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Technical article

A Study on the Interference Elements of Matrix-Specific Chemical Compositions in Radionuclide Analysis of Radioactive Isotope Waste

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ABSTRACT This study investigated the chemical composition of representative radioactive isotope waste matrices—non-woven wiper, polypropylene, and SUS304—and analyzed the effects of matrix-specific elements on chemical separation behavior. The results showed that Ca, Ti, and Fe were detected in the wiper, Ca and Mg in polypropylene, and alloy constituents such as Fe, Cr, and Ni, along with Sb and B, in SUS304. These elements were identified as interference elements that can cause recovery loss and quantitative bias through coprecipitation, competitive adsorption, and colloidal formation during the chemical separation of ^{113}Sn, ^{232}Th, and ^{226}Ra. In addition, microwave-assisted digestion was confirmed to be an effective method for achieving complete dissolution of both organic and metallic matrices while minimizing the loss of volatile elements. The findings of this study provide fundamental data for establishing matrix-specific pretreatment and chemical separation strategies in radionuclide analysis of radioactive isotope waste.

Key words: Microwave digestion, ICP-OES, Radiochemical separation, Interfering element, Chemical composition

1. Introduction

In order to treat and dispose of radioactive waste, radiological characterization must be conducted [1]. In the domestic nuclear regulatory system, radiological characterization is based on the results of destructive radiochemical analysis applying quantitative analysis of radionuclides [2]. To perform such quantitative radionuclide analysis, a series of procedures including chemical pretreatment, measurement, analysis, and evaluation is required. Among these, the dissolution process of the matrix, which constitutes the first stage of chemical pretreatment by various methods, is a critical step that must be carried out reliably [3].

The dissolution process refers to the conversion of elements adsorbed or fixed within the matrix in the form of organic or inorganic species into a liquid form through complete decomposition, thereby enabling chemical separation [4]. If complete dissolution is not achieved, the target elements or radionuclides may be lost before reaching the chemical separation stage, resulting in reduced recovery and distorted quantitative results. Furthermore, non-quantitative information of interfering elements may lead to inappropriate experimental procedures in the chemical separation process, preventing accurate reflection of the behavior of the target elements, thereby reducing the reliability of the results [5].

In this study, several matrices from radioactive isotope waste generated in medical and research institutions were selected, dissolved, and then analyzed for their chemical compositions using ICP-OES(Inductively Coupled Plasma–Optical Emission Spectrometry). The experimental chemical composition results were compared with the manufacturing characteristics or compositional literature of the matrices to verify the reliability of the analysis. In addition, among the radionuclides currently under development for radionuclide analysis techniques (^{40}K, ^{88}Y, ^{113}Sn, ^{139}Ce, ^{155}Eu, ^{211}Bi, ^{212}Pb, ^{214}Pb, ^{214}Bi, ^{228}Ac, ^{232}Th, ^{226}Ra), the chemically challenging nuclides ^{113}Sn, ^{232}Th, and ^{226}Ra were selected to investigate the behavior of major interfering elements in the matrices [4]. Consideration of the elemental characteristics obtained in these experiments is expected to provide technical guidance for chemical separation following radiochemical experiments, as well as fundamental data to justify the

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introduction of additional pretreatment processes either before or after dissolution for various matrices [5].

2. Materials and Methods

2.1. Selection of test samples

For this study, matrices of radioactive isotope waste were selected, including wipers used for decontamination, plastics from the body of medical and research syringes, and metallic matrices such as syringe needles. The samples consisted of an ASTAR ULTRA 3000S non-woven wiper, polypropylene from KOVAX-SYRINGE 3 mL, and SUS304 from KOVAX-NEEDLE 18G [6,7].

For microwave-assisted dissolution, the standard sample weights were determined according to EPA Method 3052, which recommends less than 0.5 g for the digestion of siliceous and organic-based samples [8]. Therefore, 0.5 g, 0.3 g, and 0.5 g were used for the wiper, polypropylene, and SUS304 samples, respectively.

2.2. Method for sample dissolution

Complete dissolution of the matrices cannot be achieved with ordinary acids alone. For full digestion, a combination of strong oxidizing agents, auxiliary oxidants, and hydrofluoric acid is required [9,10]. As shown in Table 1, the wiper samples were treated with a mixture of nitric acid (HNO_3) and hydrofluoric acid (HF). In this system, HNO_3 acted as a strong oxidant to decompose organic matter, while HF dissolved silica (SiO_2), thereby minimizing insoluble residues [11].

For polypropylene, HNO_3 was employed to oxidize and decompose organic matter and to dissolve metal oxides effectively. Hydrogen peroxide (H_2O_2) was added to provide additional oxidation power, promoting the decomposition of refractory organic structures. Furthermore, HF played a critical role in dissolving silica-based impurities and chemically resistant substances, thereby removing residual silicon-based components from the matrix [11,12].

SUS304 is an alloy mainly composed of iron (Fe), chromium (Cr), and nickel (Ni) [13]. Among them, Cr forms a protective Cr_2O_3 passive layer that provides high resistance to acid attack. In this study, chloride ions (Cl^-) from hydrochloric acid (HCl) were used to disrupt this passive film, thereby promoting the dissolution of Cr, Fe, and Ni [14]. Typically, aqua regia ($HNO_3 : HCl = 1 : 3$) is used to dissolve noble metals such as gold or platinum. However, Fe-Cr-Ni alloys like SUS304 are more reactive than noble metals and do not require such excessive oxidative conditions. Therefore, a modified acid mixture of 10 mL HCl and 2 mL HNO_3 (5 : 1 ratio) was applied to facilitate dissolution [15].

2.3. Method for dissolution and compositional analysis

Sample dissolution was performed using a microwave digestion system (Milestone ETHOS EASY). Compared with conventional wet digestion, microwave-assisted digestion enhances reaction rates through focused energy input and minimizes the loss of volatile elements due to the use of sealed vessels [16,17].

In this experiment, the microwave operation was carried out as shown in Fig. 2: the wiper sample was gradually heated to 230°C over 25 minutes and then held at 230°C for 15 minutes; polypropylene was heated to 210°C over 25 minutes and maintained for 10 minutes; and SUS304 was heated to 200°C over 25 minutes and held for 20 minutes to promote decomposition [18].

Since the interior of the vessel remains at high temperature (200-230°C) and high pressure (several tens of bars) after mi-

Table 1. Reagents used for matrix-specific liquefaction

Wiper (ASTAR ULTRA 3000S)	Polypropylene (KOVAX-SYRINGE)	SUS304 (KOVAX-NEEDLE 18G)
70% HNO_3 9 mL, 48% HF 1.0 mL	70% HNO_3 6 mL, 30% H_2O_2 0.5 mL, 48% HF 1.5 mL	37% HCl 10 mL, 70% HNO_3 1 mL

(a) ASTAR ULTRA 3000S non-woven wiper (b) KOVAX-SYRINGE (Polypropylene) (c) SUS304 (KOVAX-NEEDLE 18G)

Fig. 1. Specimen prepared for chemical composition determination

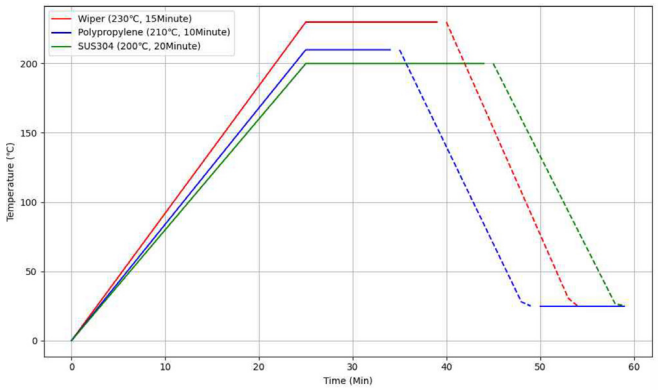


Fig. 2. Operating Temperature and Time of Microwave Digestion by Matrix

crowave operation, immediate opening could cause explosive pressure release, while ongoing reactions may result in incomplete digestion or sample loss. Therefore, the vessels were cooled for at least 30 minutes to reduce internal pressure to atmospheric levels and stabilize residual reactions, ensuring safe handling and reliable analysis [19].

After microwave dissolution, a hydrophilic filter was placed in a funnel, moistened with ultrapure water, and tightly fitted before the original solution was passed through slowly to remove fine impurities such as silica. Subsequently, elemental analysis was performed using ICP-OES. The ICP-OES analysis was carried out by constructing calibration curves with standard reference solutions, followed by quantitative determination that considered the dilution factor of the prepared sample solutions. The concentration was then calculated according to the following equation [20].

$$\text{Concentration of raw materials (ppm, mg L}^{-1}\text{)} = \text{ICP-OES results (ppm, mg L}^{-1}\text{)} \times \frac{\text{Weight of the stock solution used (g)}}{\text{Mass of the original sample (g)}} \times \frac{\text{Total amount of sample for analysis (g)}}{\text{Aliquot of stock solution (g)}}$$

2.4. Elements subjected to ICP-OES analysis

The chemical composition elements analyzed are shown in Table 2. All elements included in the ICP-OES standard solutions were analyzed after constructing calibration curves. Specifically, 24 elements from the multi-element standard solution and 7 elements from the single-element standard solution were included, resulting in a total of 31 elements being evaluated.

3. Results

In this study, experiments were conducted to investigate the chemical composition distribution of the matrices—wiper, polypropylene, and SUS304—after dissolution. The experimental results were intended to be evaluated through comparison with available literature data for each matrix; however, for wiper and polypropylene, except for metallic components, relevant literature data were not available, and therefore direct numerical comparison of chemical compositions could not be performed. Instead, the evaluation was carried out by identifying possible characteristics originating from the raw materials, additives, and other factors involved in the manufacturing process of the original matrices. Furthermore, based on the qualitative ICP-OES analysis results, the necessity for additional pretreatment processes either before or after dissolution was assessed for each matrix.

3.1. Wiper Composition Analysis Results

The non-woven wiper is primarily composed of polyester and cellulose-based synthetic fibers or natural–synthetic blends, consisting mainly of C, H, and O, and theoretically does not contain metallic elements [21]. However, depending on its intended application, metal-based additives such as TiO_2, ZnO may be used to provide wrinkle resistance and antibacterial functions, which may result in the detection of Ti. In addition, when a small amount of polypropylene is blended to reinforce fiber bonding, Ca and Mg components originating from polypropylene may be detected [22].

Table 2. ICP-OES Calibration Standard Solution Used for Quantitative Evaluation

Category	Multi-Element Standard Solution	Single-Element Standard Solution
Elements	Al, Sb, Ba, Pb, B, Ca, Cd, Cr, Co, Fe, K, Cu, Li, Mg, Mn, Mo, Na, Ni, P, Si, Ti, V, Zn	Re, Sr, Y, Ce, Sn, Bi, Th
Concentrations (mg L^{-1})	100	1,000

Table 3. Analytical results of the chemical composition of the ASTAR ULTRA 3000S non-woven wiper

Elements	Measurement value	
	weight percent (wt%)	Concentrations (mg L^{-1})
Ca	29.4	371±96
Fe	2.7	34±2
Mg	6.9	87±1
Na	2.8	35±9
Ti	58.2	735±2

As shown in Table 3, the chemical composition analysis of the wiper used in this study revealed the presence of Ca, Fe, Mg, Na, and Ti. These elements may originate from the manufacturing process or be introduced during use, and can act as interfering elements in radionuclide analysis during chemical separation. Among them, Ti and Ca were detected at the highest concentrations and were found to influence the separation of Sn, Th, and Ra [23].

For Ti, Sn can exist in the +2 or +4 oxidation states, with Sn(IV) exhibiting similar chemical behavior to Ti(IV). As a result, ^{113}Sn may co-precipitate with Ti matrices or be adsorbed onto TiO_2, reducing recovery and leading to underestimation in gamma spectrometric analysis. Furthermore, ^{232}Th exhibits strong binding affinity with TiO_2, which can facilitate selective adsorption; however, excessive Ti concentrations may cause competitive adsorption and redistribution with Th, thereby decreasing recovery. In the case of ^{226}Ra, which is typically separated using its chemical similarity to Ba^{2+}, the presence of Ti colloids may lead to Ra^{2+} adsorption on Ti surfaces or filtration losses, reducing recovery efficiency [24].

For Ca, although it does not share strong direct chemical similarity with Sn, excessive Ca may cause the co-precipitation of Sn through the formation of Ca-carbonates and Ca-phosphates, lowering recovery. Similarly, Th may be incorporated into Ca-phosphates and Ca-fluorides, while an excess of Ca^{2+} in ion-exchange resins can reduce Th selectivity, thereby decreasing separation efficiency [25].

3.2. Polypropylene Composition Analysis Results

Polypropylene is a polymer of propylene (C_3H_6) and is fundamentally an organic macromolecule composed only of carbon and hydrogen, and therefore theoretically contains no metallic elements [26]. However, industrial-grade polypropylene products may contain trace metallic residues originating from catalysts or additives used during manufacturing. The use of Ziegler–Natta or metallocene catalysts can leave residual metals such as Ti, Al, and Mg, while fillers such as talc or $CaCO_3$, added to improve heat resistance and mechanical strength, are major contributors to the detection of Ca and Mg [27].

Table 4. Analytical results of the chemical composition of KOVAX-SYRINGE (Polypropylene)

Elements	Measurement value	
	weight percent (wt%)	Concentrations (mg L^{-1})
Ca	89.0	1,390±13
Mg	11.0	172±0.5

As shown in Table 4, the chemical composition analysis performed in this study revealed the presence of Ca and Mg in the polypropylene samples, which can be attributed to manufacturing additives and catalyst residues. These elements can act as interfering elements during the chemical separation of ^{113}Sn, ^{232}Th, and ^{226}Ra [28].

Among them, Ca^{2+}, detected at the highest concentration, can form Ca-carbonate or Ca-phosphate precipitates in acidic solutions, causing co-precipitation with ^{113}Sn and ^{232}Th and thereby reducing recovery. Excess Ca^{2+} can also complicate the filtration and separation processes. In addition, since ^{226}Ra exhibits chemical similarity to Ca^{2+}, higher concentrations of Ca^{2+} decrease the selective co-precipitation efficiency of Ra^{2+}, leading to simultaneous precipitation of Ra and Ca and significantly reducing the reliability of quantitative results [29].

Meanwhile, Mg^{2+} can induce unintentional co-precipitation of ^{113}Sn during separation by forming Mg-carbonates or Mg-hydroxides. At lower acid concentrations, $Mg(OH)_2$ precipitates may form, adsorbing Sn and resulting in uneven recovery. Although ^{232}Th can be selectively separated due to its high charge density, the presence of high Mg^{2+} concentrations may competitively interfere in ion-exchange resins, lowering the distribution coefficient (Kd), and Th may be entrapped in Mg-phosphate precipitates, reducing recovery and increasing quantitative uncertainty. For ^{226}Ra, in the Ra–Ba co-precipitation method, Mg^{2+} disturbs co-precipitation equilibrium, and under high concentrations, interferes with the homogeneous incorporation of Ra into the precipitate lattice, thereby decreasing analytical precision [30].

3.3. SUS 304 Composition Analysis Results

The results of chemical composition analysis after dissolution

Table 5. Analytical results of the chemical composition of SUS 304

Elements	JIS/ASTM specifications	Measurement value	
	weight percent (wt%)	weight percent (wt%)	Concentrations (mg L^{-1})
Sb	-	0.4	4,735±326
B	-	0.3	3,364±130
Cr	18.0-20.0	19.5	211,182±1,027
Fe	Balance	69.3	749,614±2,408
Mn	≤2.0	1.2	12,638±799
Ni	8.0-10.5	8.6	93,128±1,011
Mg	≤2.0	0.0	101±0.3
P	≤0.045	0.1	580±33
Si	≤1.0	0.1	642±14

of the SUS304 samples are presented in Table 5. The detected elements included Sb, B, Cr, Fe, Mn, Ni, Mg, P, and Si, and the major components were found to be almost consistent with the specifications of ASTM A240 Type 304 and JIS G 4303, confirming that dissolution was appropriately performed [31]. However, Sb and B lacked sufficient comparable literature data, yet their significant detection concentrations suggest that they should be considered as potential interfering elements in subsequent chemical separation processes. In particular, Sb can disturb the adsorption and desorption behavior of commercial ion-exchange resins such as Sr, TRU, and TEVA (TEtraValent Actinide Resin) resins, and due to its chemical similarity to Ru, Mo, Zr, and Sn, it may act as an interfering element during radionuclide analysis [32].

The major metallic components of SUS304, namely Fe, Cr, Ni, and Mn, can act as interfering elements in the chemical separation of ^{113}Sn, ^{232}Th, and ^{226}Ra. Fe strongly adsorbs to ion-exchange resins during the separation of ^{113}Sn, distorting distribution behavior, while $Fe(OH)_3$ colloid formation leads to co-precipitation of Sn, thereby reducing recovery. For ^{232}Th, competitive adsorption with Fe^{3+} and co-precipitation losses lower its distribution coefficient, and in the case of ^{226}Ra, Fe interferes with sulfate or phosphate precipitation or promotes colloidal adsorption, thereby reducing Ba–Ra co-precipitation efficiency [33].

Cr exhibits different effects depending on its oxidation state. Cr^{3+} causes co-precipitation of ^{113}Sn and ^{232}Th in $Cr(OH)_3$ precipitates or competes in ion-exchange resins, reducing recovery, whereas Cr^{6+} oxidizes Sn(II), altering its chemical behavior, and disrupts the redox environment of Th, making its distribution properties unstable. Furthermore, Cr^{3+} reduces separation efficiency in the ^{226}Ra–Ba co-precipitation process by altering precipitate crystallinity and induces Ra losses through colloidal adsorption [34].

In the case of Ni, excess Ni^{2+} leads to competitive adsorption in ion-exchange resins or co-precipitation with $Ni(OH)_2$, lowering the recovery of ^{113}Sn. For ^{232}Th, competition with Ni^{2+} reduces separation efficiency, while for ^{226}Ra, precipitation of $NiSO_4$ and $NiCO_3$, as well as adsorption onto Ba–Ra precipitate surfaces, results in recovery losses [35].

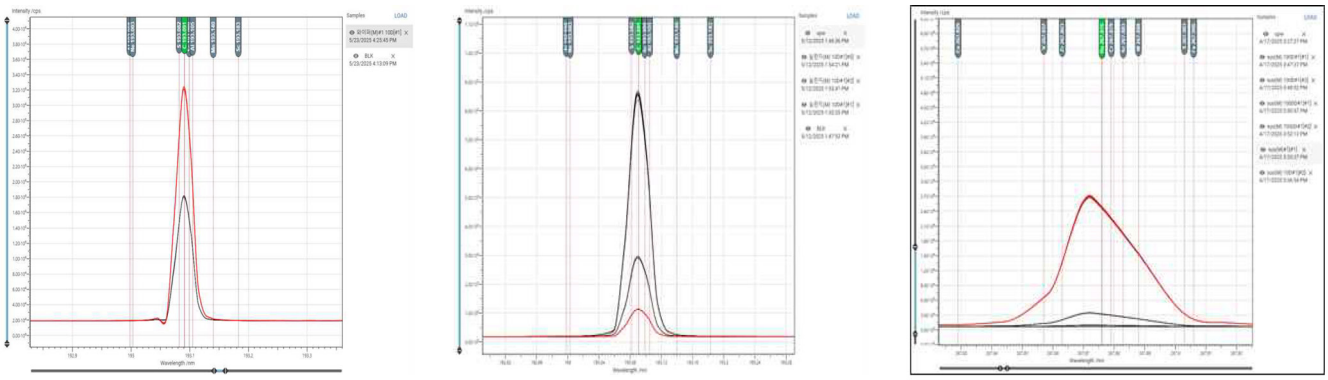
Finally, Mn interferes with ^{113}Sn separation by competitively interacting in ion-exchange resins or through the formation of MnO_2 precipitates that adsorb Sn, leading to losses. Similarly, ^{232}Th separation is hindered by competitive adsorption of Mn^{2+} and entrapment in MnO_2 precipitates, while ^{226}Ra is affected by competitive interactions with Mn^{2+} and adsorption onto MnO_2 surfaces, thereby decreasing Ba–Ra co-precipitation efficiency [37].

3.4. Microwave-Assisted Solutionizing Results

The microwave digestion method applied for matrix dissolution in this study was demonstrated to be effective in terms of reduced processing time, improved reproducibility, and preservation of volatile elements compared with conventional wet digestion methods [37]. Using sealed vessels, microwave digestion created high-temperature and high-pressure conditions that enabled complete decomposition of fiber-based wipers, refractory-structured polypropylene, and SUS304 alloy, which possesses high corrosion resistance due to its Cr_2O_3 passive film. Moreover, sealed digestion minimized the loss of volatile elements such as Sn, Ra, B, and Sb, thereby maintaining analytical reliability. The ICP-OES results, which were consistent

(a) Assembly of the microwave vessel and rotor (b) Wiper (c) Polypropylene (d) SUS304

Fig. 3. Microwave system and procedure for matrix-specific dissolution



(a) ASTAR ULTRA 3000S non-woven wiper (b) KOVAX-SYRINGE (Polypropylene) (c) SUS304 (KOVAX-NEEDLE 18G)

Fig. 4. Qualitative Spectral Analysis Results by Medium Using ICP-OES

with reported literature values and alloy specifications, further supported the applicability of this method [8]. Therefore, microwave digestion is considered an effective pretreatment approach for radioactive waste matrices to ensure chemical completeness and to minimize recovery losses and quantitative distortions in subsequent chemical separation processes.

3.5. Results of Qualitative ICP-OES Analysis for Different Media

Qualitative analysis using ICP-OES is employed as an exploratory technique to identify which elements are present in a sample. Even without standard reference materials, the presence of a specific element can be confirmed if its characteristic emission line is observed in the sample. Although precise concentration determination is difficult, comparing peak intensities allows the inference of relative abundance or compositional trends among elements. This method also serves as a rapid screening tool to investigate the elemental composition of complex mixtures [21].

For the matrices analyzed in this study, the qualitative ICP-OES results (Fig. 4) revealed distinct peaks at the characteristic wavelength of carbon (C) at 193.091 nm for both the wiper and polypropylene samples, while the SUS304 sample exhibited a peak at the characteristic wavelength of ruthenium (Ru) at 267.876 nm [38].

These findings suggest that in the case of the wiper and polypropylene, residual organic matter may destabilize nebulization, plasma loading, and background correction in ICP-OES, thereby interfering with the response and recovery of trace radionuclides. For SUS304, the Fe-Cr-Ni-rich matrix is likely to act as a strong chemical and spectral interferent for most radionuclides [39].

Therefore, it is considered effective to apply additional pretreatment processes, such as ashing, to remove organic matter prior to dissolution for wiper and polypropylene samples, while for SUS304, chemical separation procedures combined with solvent extraction for the removal of high concentrations of Fe should be applied to improve recovery and ensure the reliability of quantitative radionuclide analysis.

4. Discussion

In this study, three different matrices (wiper, polypropylene, and SUS304) were analyzed after dissolution to evaluate their chemical composition and potential interfering factors during radionuclide separation.

The wiper, being a fiber-based matrix with low intrinsic metal content, showed detectable amounts of Ti, Ca, and Mg introduced during manufacturing. These elements were identified as interfering components in the chemical separation of ^{113}Sn, ^{232}Th, and ^{226}Ra by inducing co-precipitation, adsorption, and distribution coefficient distortions, ultimately reducing recovery and analytical reliability. In particular, Ti was a major interfering factor in the analysis of Sn and Th, while Ca significantly affected the separation of Sn and Ra.

Polypropylene, as a polymeric matrix, is theoretically free of metals; however, significant amounts of Ca and Mg were detected, attributable to additives and catalysts used during production. Ca interfered with the separation of Sn and Th through co-precipitation and hindered Ra separation due to its chemical similarity to Ra. Mg caused additional losses of Sn and Th via carbonate and hydroxide precipitation and reduced the efficiency of the Ra–Ba co-precipitation method. These results suggest that the removal or correction of Ca and Mg is essential in the analysis of polypropylene-based matrices.

SUS304, a metallic alloy matrix, exhibited strong matrix effects due to the leaching of major alloy constituents such as Fe, Cr, Ni, and Mn. Fe interfered with the separation of Sn, Th, and Ra through co-precipitation and adsorption. Cr, depending on its oxidation state, disrupted the redox environment, thereby complicating Sn and Th analysis. Ni interfered with Sn, Th, and Ra separation by competitive ion-exchange adsorption and precipitation formation, while Mn caused losses of Sn, Th, and Ra through the formation of MnO_2 precipitates. Thus, the multicomponent metallic matrix of SUS304 demonstrated strong chemical and spectral interferences, necessitating selective removal procedures or solvent extraction techniques.

Qualitative ICP-OES analysis confirmed the presence of major elements in each matrix. In the case of organic matrices (wiper and polypropylene), residual organics induced instability, while in the metallic matrix (SUS304), the abundance of metallic elements caused strong interferences. Therefore, applying matrix-specific pretreatment procedures—such as ashing, selective removal, or correction—is crucial for improving analytical reliability.

Overall, this study experimentally demonstrated that matrix-specific chemical background constituents in various media can act as interference elements in radionuclide analysis, highlighting the necessity of pretreatment and chemical separation strategies tailored to matrix characteristics. These findings indicate that accurate chemical separation and quantitative radionuclide analysis of radioactive isotope waste can be achieved by identifying the chemical composition and elemental concentrations of the target matrix and by accounting for the behavioral interactions between target radionuclides and interference elements.

5. Conclusions

For radiological characterization of radioactive waste, radionuclide identification and quantitative determination of radioactivity through radionuclide analysis are essential. Radioactive waste matrices are highly diverse, and each exhibits distinct chemical compositions. Such diversity introduces interfering elements that complicate the application of radiochemical separation techniques for isolating target radionuclides.

Therefore, the quantitative and qualitative analysis of chemical compositions obtained in this study through complete decomposition of elements (both organic and inorganic) in various matrices—wiper, polypropylene, and SUS304—provides fundamental data to ensure the integrity of liquid samples used in subsequent radiochemical separation processes following pretreatment.

In addition, since radioactive waste matrices include a broader variety of materials beyond those examined in this study—such as rubber, vinyl, cotton, plastics, and steel—future studies should aim to analyze the chemical compositions of these additional matrices and to generate data that reflect the behavior of specific elements in chemical separation. Such data will be crucial for improving the precision and applicability of radiochemical separation technologies across the full spectrum of radioactive waste matrices.

Acknowledgments

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