of microchip design, changing the outer circumference while retaining almost the same cross-sectional area of the microchannels controlled the resin-packed density. In this study, we designed two types of microchips: In Microchip 1, the outer circumference was larger, and the resin-packed density was small by increasing the aspect ratio (depth, 30 µm; width, 1000 µm) of the microchannel. The resin was retained by connecting the microchannels on the upper and under glass plates so that the joint was about 10 µm due to the limitation in fabricating the dam for packing the resin in the shallow depth of the microchannel. Two-three and ca. 90 resins along the depth and width of the microchannel, respectively. In Microchip 2, the outer circumference was smaller, and the resin-packed density was large. Although the shape of the cross-section with the smallest outer circumference was a circle, it had a semicircular shape (inner diameter, 300 µm) due to the difficulty of fabricating a circular microchannel. The dam for packing the resin was fabricated in the microchannel. In the depth and width of the microchannel, ca. 10 and 30 resins were arranged, respectively. The U separation performance of these microchannels packed with the anion-exchange resin was evaluated using a standard solution of U mixed with Lanthanides (Lns). Furthermore, reduce of the matrix effect and the removal ratio of Cs were demonstrated using a seawater sample containing Cs as reference of a high matrix sample.

Experimental
Reagents, chemicals, and sample preparation

The strong base anion-exchange resin (CA08S, average particle size, 11 µm) was purchased from Mitsubishi Chemical Co. (Tokyo, Japan). The standard solution of U mixed with Lns (SM-1418-114) containing Sc, Y, La, Ce, Pr, Nd, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, Lu, and U (10 µg·mL⁻¹ each) was purchased from High Purity Standards (South Carolina, USA) for the performance evaluation of resin-packed microchannels. Fe standard solution (1000 mg·L⁻¹) was purchased from FUJIFILM Wako Pure Chemical Co. (Osaka, Japan). The multi-element standard solution (XSTC-331) containing 10 mg·L⁻¹ of each 29 elements (Li, Be, Na, Mg, Al, K, Ca, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, Ga, As, Se, Rb, Sr, Ag, Cd, Cs, Ba, Tl, Pb, Bi, Th, and U) was purchased from SPEX CertiPrep (New Jersey, USA) to quantify U. The Cs standard solution (1000 mg·L⁻¹) was purchased from SPEX CertiPrep (New Jersey, USA). Nitric acid (HNO₃) and hydrochloric acid (HCl) used for the chemical separation and preparation of final solutions for ICP-MS measurements of ultrapure grade (TAMAPURE AA-10) were supplied by Tama Chemicals Co., Ltd. (Kanagawa, Japan). An HCl solution with a concentration >10 M was obtained from (FUJIFILM Wako Pure Chemical Co.). All solutions were prepared using ultrapure water (>18 MΩ·cm) obtained from a Milli-Q system (Merck Millipore, Massachusetts, USA).

The nearshore seawater certificated reference material NASS-7 (National Research Council of Canada, Ottawa, Canada) was used as a reference sample containing high concentrations of Na and K. The certification of trace metals in this seawater was described in previous studies. The seawater test sample was prepared by adding 11.3 M HCl (final concentration, 9 M) and filtering it with a 0.2 µm filter (hydrophilic PTFE).
Microchip fabrication

The microchips used in this study are shown in Figs. 1 (A) and (B). Microchip 1 having a microchannel (depth, 30 ± 1.5 μm; width, 1000 ± 50 μm; outer circumference, 2060 ± 70 μm; cross-sectional area, 3.0 ± 0.2 × 10^4 μm^2) was fabricated via photolithography and dry etching, as described in previous studies. A quartz glass substrate (size, 30 × 70 mm; thickness, 0.7 mm; VIOSIL-SX, Shin-Etsu Quartz Co., Ltd., Tokyo, Japan) was used. The glass substrate was sputtered with Cr to a thickness of 50 μm. The Cr-sputtered substrate was spin-coated using a negative photoresist. After the UV lithographic micropattern was developed, a plasma dry etching was performed under an atmosphere containing mixture of C3F8/CHF3/Ar gases. The processed substrate was washed with a sulfuric acid–hydrogen peroxide (3:1) mixture and ultrapure water. Then, it was heat-sealed in a vacuum furnace at 1080 °C for 10 h.

We designed a dam-shaped borosilicate glass microchip (Microchip 2) based on the strategy described in the introduction. This microchip having a microchannel (inner diameter, 300 ± 30 μm; outer circumference, 771 ± 33 μm; cross-sectional area, 3.5 ± 0.5 × 10^4 μm^2) was fabricated by Institute of Microchemical Technologies (Kanagawa, Japan).

Resin-packed microchannel and column preparation

A dispersion solution of the anion exchange resin in ultrapure water was loaded into the microchannels of Microchips 1 and 2 to prepare 8 mm and 11 mm long resin-packed microchannels 1 and 2, respectively. A dispersion solution of the resin was loaded into 9 and 16.5 mm long Teflon tube columns (inner diameter, 300 μm) connected to a two-way cock filled with quartz wool (Figure S1 (SI)).
**Ion adsorption and elution**

All solutions were loaded into resin-packed microchannels or a tube column using a microsyringe pump (model. 200 series, KD Scientific, Massachusetts, USA). The sample solution was injected into the microchip after conditioning with 9.5–10 M HCl solution, and the effluent was collected in a vial bottle (injection and washing fraction). Subsequently, ions that were not adsorbed on the column were washed with 9.5–10 M HCl (washing solution), and the effluent was collected in the same bottle. Ions adsorbed on the column were eluted with 0.1 M HCl (elution solution), and the effluent was collected in a separate bottle (elution fraction) from the adsorption fraction. The conditions for each column are shown in Scheme S1 (SI). These fractions were evaporated, and the residues were dissolved using an adequate amount of 0.32 M HNO₃ solution. The concentrations of U and Ln ions in the solutions were determined via a quadrupole ICP-MS (Agilent 7700x). Fe ion concentrations in the solutions were determined via an ICP-OES (Agilent 5800).

**Performance evaluations of resin-packed microchannels and column**

The adsorption, elution and recovery ratios of the U (or other elements) ion were determined using Eqs. 1–3.

\[
\text{Adsorption ratio [\%]} = \frac{w_i - w_a}{w_i} \times 100 \quad (1)
\]

\[
\text{Elution ratio [\%]} = \frac{w_e}{(w_i - w_a)} \times 100 \quad (2)
\]

\[
\text{Recovery ratio [\%]} = \frac{w_e}{w_i} \times 100 \quad (3)
\]

where \(w_i\) is the amount of U (or other elements) injected into the microchannel, \(w_a\) is the amount of U (or other elements) in the injection and washing fraction which is the effluent of sample and washing solutions, and \(w_e\) is the amount of U (or other elements) in the elution fraction.
Results and Discussion

Performance evaluation of resin-packed microchannels 1 and 2

The adsorption and elution performance of the U ion on resin-packed microchannel 1 was investigated by injecting the standard solution of U mixed with Lns (injection volume, 100 µL; ion concentrations, 0.5 ppm each; injected U weight, 52 ng) at a linear velocity and flow rate of 10 m·h⁻¹ and 300 µl·h⁻¹, respectively. On the strong-base anion-exchange resin, U was adsorbed under a high concentration of HCl (>6 M) and eluted at a low concentration of HCl (<1 M). Lns were not adsorbed under the aforementioned conditions of HCl system. The obtained adsorption ratios of U were in the range of 50–60 % (Table 1). The decrease in the adsorption ratio may indicate that the injection amount exceeded the adsorption capacity of the microchannel.

A dynamic adsorption capacity of U was obtained by measuring the concentration of U in the effluent of the injected standard solution of U mixed with Lns (1 ppm each; 10 M HCl). The obtained breakthrough curve is shown in Fig. S2 (SI). A dynamic adsorption capacity \( q_d \) [mol] is given by

\[
q_d = (C_i - C_e) V_D
\]  

where \( C_i \) and \( C_e \) are the concentrations [mol·mL⁻¹] of U in the influent and effluent, respectively, \( V_D \) is the volume [ml] of the effluent that reached 10 % of the U concentration ratio \( (C_e/C_i) \). The obtained \( V_D \) was 0.063 mL from the breakthrough curve, and \( q_d \) was determined to be \( 2.2 \times 10^{-10} \) mol (53 ng as U-238) at a linear velocity and flow rate of 10 m·h⁻¹ and 300 µl·h⁻¹, respectively. Since the injection amount did not exceed the adsorption capacity, the injected U was assumed to have been fully adsorbed.
Though the elution solution was continuously loaded after washing, U was not eluted at all. Therefore, the elution solution was loaded into the microchannel and temporarily stopped for 6 or 149 h before elution. The elution ratio improved to approximately 40% by prolonging the stop time. The adsorption and elution performances of resin-packed microchannel 1 were much lower than the reported performance values.

The adsorption and elution performance of the resin-packed microchannel 2 column was investigated using the standard solution of U mixed with Lns (50 ppb each) at a linear velocity and flow rate of 30 m·h⁻¹ and 1000 μl·h⁻¹, respectively. As shown in Fig. 2, the adsorption, elution and recovery ratios of U were >95% and those of Lns were <1.5%. This result indicates that U can be separated from Lns. The Fe ion adsorption on the microchannel was observed, which has the same adsorption and elution behaviors as the U ion. The adsorption of Fe ions was visually confirmed near the tip of the microchannel when 104 μL of 50-ppm Fe solution was injected (Fig. S3 (A) in SI). The elution of the Fe ion was confirmed by the loading of the elution solution (Fig. S3 (B) in SI). The adsorption, elution, and recovery ratios of the Fe ion obtained using ICP-OES were >95% (Table. S1 in SI). Therefore, we succeeded in preparing a resin-packed microchannel for the U separation.

*Factors for obtaining satisfactory separation performance*

We considered the factors for obtaining satisfactory separation performance by varying 1) linear velocity, 2) injected sample volume, and 3) outer circumference of Microchips 1 and 2. First, the linear velocity of solutions loaded into resin-packed microchannel 2 (30 m·h⁻¹) was faster than one of the solutions loaded into resin-packed microchannel 1 (10 m·h⁻¹). A simple verification of linear velocity using a Teflon tube column was conducted. We used Fe ion, which has the same adsorption and elution
behaviors as U ion to conduct the verification while visually confirming the adsorption. The adsorption and elution ratios at various linear velocities were investigated (Fig. 3 (a)). The adsorption and elution ratios were >95 % for a linear velocity range from 1.7 to 32 m·h$^{-1}$. This indicates that the range of the investigated linear velocity did not affect the separation performance. Second, the injected sample volume in resin-packed microchannel 2 (ratio of the sample injection volume ($V_i$) to the column volume ($V_c$), $V_i/V_c = 290$) was smaller than that injected in resin-packed microchannel 1 ($V_i/V_c = 417$). This verification was also conducted using a Teflon tube column. The adsorption and elution ratios at various sample injection volumes were investigated (Fig. 3 (b)). The adsorption and elution ratios at $V_i/V_c = 64$–943 were >95 %. Sample injection volume also did not affect the separation performance. Third, the microchannel of Microchip 2 with the high U separation performance had ca. 10 and ca. 30 resins (average particle size, 11 μm) along the depth and width of the microchannel, respectively, whereas only 2–3 resins existed in the depth direction of the microchannel of Microchip 1, whose separation performance was relatively poor. Additionally, the cross-sectional shape of Microchip 1 was rectangular, and that of Microchip 2 was semicircular. Since even rectangular microchannels with more than ca. 10 anion-exchange resins can separate metal ions,$^{18}$ there is almost no effect of the shape of the cross-section. These results indicate that resins need to be properly arranged in the microchannel, and the size of the depth and width of the microchannel should be large enough to arrange >10 resins. The lack of arranged resins may form channeling in a microchannel and limit contact with the loaded solution.

*Optimization of ion adsorption and elution*

The recovery ratios of U and Ln ions from 0–120 μL of the washing solution and
30–130 μL of the elution solution were investigated to optimize the washing and elution volume. In the washing fraction, the recovery ratios of Lns were approximately 85 % when the washing volume was 0 μL (Fig. 4 (A)). This is because the injected sample remained in the microchannel and connected tube. The recovery ratios of Lns were >95 % upon the injection of the washing solution >14 μL, and the Lns were completely washed. The recovery ratio of U was <1 % up to 85 μl of washing solution. When the washing solution was 130 μL, the recovery ratio of U was 1.1 %. Although the detected U was negligible for the injection amount, it indicates that the adsorbed U on the column may elute with an increase in the amount of washing volume. Based on these results, the washing volume was set to 50 μL. In elution fraction, the adsorbed U was completely eluted at >30 μL, and the elution ratios of Lns were <0.1 % (Fig. 4 (B)). The elution volume was also set to 50 μL.

Demonstration using seawater

The applicability of resin-packed microchannel 2 to a high matrix sample was also confirmed using a standard seawater sample (Scheme S2 in SI). The adsorption, elution, and recovery ratios of U in the seawater were ~100 % (Table S2 in SI). Although it contained various trace metals, the coexistence of these ions did not affect the separation and quantification of U. Next, the removal ratio of Cs and the reduce of the matrix effect were evaluated adding stable isotope Cs-133 (431 ppb) to the standard seawater at almost the same concentration as that of Cs-137 (1.4 × 10⁹ Bq·L⁻¹, 437 ppb) (contained in the retained water in a nuclear reactor). As shown in Table 2, the adsorption, elution, and recovery ratios of U were ~100 % and those of Cs were <3 %. The measured value of U in the seawater standard was 2.85 ± 0.07 ppb, which was in good agreement with the certified value (2.81 ± 0.16 ppb). The concentration of Cs in
the elution fraction was <1/1000 of the initial concentration. The value of U obtained without separation was smaller than the certified value due to the matrix effect of seawater elements. Additionally, the standard deviation of the value obtained by separating U was smaller than that obtained without separating U (Fig. 5). These results indicate that the prepared resin-packed microchannel 2 can remove Cs and reduce the effect of matrix elements.

Conclusions

Two types of Microchips with resin-packed microchannels were designed to densely pack the microchannel with resins and the effects of varying the outer circumference on U separation performance were evaluated. A satisfactory separation performance was obtained for Microchip 2 by arranging more than ca. 10 resins along the depth and width of the microchannel. Then, separation performance of the microchannel 2 was evaluated by adding Cs-133 (431 ppb) to the seawater standard (1F retained water reported in ref. 1). The measured value of U in the seawater sample was in good agreement with the certified value. The concentration of Cs in the U fraction was <1/1000 of the initial concentration, and the sample solution used for ICP-MS measurement was decontaminated <1/1000. Additionally, the accuracy of analysis improved for the separation of U from the matrix elements. These results indicate that a resin-packed microchannel is suitable for determining U concentrations using ICP-MS and its separation from highly concentrated matrix elements. Compared to the previous studies, the size of the separation site was successfully scaled down to <1/5000 (Table. S3 in SI). The handling time of the resin-packed microchannel is 2 to 13 times shorter
than the previous studies, and the risk of radiation exposure during U separation was approximately reduced 10-fold. The amount of washing/elution volume was 100 to 800 times smaller, indicating that secondary wastes could be reduced 800-fold. It can be expected to apply this microchip to U separation from radioactive wastes and 1F samples in the future.

Acknowledgments

Lns and U were quantified using ICP-MS in the Laboratory Building No.4 (Lab4) of the Japan Atomic Energy Agency. We would like to thank Dr. Shiho Asai of the National Institute of Advanced Industrial Science and Technology for fruitful discussions on performance evaluation of resin-packed microchannel 1.

Supporting Information: The material containing details on separation procedures, the breakthrough curve of the U ion with resin-packed microchannel 1, the adsorption and elution performances of Fe and U ions with resin-packed microchannel 2, and comparison with previous column separation studies is available free of charge on the Web at http://www.jsac.or.jp/analsci/.
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Table 1  Adsorption, elution, and recovery ratios of the U ion on resin-packed microchannel 1 and 2

| Microchannel | Stop time / h | Adsorption ratio / % | Elution ratio / % | Recovery ratio / % |
|--------------|--------------|----------------------|-------------------|-------------------|
| 1            | 6            | 57.6                 | 0.6               | 0.3               |
|              | 149          | 52.6                 | 43.2              | 22.7              |
| 2            | –            | 96.6                 | 100               | 96.9              |

Table 2  Adsorption, elution, and recovery ratios of the U ion in a seawater sample spiked with stable isotope Cs-133 (431 ppb) on resin-packed microchannel 2

|          | Adsorption ratio / % | Elution ratio / % | Recovery ratio / % | Concentration / ppb |
|----------|----------------------|-------------------|--------------------|---------------------|
| U        | Measured value a     | 99.3 ± 0.3        | 102 ± 2            | 102 ± 2             | 2.85 ± 0.07         |
|          | Certified value      | –                 | –                  | –                   | 2.81 ± 0.16         |
| Cs       | Measured value a     | 3 ± 1             | 2.5 ± 0.3          | 0.08 ± 0.03         | 421 ± 6             |
|          | Added value          | –                 | –                  | –                   | 431                 |

a: n = 3.
Figure Captions

Fig. 1  Diagrams of resin-packed microchannels prepared in this study.
(A) Microchip 1; (B) Microchip 2.

Fig. 2  Adsorption, elution, and recovery ratios of U and Lns ions with the prepared resin-packed microchannel 2.
Concentration of U and lanthanides, 50 ppb each; linear velocity, 30 m·h⁻¹.

Fig. 3  Adsorption and elution ratios of the Fe ion with Teflon tube column at various (A) linear velocities and (B) injection volume.
(A) Concentration of Fe, 50 ppm; injection volume, 75 µL. (B) Concentration of Fe, 50 ppm; linear velocity, 12 m·h⁻¹. The broken and solid lines in the figure show the conditions for Microchips 1 and 2, respectively.

Fig. 4  Recovery ratios of U and Lns for (A) 0–120 µL fractions of the washing solution and (B) 30–130 µL fractions of the elution solution.
◆, Sc; □, La; △, Tb, +, Lu; ●, U. Concentrations of U and lanthanides, 50 ppb each; linear velocity, 30 m·h⁻¹.

Fig. 5  Measured value of U in the seawater sample spiked with Cs-133 (431 ppb). n = 3.
Fig. 1
Fig. 2
Fig. 3
Fig. 4
Fig. 5
Graphical Index

Design of resin-packed microchannel for anion exchange

Arrangement of >10 anion exchange resins along the depth and width of the microchannel