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A Systematic Study on Uranium Purification from Seawater Matrices for Alpha Spectrometry

Lee Dayeong*, Jang Mi
Environmental Radioactivity Assessment Team, Korea Atomic Energy Research Institute, 111, Daedeok-daero 989beon-gil, Yuseoung-gu, Daejeon, 34057, Republic of Korea
**Corresponding author: dylee@kaeri.re.kr*

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1. Introduction

A precise monitoring system for natural and anthropogenic radionuclides in the marine environment is essential to prepare for potential radioactive inflows from nuclear accidents or clandestine nuclear activities in neighboring countries. Currently, uranium analysis in South Korea primarily focuses on environmental samples such as soil, surface water, and groundwater. However, seawater is characterized by a high salinity matrix and relatively low radionuclide concentrations compared to other environmental samples. Applying traditional evaporation-based pre-concentration methods to seawater leads to significant salt precipitation, which reduces analytical efficiency. Therefore, advanced pretreatment techniques for selective uranium enrichment and matrix removal are indispensable for high-precision analysis.

Iron hydroxide ($Fe(OH_3)$) co-precipitation is widely utilized in trace metal analysis. At pH 8–9, it effectively removes major seawater constituents, such as alkali and alkaline earth metals (Na, Mg, Ca, K) and various anions, by retaining them in the aqueous phase. In this study, we applied iron co-precipitation to 1 L seawater samples for initial enrichment, followed by selective purification using UTEVA (Uranium and Tetravalent Actinides) extraction chromatography resin [1]. In alpha spectrometry, managing interference factors that degrade spectral resolution is critical. ^{237}Np has alpha decay energies very similar to ^{234}U (4.77 MeV), which can lead to overestimation. Although ^{239,240}Pu peaks do not directly overlap with uranium, poor source preparation can cause tailing that interferes with the ^{232}U tracer region. Furthermore, transition metals such as Fe) or V remaining after pretreatment reduce electrodeposition efficiency and cause self-absorption of alpha particles within the sample layer, deteriorating the spectral quality.

This study aims to maximize spectral resolution by effectively removing these chemical and radioactive interferences using UTEVA resin. The established protocol was validated by evaluating uranium recovery and confirming the complete separation of interfering radionuclides to ensure the reliability of seawater uranium analysis.

2. Methods and Results

Filtered 1 L seawater samples were aliquoted into glass beakers, and their precise weights were recorded. A known amount of ^{232}U was spiked as an internal tracer for quantification, followed by the addition of approximately 10 mg of Fe carrier. While stirring with a magnetic bar, concentrated NH_4OH was added to adjust the pH to 8–9. After 30 minutes of stirring, the solution was allowed to stand until the $Fe(OH)_3$ precipitate settled completely. The supernatant was removed via siphoning using a U-tube, and the remaining concentrate was centrifuged (250 mL tubes) to discard the residual liquid. The recovered precipitate was fully dissolved in 10 mL of a 3 M HNO_3- 1 M $Al(NO_3)_3$ mixture. To prevent Pu adsorption onto the resin, 200 mg of ascorbic acid and 1 mL of 1.5 M sulfamic acid were added as reducing agents to convert Pu to its trivalent state. The solution was left to react for 10 minutes. For separation, 2 mL UTEVA resin columns conditioned with 3 M HNO_3 were used. After loading the sample, the columns were rinsed with 20 mL of 3 M HNO_3 to remove matrix components. Subsequently, 20 mL of a 5 M HCl - 0.05 M oxalic acid mixture was applied to selectively elute tetravalent ions such as Np through strong oxalate complexation. Finally, uranium was eluted using 20 mL of 0.01 M HNO_3. The uranium fraction was prepared as a counting source via electrodeposition and quantitatively analyzed using alpha spectrometry.

3. Result

3.1. Optimization of Reducing Agents for Pu Separation

The removal efficiency of interfering radionuclides (Pu) was evaluated based on the combination of reducing agents in the loading solution. When ascorbic acid was used alone, 30–70% of Pu co-eluted with the uranium fraction. This suggests that ascorbic acid alone is insufficient to fully reduce and maintain Pu in the Pu^{3+} state in a nitric acid medium, allowing some Pu^{4+} to adsorb onto the UTEVA resin.

The addition of a combined reducing agent (Ascorbic + Sulfamic acid) completely eliminated Pu interference from the alpha spectrum. Sulfamic acid effectively removes nitrous acid HNO_2, which acts as a catalyst for the re-oxidation of Pu^{3+} in nitric acid solutions, thereby stabilizing the Pu^{3+} state and preventing its interaction with the UTEVA resin.

3.2. Decontamination of Am and Np

Am was mostly removed during the loading and initial rinsing stages due to its extremely low distribution coefficient (Kd) on UTEVA resin. While Np^{4+} exhibits strong adsorption behavior similar to uranium, the addition of 0.05 M oxalic acid during the rinsing stage successfully removed it. Np preferentially forms a strong oxalate complex, allowing for its selective elution before uranium. This step ensured the acquisition of a high-purity uranium fraction.

3.3. Application to Seawater Samples

In this study, the reliability of the analytical procedure was ensured using a ^{232}U internal tracer. The chemical recovery of uranium following pretreatment and alpha spectrometry ranged from 80% to 90%. This high recovery demonstrates that the proposed iron co-precipitation and UTEVA resin purification process is a robust method, effectively eliminating complex matrix interferences in seawater while minimizing sample loss. Analytical results showed that the average activity concentrations of ^{238}U and ^{234}U in the collected seawater samples were 34.3±2.3 mBq/L and 39.1±2.9 mBq/L, respectively. Typically, uranium in the open ocean exhibits conservative behavior, meaning its concentration varies linearly with salinity. According to the relationship established by Owens et al. [2]

^{238}U [dpm/L] = 0.07081 x Salinity,

the theoretical ^{238}U activity concentration at a standard global salinity of 35 psu is approximately 41.3 mBq/L. The lower ^{238}U concentration observed in this study compared to the open ocean average is attributed to the lower salinity characteristics of the sampling sites. Meanwhile, the $^{234}U/^{238}U$ activity ratio (AR) was calculated to be approximately 1.14. This value is in good agreement with the established seawater activity ratio of 1.15 reported by Thurber [3] The alpha recoil effect, which leads to the enrichment of ^{234}U in seawater—distinct from the secular equilibrium (AR=1.0) observed in the Earth's crust—was clearly evident in our analytical data. The consistency of this activity ratio confirms that the developed method accurately captures the natural radiochemical fingerprint of the marine environment without external contamination, thereby validating the scientific integrity of the results.

CONCLUSION

This study established a precise analytical protocol for uranium in seawater by integrating iron hydroxide co-precipitation and UTEVA extraction chromatography. The proposed method demonstrated excellent robustness with stable chemical recoveries of 80–90% and maximized alpha spectral resolution through the effective removal of interfering nuclides. Analytical results showed that the ^{238}U concentrations were consistent with the characteristics of low-salinity seawater, while the $^{234}U/^{238}U$ activity ratio of 1.14 confirmed the scientific validity of the data by matching the unique radiochemical fingerprint of seawater. Consequently, this analytical protocol is highly effective for characterizing unknown marine samples and monitoring trace uranium, making it a valuable tool for radioactivity monitoring systems across diverse marine environments.

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