Sensors and Materials, Vol. 38, No. 8 (2026) 4963–4974
MYU Tokyo

S & M 4604

Optimization of an Automated Radiochemical Pretreatment Procedure for Radiostrontium Analysis in Large-volume Seawater Samples

Myoung Jung Kim,* Kun Ho Chung, Wanook Ji, and Young-Yong Ji

Environmental Safety Technology Research Division, Korea Atomic Energy Research Institute (KAERI),
111, Daedeok-daero 989 beon-gil, Yuseong-gu, Daejeon 34057, Republic of Korea

(Received December 12, 2025; accepted August 17, 2026)

Keywords: automated pretreatment, radiostrontium, seawater, LabVIEW-based digital twin, column chromatography

Radiostrontium isotopes (^{89}Sr and ^{90}Sr) are important indicators for assessing radioactive contamination and radionuclide transport in marine environments. However, their determination in seawater remains analytically challenging because radiostrontium is a pure beta emitter and occurs at trace levels within a complex, high-salinity matrix. In this study, an automated radiochemical preconcentration and purification system was developed for the rapid pretreatment of radiostrontium from large-volume seawater samples, serving as a front-end module to interface with downstream radiation sensing applications. The automated radionuclide separator (ARS) platform integrates an ion-exchange chromatography (IEX) module for bulk Sr preconcentration, an extraction chromatography (EXC) module for selective Sr purification, and a LabVIEW-based digital-twin platform for automated process control. Resin amounts were optimized through breakthrough tests for both modules. The automated fluid-handling system showed excellent linearity between pump control voltage and flow rate (R^2 = 0.9996) and high reproducibility. Under the optimized conditions, Sr was effectively retained and recovered with high yield while matrix carryover was reduced. The EXC module further demonstrated reproducible Sr recovery from the IEX elution solution, while Ba and Y were monitored as representative interfering elements for future selectivity optimization. These results demonstrate that the proposed automated system provides a reliable and reproducible pretreatment platform for radiostrontium analysis in large-volume seawater. The integration of digital-twin-based process control, stable fluid handling, optimized IEX preconcentration, and EXC purification offers a practical approach for rapid marine radioactivity monitoring and future scale-up toward onboard seawater analysis.

1. Introduction

Radiostrontium isotopes (^{89}Sr and ^{90}Sr) are widely used indicators for evaluating radioactive contamination in marine environments and tracing radionuclide transport in seawater. Because both isotopes are pure beta emitters and exhibit high chemical mobility in seawater, the accurate

*Corresponding author: e-mail: kmj4556@kaeri.re.kr
https://doi.org/10.18494/SAM6203

ISSN 0914-4935

determination of radiostrontium is essential for the long-term assessment of marine environmental impact and for understanding radionuclide transport processes in the ocean. In particular, ^{90}Sr, with a half-life of 28.8 years, is of long-term environmental concern, while ^{89}Sr, with a much shorter half-life of 50.5 days, provides information on recent contamination events.[1–4]

The determination of radiostrontium in seawater remains analytically challenging because radiostrontium occurs at trace levels in a complex, high-salinity matrix. Large amounts of stable matrix elements, including Na, Mg, K, and Ca, can interfere with chemical separation and instrumental measurement. Therefore, preconcentration and purification steps are required before final detection. Conventional radiochemical methods generally involve large-volume seawater sampling followed by coprecipitation, ion-exchange chromatography (IEX), or extraction chromatography (EXC) performed manually.[5–8] Although these methods provide reliable analytical performance, they are time-consuming, labor-intensive, and prone to operator-dependent variability. Such limitations hinder rapid response monitoring and restrict the feasibility of onboard analysis during marine surveys.

To overcome these challenges, the automation of radiochemical separation procedures has attracted increasing attention.[9–12] Automated fluid handling can reduce manual operations, minimize sample contamination, and improve the consistency of chromatographic separation. In addition, real-time process monitoring using sensors and digital control systems can support the stable operation of complex pretreatment sequences. Digital-twin technology, which synchronizes a physical system with a virtual control platform, offers further advantages for visualizing process status, controlling valves and pumps, and optimizing operating conditions in automated analytical systems.

In this study, we developed and evaluated an automated radionuclide separator (ARS) for the automated preconcentration and purification of Sr from large-volume seawater samples. The ARS platform integrates an IEX module for bulk Sr preconcentration and an EXC module for selective Sr purification. The system is controlled by a LabVIEW-based digital-twin platform employing a state-machine architecture. The proposed system was designed as a front-end automated pretreatment module for radiostrontium sensing and marine radioactivity monitoring applications.

The objective of this study was to optimize the automated chemical pretreatment procedure rather than to determine site-specific radiostrontium activity concentrations in environmental seawater. Therefore, stable Sr was used as a chemical surrogate for radiostrontium to evaluate Sr retention, elution, and recovery behavior under nonradioactive experimental conditions. Ba and Y were used as representative interfering elements, and Mg, K, and Ca were monitored as major seawater matrix ions. The performance of the ARS platform was evaluated in terms of fluid-handling stability, bed-volume suitability, Sr recovery, matrix removal, and applicability to future scale-up for large-volume seawater pretreatment.[13–15]

2. Materials and Methods

2.1 Automated radiochemical pretreatment system and digital twin control

The automated radiochemical pretreatment system was designed to perform the sequential preconcentration and purification of radiostrontium from large-volume seawater samples. The system consists of two chromatographic modules: an IEX column for the bulk preconcentration of Sr from seawater and an EXC column packed with Sr-selective resin for purification. The system hardware includes two six-way selector valves, two four-way solenoid valves, two peristaltic pumps, flow sensors, reagent reservoirs, fraction collection lines, and a supporting frame designed for laboratory-scale operation. A photograph and three-dimensional design of the fabricated ARS are shown in Fig. 1. The automated system was controlled using a LabVIEW-based digital-twin platform.[13] The control software was developed using state-machine architecture to execute sequential radiochemical procedures, including column conditioning, sample loading, rinsing, elution, and clean-up. The ARS graphical user interface (GUI) was used to define operating parameters, control valves and pumps, monitor process status, and execute automated separation sequences. The state-machine architecture and ARS GUI are shown in Fig. 2.

The fluid-handling performance of the automated system was evaluated by measuring the flow rate of the peristaltic pump as a function of applied control voltage. Repeated measurements were performed under each voltage condition to assess flow-rate reproducibility. The relationship between pump control voltage and flow rate was used to confirm the stability and predictability of the automated fluid delivery, as will be discussed in Sect. 3.1.

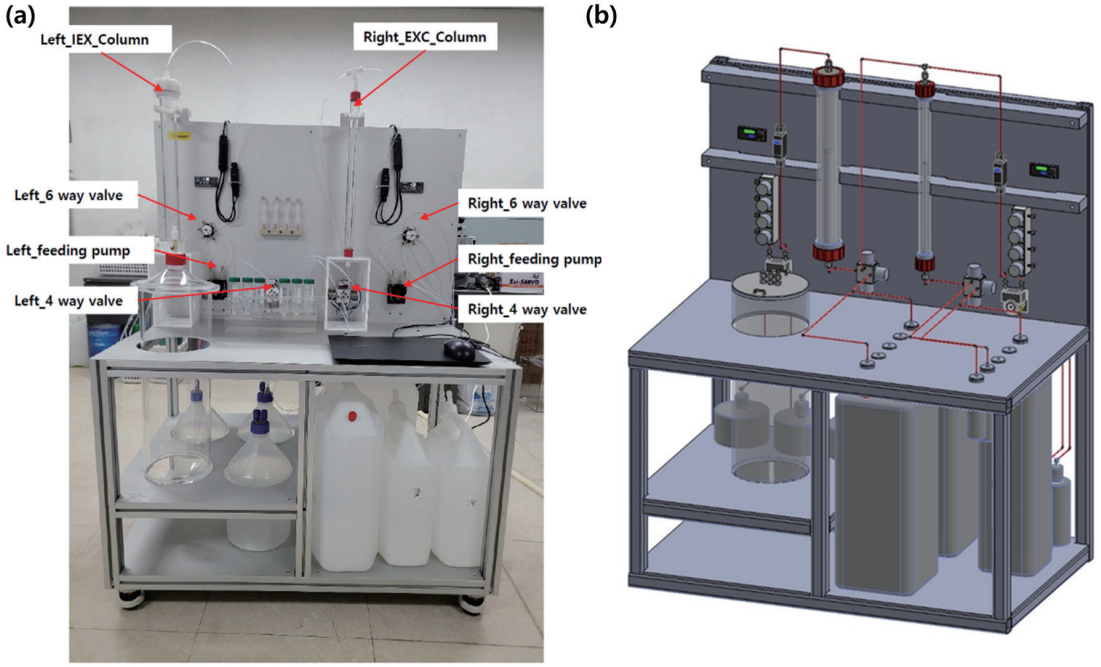

Fig. 1. (Color online) Automated radiochemical pretreatment system. (a) Photograph of the fabricated ARS showing the IEX and EXC modules. (b) Three-dimensional design of the ARS.

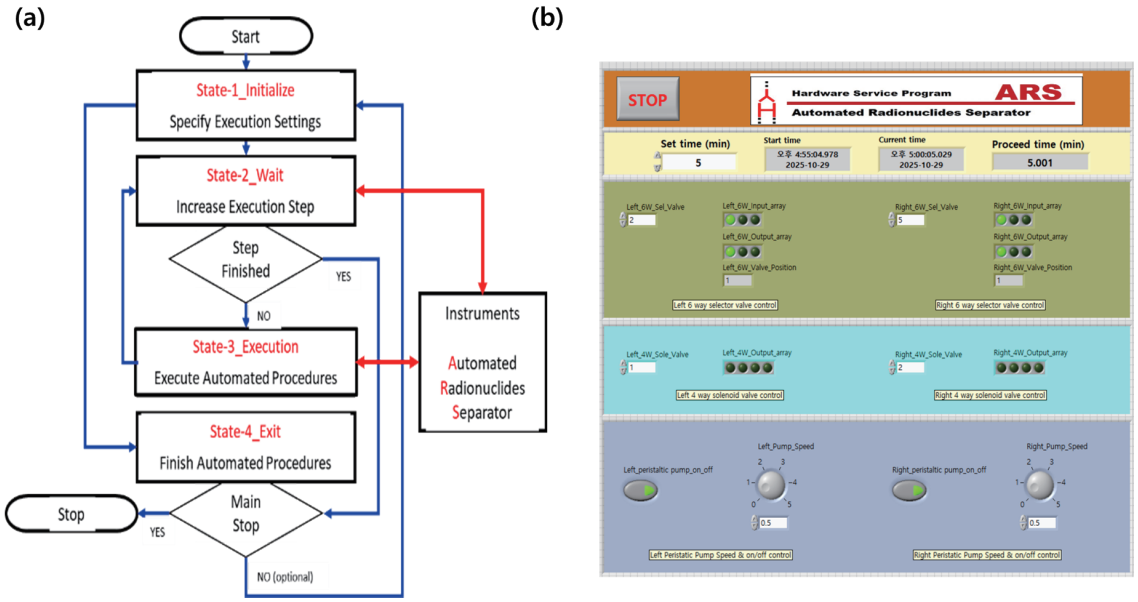

Fig. 2. (Color online) Digital-twin-based control platform. (a) State-machine architecture for automated radiochemical separation. (b) LabVIEW-based ARS GUI for process control and monitoring.

2.2 Chromatographic materials and sample preparation

The IEX column was packed with a strong cation-exchange resin (Bio-Rad, AG50W-X8) to retain Sr and other alkaline earth elements from a high-salinity seawater matrix. The resin was activated before use and packed into the IEX column to a bed volume of 130 mL. This bed volume was selected on the basis of preliminary breakthrough tests using natural seawater. The EXC column was packed with TK102 Sr-selective extraction chromatographic resin (Triskem, France) for the selective purification of Sr from interfering elements such as Ca, Ba, Y, and Ra.[16] The EXC resin was activated before use and packed to a bed volume of 10 mL.

Natural seawater was used as a representative high-salinity matrix to evaluate the automated Sr preconcentration and separation performance of the ARS platform. The seawater sample was utilized to reproduce the major matrix interference conditions encountered during marine radiostrontium analysis, specifically focusing on the competitive behavior of high-concentration macro-ions such as Mg^{2+}, K^{+}, and Ca^{2+}.[1,3] Because the objective of this study was to optimize the engineering and chemical performances of the automated pretreatment procedure rather than to investigate site-specific environmental variations, the exact geographic sampling location was not treated as a critical experimental variable.[1,3]

A 2 L seawater sample was used for the automated IEX pretreatment experiments. Stable Sr was added as a chemical surrogate for radiostrontium to evaluate the retention, elution, and recovery behavior of Sr during the automated chromatographic process. From a chemical standpoint, stable Sr shares identical ionic radii, chemical valence, and thermodynamic/kinetic adsorption mechanisms with radiostrontium isotopes (^{89}Sr and ^{90}Sr) during ion-exchange and

extraction chromatography.[1,3,5] Therefore, employing macrolevel stable Sr as a yield tracer is scientifically justified and widely accepted for validating the mechanical and radiochemical recovery of automated separation platforms under nonradioactive conditions.[3,7,8] However, the use of stable Sr spikes does not directly reflect the actual trace-level detection limits or radiometric counting performance of trace-level radioactive Sr isotopes in environmental samples, which should be evaluated separately during final measurement system validation.[1,3]

2.3 Automated IEX and EXC operating conditions

The automated IEX pretreatment step was performed using the ARS GUI under the operating sequence defined in the LabVIEW-based control program. The IEX procedure consisted of column conditioning, seawater sample loading, rinsing, and Sr elution. A 2 L seawater sample was loaded onto the IEX column at a rate of approximately 50 mL min^{-1}. After sample loading, the column was rinsed to remove major seawater matrix ions. Sr retained on the AG50W-X8 resin was then eluted using nitric acid, and stepwise fractions were collected throughout the procedure for elemental profiling.

The EXC purification step was evaluated separately using the TK102 resin column under automated control. The EXC experiment was designed to assess the selective purification of Sr from an acid matrix representative of the IEX eluate. The EXC procedure consisted of column conditioning, loading, rinsing, and Sr elution. The column was conditioned with 8 M HNO_3, and the loading solution was introduced into the EXC column at approximately 5 mL min^{-1}. Interfering elements such as Ba and Y were removed during the loading and rinsing steps, whereas Sr was recovered in the final elution step using deionized water.

In the present study, the IEX and EXC modules were evaluated as separate automated chromatographic steps to optimize the operating conditions of each module before full integration. The IEX sample-loading step was performed at 50 mL min^{-1}, whereas both the IEX elution step and the EXC loading step were operated at 5 mL min^{-1}. Thus, the Sr-rich IEX eluate can be transferred to the EXC module without a flow-rate mismatch. For future coupled operation, the IEX eluate will be collected in an intermediate reservoir with the solution acidity adjusted to the required EXC loading condition, such as an 8 M HNO_3 matrix. The conditioned solution will then be introduced into the EXC column at 5 mL min^{-1}. This interface design provides a practical strategy for integrating the separately optimized IEX and EXC modules into a fully automated workflow. The detailed operating conditions for the IEX and EXC modules are summarized in Table 1.

2.4 Elemental analysis and performance evaluation

The collected fractions from the IEX and EXC experiments were analyzed to evaluate Sr recovery, matrix removal, and carryover of interfering elements. Mg, K, and Ca were measured as representative major seawater matrix ions, whereas Sr was measured as the target element. Ba and Y were measured as representative interfering elements for the downstream Sr purification step.

Table 1
Operating conditions for the automated IEX and EXC modules.

Parameter	IEX module	EXC module
Resin	AG50W-X8 Cation-exchange resin	TK102 Sr-selective extraction resin
Column dimension	I.D. 30 mm × H 450 mm	I.D. 10 mm × H 450 mm
Packed resin volume	130 mL	10 mL
Bed volume	1 BV = 130 mL	1 BV = 10 mL
Sample/ loading solution	2 L seawater	IEX elution solution
Loading flow rate	50 mL min^{-1}	5 mL min^{-1}
Conditioning	0.1 M HNO_3, 10 BV; DIW, 10 BV	8 M HNO_3, 10 BV
Conditioning flow rate	50 mL min^{-1}	5 mL min^{-1}
Rinsing	DIW, 10 BV	8 M HNO_3, 10 BV
Rinsing flow rate	50 mL min^{-1}	5 mL min^{-1}
Elution	8 M HNO_3, 2 BV	DIW, 3 BV
Elution flow rate	5 mL min^{-1}	5 mL min^{-1}
Evaluation mode	Separate automated IEX test	Separate automated EXC test

Note: IEX, ion-exchange chromatography; EXC, extraction chromatography; I.D., inner diameter; BV, bed volume; DIW, deionized water.

The amount of each element in each fraction was calculated from the measured concentration, dilution factor, and fraction volume. Because the density of each fraction can differ depending on the solution composition and acid concentration, the fraction volume was calculated from the measured fraction mass and solution density, when necessary, as

$$V_i = \frac{m_i}{\rho_i} \tag{1}$$

where V_i is the volume of fraction i, m_i is the measured mass of fraction i, and ρ_i is the density of fraction i. The amount of Sr in each fraction was then calculated as

$$n_i = C_i \times D_i \times V_i, \tag{2}$$

where n_i is the amount of Sr in fraction i, C_i is the measured Sr concentration in the diluted analytical solution, D_i is the dilution factor, and V_i is the corrected fraction volume. The Sr recovery was calculated as the ratio of the total amount of Sr recovered in the elution fractions to the total amount of Sr introduced into the system.

$$Recovery\ (\%) = \frac{\sum (C_i \times D_i \times V_i)}{C_0 \times D_0 \times V_0} \times 100 \tag{3}$$

Here, C_0, D_0, and V_0 are the Sr concentration, dilution factor, and volume of the initial loading solution, respectively. Because natural seawater contains naturally occurring stable Sr, the total Sr concentration in the initial loading solution, including both naturally occurring and added stable Sr, was used to calculate the overall chemical recovery.

Matrix carryover was evaluated from the amounts of Mg, K, and Ca detected in the Sr elution fractions. The removal behavior of Ba and Y was evaluated from their fraction profiles during the loading, rinsing, and elution steps. The performance of the automated pretreatment procedure was assessed in terms of flow-rate stability, Sr recovery, matrix removal, interfering-element removal, and reproducibility. Because stable Sr was used as a nonradioactive chemical surrogate, the calculated recovery values represent chemical separation performance rather than the final detection performance or activity concentration of radioactive Sr isotopes.

3. Results and Discussion

3.1 Fluid-handling performance of the automated system

Stable and reproducible fluid handling is essential for automated radiochemical separation because chromatographic retention, rinsing, and elution behavior are strongly affected by flow-rate control. The performance of the peristaltic pump was evaluated by measuring the flow rate as a function of applied control voltage. As shown in Fig. 3, the flow rate increased linearly with pump control voltage over the tested range, with a coefficient of determination (R^2) of 0.9996. Repeated measurements at each voltage condition showed low relative standard deviations, confirming that the automated pumping system provided reproducible and predictable fluid delivery.

These results indicate that the ARS platform can maintain stable flow conditions during automated IEX and EXC operation. The linear voltage-flow relationship also allows the target flow rates for sample loading, rinsing, and elution to be set through software control, which is important for the reproducible execution of sequential chromatographic procedures.

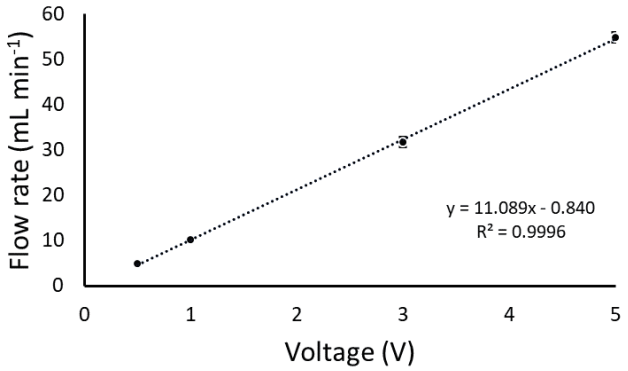

Fig. 3. Relationship between pump control voltage and flow rate for the peristaltic pump used in the automated system. The linear response demonstrates stable and reproducible fluid handling for automated chromatographic operation.

3.2 Breakthrough behavior and bed-volume optimization

The bed volumes of the IEX and EXC columns were selected on the basis of breakthrough behavior under the target loading conditions. Figure 4(a) shows the breakthrough behavior obtained during seawater loading onto the IEX column. Sr was effectively retained by the AG50W-X8 resin under the tested loading condition, indicating that the selected IEX bed volume of 130 mL was suitable for the 2 L seawater pretreatment experiment. Major seawater matrix ions were not intended to be retained quantitatively during this step; instead, the IEX column was designed to retain Sr while allowing a substantial portion of the seawater matrix to pass through during loading and rinsing.

Figure 4(b) shows the breakthrough behavior of the EXC column packed with TK102 resin. The selected EXC bed volume of 10 mL was sufficient to retain Sr from the acid loading solution representative of the IEX eluate, while allowing effective separation from interfering elements. These breakthrough tests provided the experimental basis for selecting the column dimensions used in the automated IEX and EXC experiments.

The current bed-volume optimization was performed specifically for a 2 L seawater loading condition. Theoretical calculations and breakthrough kinetics indicate that directly processing a 40 L seawater sample through the current 130 mL IEX column would induce premature column breakthrough owing to the massive competitive loading of co-existing marine cations. Therefore, scaling up the platform to a 40 L capacity for onboard operations will necessitate engineering modifications, such as a proportional scale-up of the resin bed volume, the utilization of parallel IEX column arrays, or the implementation of an automated sequential column-switching valve configuration. The 2 L optimization data established in this study thus serve as the baseline engineering foundation for designing such large-scale automated hardware networks.

3.3 Automated IEX preconcentration and matrix removal

The automated IEX pretreatment step was evaluated using a 2 L natural seawater sample. Stable Sr was used as a chemical surrogate for radiostrontium to assess the retention, elution,

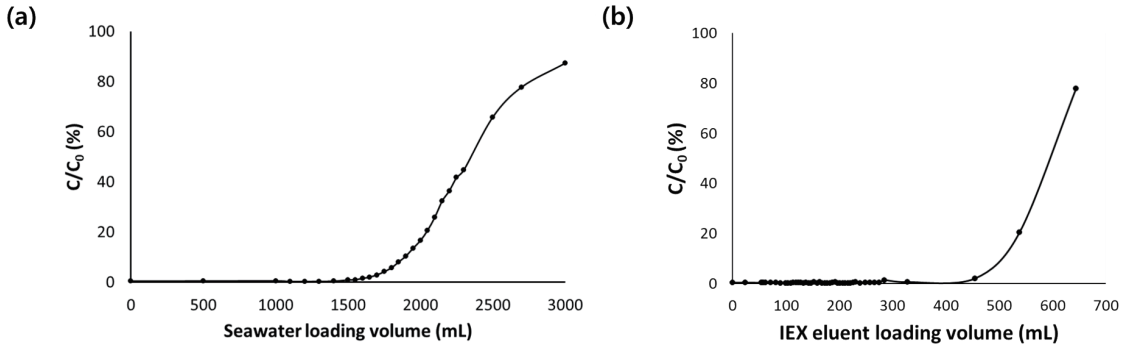

Fig. 4. Breakthrough curves of the chromatographic columns. (a) Breakthrough behavior obtained during seawater loading onto the IEX column. (b) Breakthrough behavior obtained during loading of the acid solution representative of the IEX eluate onto the EXC column.

and recovery behavior of Sr under nonradioactive experimental conditions. As shown in Fig. 5, Sr was retained on the AG50W-X8 resin during sample loading and was subsequently recovered in the elution fraction. The Sr recovery obtained for the automated IEX stage was approximately 106%, which was regarded as near-quantitative recovery within experimental uncertainty.

The fraction profiles of Mg, K, and Ca showed that major seawater matrix ions were largely removed during the loading and rinsing steps.(1) However, minor matrix tails were observed in the later fractions.(1,2) Reviewing the potential impact of this residual matrix tailing is crucial, as co-eluted Mg, K, and Ca can theoretically compete for active sites on the downstream EXC resin, potentially degrading its capacity or chromatographic resolution.(2) Crucially, our separate automated EXC experiments demonstrated that the TK102 resin exhibits exceptionally high selectivity for Sr^{2+} over major alkaline earth matrix ions in strongly acidic media, maintaining a near-quantitative Sr recovery of 96 ± 6% even when processing the actual matrix-bearing IEX eluate.(1,2) This proves that the minor matrix carryover observed does not exceed the operational tolerance of the current EXC module.(2) Nevertheless, to ensure long-term reproducibility and minimize resin degradation in continuous operations, the IEX rinsing protocol should be further tightened either by increasing the rinse volume or optimizing the rinse solution acidity to completely eliminate matrix tailing before full integration.(1,2)

3.4 Automated EXC operation and Sr recovery

The automated EXC module was evaluated using the actual IEX elution solution as the loading solution to verify the applicability of the automated purification process. Stable Sr was used as a chemical surrogate for radiostrontium, while Ba and Y were included to monitor elemental behavior during automated EXC operation. Fractional recoveries were calculated using the total amount of each element introduced into the EXC column as the reference.

As shown in Fig. 6, stable Sr was reproducibly recovered during the automated EXC operation, with an average recovery of 96 ± 6%. This result indicates that the automated EXC module provided stable solution transfer, reproducible column operation, and minimal Sr loss during purification. Although Ba and Y were monitored as representative interfering elements,

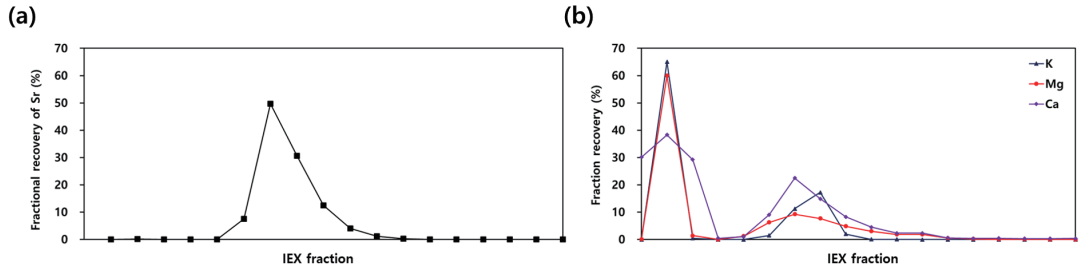

Fig. 5. (Color online) Fractional recovery profiles of stable Sr and major seawater matrix ions during automated IEX operation using 2 L of natural seawater. (a) Sr recovery profile across the collected IEX fraction. Stable Sr was mainly recovered in the elution fractions, with the highest recovery observed in the early elution fraction. (b) Fractional distribution profiles of Mg, K, and Ca. Major seawater matrix ions were largely removed during the loading and rinsing steps, although minor matrix tailing was observed in later fractions.

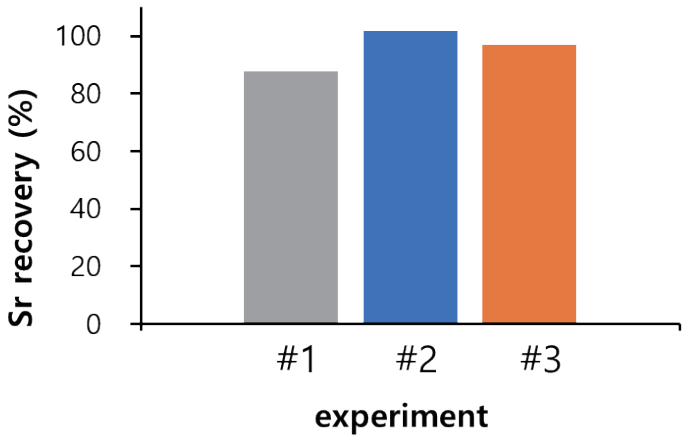

Fig. 6. (Color online) Overall recovery of stable Sr during automated EXC operation using the actual IEX elution solution as the loading solution. The average recovery obtained from three independent experiments was 96 ± 6%, demonstrating reproducible automated EXC operation with minimal Sr loss.

the present EXC experiment focused primarily on evaluating automated Sr recovery rather than complete chromatographic selectivity.

The objective of the present EXC experiment was to evaluate the automated operation and Sr recovery of the EXC module rather than to optimize chromatographic selectivity. Therefore, although stable and reproducible recoveries were obtained, further optimization of the EXC operating conditions, including acid concentration, rinsing protocol, and elution conditions, will be required to improve the separation of Sr from interfering elements during future implementation of a fully integrated automated IEX–EXC workflow.

3.5 Applicability of automated pretreatment platform

The proposed ARS integrates automated fluid handling, digital-twin-based process control, optimized IEX, and automated EXC into a single pretreatment platform for radiostrontium analysis. Compared with conventional manual radiochemical pretreatment procedures, the ARS platform minimizes manual intervention, improves operational reproducibility, and enables real-time monitoring of the chromatographic process. These characteristics are advantageous for routine environmental monitoring and rapid emergency response requiring the reproducible processing of large-volume seawater samples.

The optimized IEX module achieved near-quantitative recovery of stable Sr while substantially reducing the major seawater matrix. Although minor matrix tailing of Mg, K, and Ca was observed in the later fractions, stable Sr recovery during the subsequent automated EXC operation remained high. The influence of residual matrix components on EXC performance should be further investigated after the implementation of a fully integrated IEX–EXC workflow. Optimization of the rinsing conditions and matrix control is expected to further improve the robustness of the overall pretreatment procedure.

Table 2
Comparison of the proposed ARS platform with conventional manual and automated radiostrontium pretreatment methods.

Pretreatment method	Sample volume (L)	Processing time (h)	Flowrate linearity (R^2)	Chemical yield (%)	Operator dependence/labor intensity
Conventional manual method (co-precipitation & gravity Flow)[5–8]	2 to 40	24 to 48	N/A (Manual gravity flow)	60 to 80	Extremely high/ high labor intensity
Flow-injection-based automated system[11,12]	≤1	1 to 3	Not specified/ Flow fluctuations under pressure	80 to 90	Low/limited to small volumes
Proposed ARS platform (digital twin controlled)[this work]	2	≤3	0.9996	~100% (IEX) ~96% (EXC)	Low/automated workflow

In the present study, we demonstrated the applicability of the ARS platform for the automated pretreatment of 2 L seawater samples. Extension to larger sample volumes, such as 40 L, will require the additional optimization of resin capacity, column dimensions, flow synchronization, and pressure management. Nevertheless, the optimized operating conditions established in this study provide an engineering basis for future scale-up of the automated pretreatment system.

Although the IEX and EXC modules were evaluated independently in the present work, the results demonstrate the feasibility of integrating the two optimized modules into a fully automated pretreatment workflow. Future studies will focus on coupling the optimized IEX and EXC modules and validating the integrated automated pretreatment workflow for radiostrontium analysis in large-volume seawater samples.

To highlight the operational advantages and performance of the proposed ARS platform, a comprehensive, quantitative comparison with conventional manual methods and existing automated radionuclide separation techniques is summarized in Table 2.

4. Conclusions

An ARS was developed to automate the radiochemical pretreatment procedure for radiostrontium analysis in large-volume seawater samples. The proposed system integrates automated fluid handling, a LabVIEW-based digital-twin control platform, IEX, and EXC into a reproducible pretreatment platform. The automated fluid-handling system demonstrated excellent flow-rate linearity and reproducibility, providing stable operating conditions for sequential chromatographic processes.

The optimized IEX module achieved near-quantitative recovery of stable Sr while substantially reducing major seawater matrix ions, demonstrating its suitability for the automated preconcentration of Sr from 2 L seawater samples. The automated EXC module further demonstrated the reproducible recovery of stable Sr using the actual IEX elution solution with minimal Sr loss during automated operation. The IEX and EXC modules were evaluated independently in the present study to optimize the operating conditions of each module before integration into a single automated workflow.

Overall, the results demonstrate the feasibility of the proposed ARS platform as an automated front-end pretreatment system that can interface with downstream radiation sensing technologies for radiostrontium analysis in large-volume seawater. Future work will focus on integrating the optimized IEX and EXC modules into a fully automated workflow, further optimizing matrix removal and chromatographic separation, and validating the system for large-volume marine radioactivity monitoring applications.

Acknowledgments

This research was supported by Korea Institute of Marine Science & Technology Promotion (KIMST) funded by the Ministry of Oceans and Fisheries (RS-2023-00256174).

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