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Alpha source preparation using electrodeposition method and characterization by SEM-EDS

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HIGHLIGHTS

- Alpha sources prepared via electrodeposition for alpha spectrometry.
- Stainless steel discs used as cost-effective and practical electrode substrates.
- Optimized parameters: current density, pH, and deposition time for quality layers.
- SEM-EDS analysis was employed to assess uranium distribution on the steel surface.

ABSTRACT

This study investigates the preparation of alpha-emitting sources using the electrodeposition technique for alpha spectrometry. Since source preparation is often costly and may involve the use of enriched radionuclides, highly efficient methods are essential. Among the available techniques, electrodeposition is one of the most widely employed methods for producing alpha sources. In this work, uranium layers were deposited onto stainless steel discs using the electrodeposition method. Key deposition parameters, including anode material (platinum wire with defined geometry), cathode material (polished stainless steel), current density (0.5 A.cm^{-2}), electrolyte pH (2-2.4), and deposition time (60 minutes), were carefully selected and applied. Following the preparation of the alpha sources, their characterization was performed using alpha spectrometry with a semiconductor detector and Scanning Electron Microscopy coupled with Energy-Dispersive X-ray Spectroscopy (SEM-EDS). Alpha spectrometry confirmed the presence of U-238, U-235, and U-234 isotopes, and SEM-EDS analysis was employed to evaluate the uranium distribution on the disc.

KEYWORDS

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1 Introduction

The accurate identification and measurement of alpha-emitting radionuclides are of significant importance in various fields, including environmental studies, nuclear safety monitoring, and nuclear metrology applications (Arimoto et al., 2005; Bolívar et al., 2009; Crespo et al., 2003; Danesi et al., 2003; Rentería Villalobos et al., 2007; García-Toraño et al., 2005). In this context, alpha-particle spectrometry plays a key role as an efficient and highly sensitive technique. However, achieving accurate spectroscopic results requires the preparation of alpha sources with desirable characteristics, including a thin layer, homogeneous distribution, and high radiochemical purity. Several techniques have been developed to prepare alpha sources. These techniques include evaporation, microcoprecipitation, and electrodeposition (Vajda et al., 2020). Each of these methods is described in numerous references (Crespo, 2012; Jobbágy et al., 2013; Ko, 2020; Lally and Glover, 1984; Vajda et al., 2020). Among these methods for preparing alpha sources, electrodeposition is widely used due to its simplicity, parameter controllability, and the ability to produce uniform sources (Da Cruz and Poledna, 1990; Jobbágy et al., 2013; Panta et al., 2010). In the electrodeposition method, alpha-emitting nuclide, specifically actinide elements, is electrochemically plated onto a polished metal backing from either an organic or an aqueous electrolyte solution. The conventional setup for the electrodeposition typically involves a cylindrical cell, which is commonly made of materials such as Teflon, polyethylene, or plastic vials. Within the cell, a planar metallic cathode with a diameter of approximately 20-30 mm is positioned at the bottom, along with an anode (Crespo, 2012).

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This research aims to prepare sources for alpha-particle spectrometry. Electrodeposition was selected as the preparation method due to its proven track record and ease of implementation. However, the electrodeposition process is influenced by a multitude of parameters, including the cathode material and its surface condition, the anode material and its geometry, the applied current density, the pH of the electrolyte solution, and the deposition time (Crespo, 2012; dos Santos et al., 2004; Klemenčič and Benedik, 2010; Martin and Hancock, 2004; Maya et al., 2004; Salar Amoli et al., 2006). To optimize source quality, findings from previous studies that had investigated the impact of each of these parameters were utilized. These findings were then carefully considered in the design and execution of the experimental setup. Key observations from those previous investigations that guided this work are summarized below:

- Anode material and its geometry: Platinum is a common anode material in electrodeposition systems. Anode geometry plays a crucial role, not only by facilitating the evolution of hydrogen gas generated at the cathode and oxygen produced at the anode (Crespo, 2012), but also by influencing the uniformity of the deposited layer. Studies (Jobbágy et al., 2013; Klemenčič and Benedik, 2010) indicate that spiral and mesh anode designs yield superior material distribution compared to rectangular configurations, which tend to concentrate the deposition in the central region.

- Cathode material and its surface condition: To achieve high deposition yields and narrow FWHM values in alpha source preparation, various cathode disc materials have been investigated, including stainless steel, aluminum, platinum, titanium, silver, and copper (Bond et al., 2013; Henderson et al., 2011; Trdin et al., 2012). While platinum and titanium exhibited superior performance with high deposition yields and small FWHM, copper and aluminum demonstrated lower yields and broader FWHM. Stainless steel provides a practical compromise, offering a balance of performance, cost-effectiveness, and disposability, making it a suitable choice for routine applications (Ko, 2020). Moreover, substrate surface quality significantly influences the outcome; polished surfaces are crucial for minimizing self-absorption and maximizing spectral resolution by facilitating uniform and thin deposits (Crespo, 2012; Maya et al., 2004; Weber et al., 1999).

- Current density: Electrodeposition necessitates a delicate balance in current density. While a hydroxyl layer is crucial for precipitating insoluble hydroxides and facilitating actinide deposition, exceeding a critical current can thicken this layer, and reducing deposition yield (Crespo, 2012). Adhering to standards such as the ASTM C1284-05 recommendation of approximately 0.5 A.cm^{-2} aids in achieving this balance, promoting efficient and uniform deposition for high-quality alpha sources.

- PH of the electrolyte solution: Electrodeposition demands careful control of electrolyte pH to optimize deposition yield. The process is sensitive to electrolyte pH, requiring careful control to prevent re-dissolution or complex formation. A common strategy involves adjusting the electrolyte pH to a slightly acidic range and then briefly shifting to alkaline conditions just before terminating the process to mitigate uranium re-dissolution from the prepared source (Jobbágy et al., 2013; Maya et al., 2004).

- Deposition time: Across various studies (Kressin, 1977; Lee and Lee, 2000; Oh et al., 2014), actinide electrodeposition typically requires 60 to 120 minutes to achieve deposition yields ranging from 50 to 100%.

2 Experimental procedure

Figure 1 briefly illustrates the steps of the electrodeposition method used for alpha source preparation, following ASTM standard practice C1284-05. Each section is explained in more detail below.

2.1 Preparation of the backing and setup of the electrodeposition cell

Considering the effect of anode materials and geometry on the electrodeposition process, a platinum wire with a 1.5 mm diameter and 100 mm length was used as the anode in this study. The wire had several holes in its circular cross-section to facilitate the evolution of hydrogen gas generated at the cathode and oxygen at the anode (see Fig. 2-a). To comply with the standard, the circular section at the end of the anode was slightly smaller than the cathode. Although previous studies have reported that spiral or mesh anode geometries may provide improved material distribution, the present study did not aim to perform a comparative evaluation of different anode designs. The selected anode configuration was adopted based on practical considerations and the available experimental setup.

A stainless steel disc was used as the backing material. As previously mentioned, a glossy surface is crucial for deposition quality, affecting source thickness and homogeneity. Therefore, the preparation of the backing involved several steps. Initially, the protective plastic tape was removed from the disc surface. Subsequently, the surface was polished using diamond polishing paste, rinsed with acetone, and cleaned with deionized water to remove any remaining grease. Figure 2-b depicts a thoroughly cleaned and polished stainless steel disc. The cathode is positioned within a plastic vial (falcon tube) that serves as the electrodeposition cell. The assembled cell (see Fig. 3) was filled with deionized water and visually inspected for leaks.

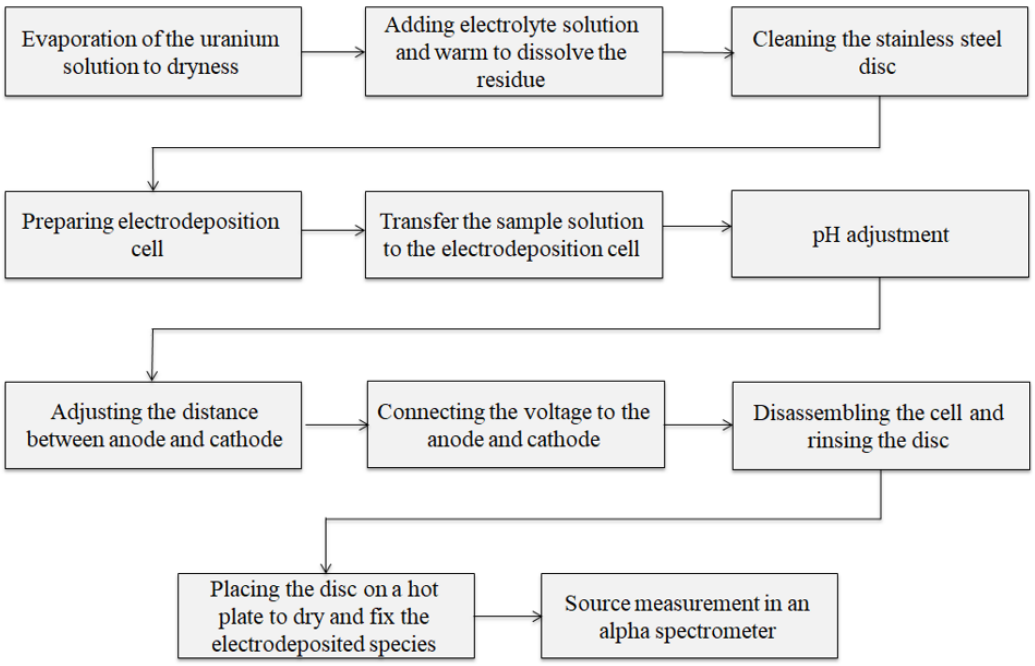

Figure 1: Flow chart of the electrodeposition process.

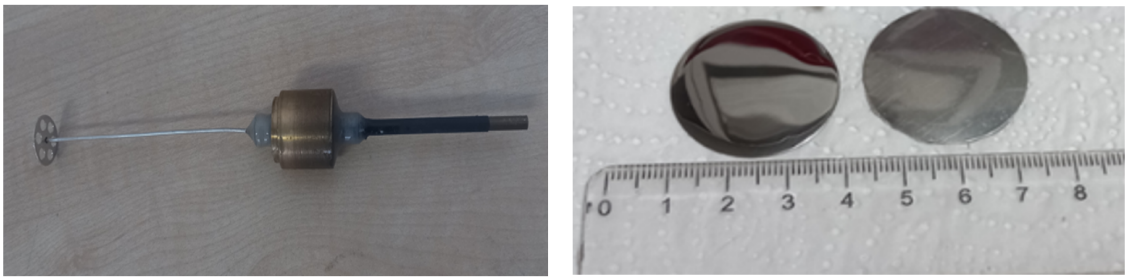

Figure 2: (a) Pt anode used for electrodeposition and (b) stainless steel disc before (right) and after (left) polishing.

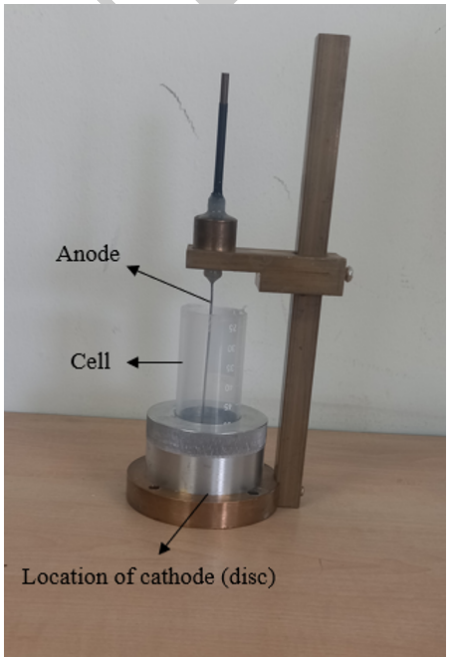

Figure 3: The simple setup of the electrodeposition cell.

2.2 Preparation of the sample solution and the electrodeposition process

A 1 ml aliquot of uranyl nitrate solution was transferred to a glass beaker and placed on a hot plate. The solution was evaporated to dryness, and then 2 ml of concentrated HNO_3 and 0.5 ml of concentrated H_2SO_4 were added. The beaker was returned to the hot plate, and the solution was evaporated until only a few drops remained. The beaker was then removed from the heat and allowed to cool slightly. Next, 2-3 drops of an aqueous reagent containing thymol blue were added to the beaker. The inner wall of the beaker was rinsed with 3 ml of distilled water. While stirring, concentrated NH_4OH solution was gradually added to adjust the pH to approximately 4.

The sample solution was transferred into the electrodeposition cell. The beaker was rinsed three times with 2 ml of 1:99 H_2SO_4 each time, and the rinsings were added to the cell. The pH of the solution was measured using a digital pH meter, which was calibrated prior to measurement (Fig. 4-a). If the pH was below 2, NH_4OH solution was slowly added to adjust it to the range of 2-2.4 (Fig. 4-b).

Figure 4: (a) pH meter calibration and (b) sample solution pH adjustment.

Table 1: Settings applied for the electrodeposition.

Parameters	Optimized value
Deposition time (min)	60
Current (A)	0.96
Voltage (V)	<10
Source diameter (mm)	30
Deposition diameter (mm)	16
Cathode to anode distance (mm)	10
Backing material	Stainless steel

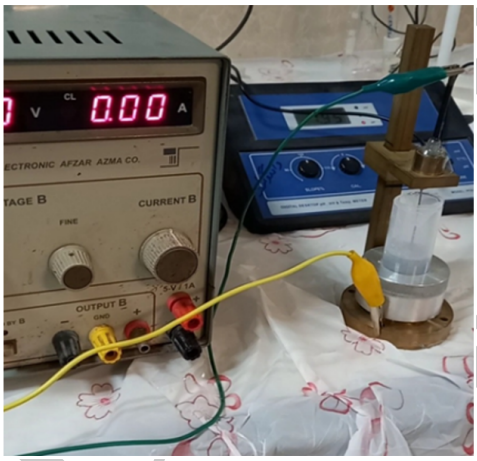

Figure 5: Experimental setup of the electrodeposition cell.

Figure 6: Sources prepared using electrodeposition.

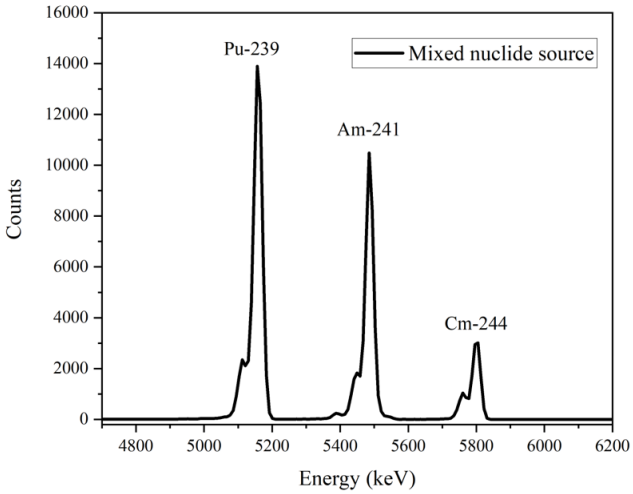

Figure 7: Energy spectrum of the mixed nuclide energy calibration source obtained with the ion-implanted Si detector.

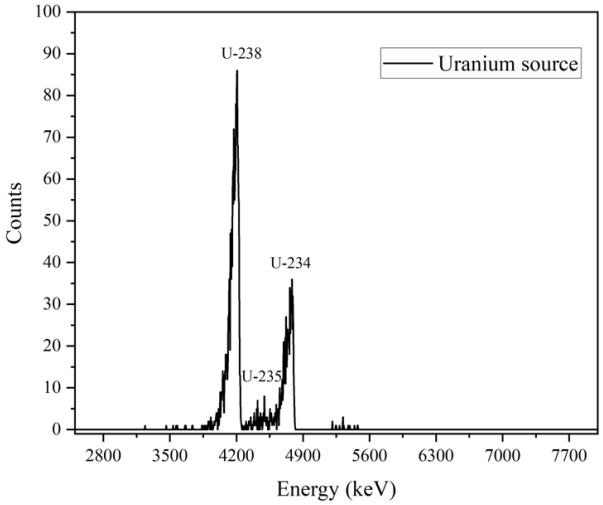

Figure 8: Energy spectrum of uranium source obtained with ion-implanted Si detector.

As previously mentioned, the electrolyte pH significantly affects the electrodeposition yield. At a low pH (< 2), the deposited uranium layer can redissolve into the electrolyte. Conversely, at higher pH values (> 4), uranium forms hydroxide complexes with varying stoichiometry, hindering its deposition (Jobbágy et al., 2013).

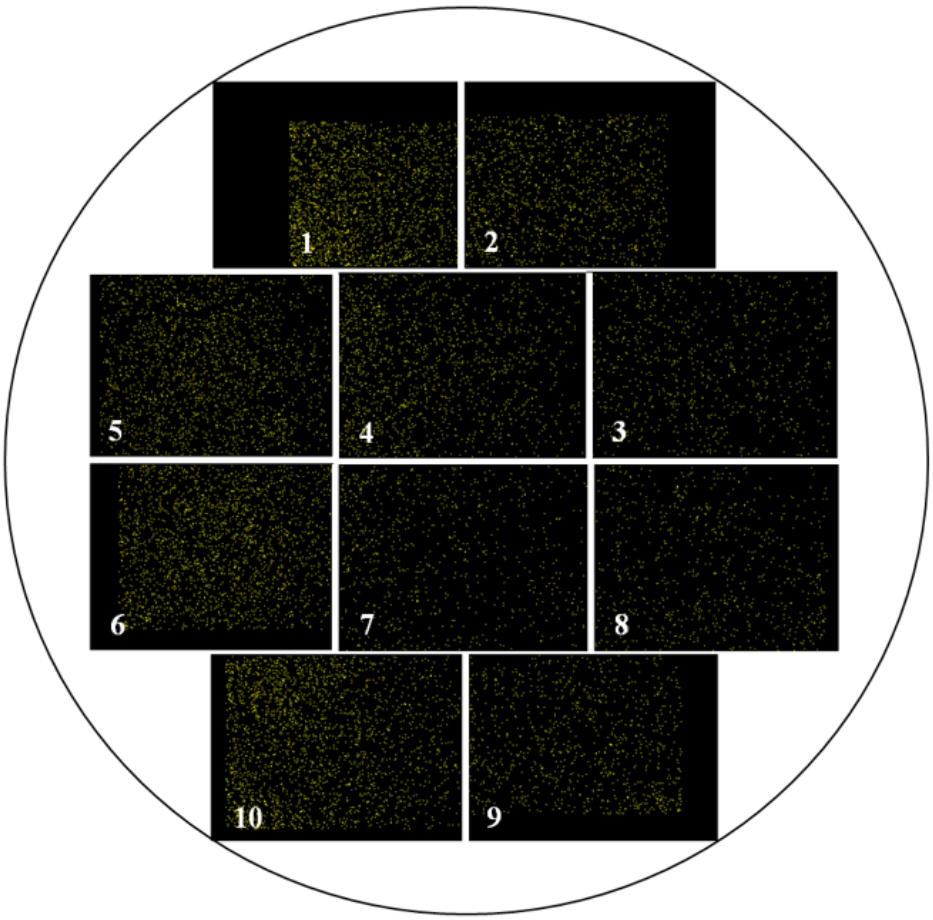

Figure 9: SEM-EDS mapping of uranium on stainless steel substrate.

The platinum anode was inserted into the solution, positioned 1 cm from the stainless steel backing cathode. The experimental arrangement for the electrodeposition process is shown in Fig. 5. To achieve a current density of approximately 0.5 A.cm^{-2} over the disc area, the electric current was adjusted to 0.96 A. The cathode and anode were connected to the base of the electrodeposition cell and the platinum anode, respectively. Electrodeposition was carried out at a constant current for 60 minutes. Then, 1 ml of concentrated NH_4OH was added to the cell, and electrodeposition continued for an additional minute. The addition of concentrated NH_4OH causes an alkaline pH shift, which prevents uranium redissolution from the deposit (Jobbágy et al., 2013).

Several preliminary electrodeposition experiments were performed to optimize the preparation conditions. Different deposition times and uranium solution amounts were evaluated to determine the most suitable parameters for obtaining acceptable alpha sources. After finalizing the deposition conditions, multiple uranium sources were prepared under identical experimental settings to verify the reproducibility of the process. The obtained results were consistent, confirming the reliability of the selected parameters. The electrodeposition settings are listed in Table 1.

After removing the anode and turning off the current, the solution was discharged. The electrodeposition cell was then disassembled, and the disc was rinsed with distilled water. The disc was placed on a hot plate set at 200 °C to stabilize and dry the electrodeposited material. Figure 6 depicts the sources prepared using this method; the deposited material is well-stabilized on the backing surface.

3 Characterization of the source

3.1 Alpha spectrometry

A vacuum chamber with an ion-implanted Si detector was used to measure the source. The measurement setup included a preamplifier and a digitizer (CAEN DT5724). The digitizer directly sampled the preamplifier output. The MC^2 software recorded the energy of each detected event in a text file. A mixed nuclide source containing Pu-239, Am-241, and Cm-244 was used to calibrate the spectrometer. The energy spectrum of the mixed nuclide energy calibration source obtained with the ion-implanted Si detector is shown in Fig. 7. The energy resolution for the energy of 5485.5 keV from Am-241 was determined to be 23 keV.

The prepared source was then positioned within the vacuum chamber, and data was collected. The measured spectrum is shown in Fig. 8.

Figure 8 clearly shows the distinct peaks corresponding to the isotopes U238, U235, and U234 at 4198, 4395,

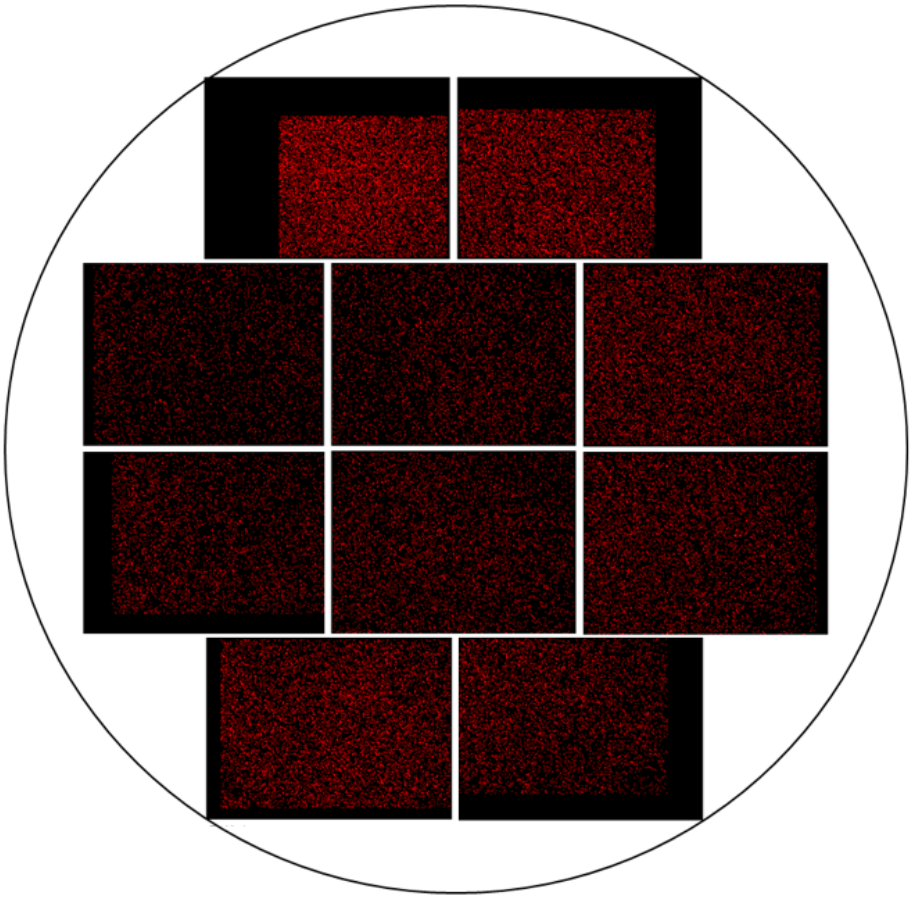

Figure 10: SEM-EDS mapping of substrate iron distribution.

and 4774 keV energy lines, respectively, with an estimated energy uncertainty of several keV based on the calibration procedure. Additionally, the energy resolution for the 4198 keV energy from U-238 was observed to be about 30 keV. To obtain the required statistics in the counting, a longer measurement time is essential.

3.2 SEM-EDS analysis

Scanning Electron Microscopy (SEM) and Energy Dispersive x-ray Spectroscopy (EDS) allow for targeted analysis of sample surfaces. These techniques are widely used for material surface analysis. SEM equipped with an EDS detector shows the dispersity of elements on the surface of the alpha-source layer (Ko, 2020). To investigate the distribution of the uranium on the surface of the layer, 10 areas were selected, and SEM-EDS analysis was subsequently conducted on them. The results of the analysis, which illustrate the uranium distribution on the surface, are presented in Fig. 9.

As shown in the Fig. 9, the uranium is deposited across the circular surface; however, a higher uranium distribution is observed on the left side of the disc. The non-uniformity in the distribution can be attributed to surface irregularities or asymmetric flow during the electrodeposition process.

The distribution of iron (as the primary substrate element) is illustrated in Fig. 10. The results indicate that in areas where the distribution of uranium is lower, a higher relative amount of iron has been observed. This phenomenon is attributed to the reduced deposition of uranium in these regions, which leads to an increase in backscattered signals from the substrate (iron). This phenomenon is attributed to the reduced deposition of uranium in these regions, allowing the electron beam to penetrate through the thin or discontinuous layer with more energy. This increases the characteristic X-ray emission from the iron and reduces the attenuation of these X-rays through the deposited uranium.

4 Conclusions

The successful preparation and characterization of alpha sources via the electrodeposition method were demonstrated, achieving reliable uranium deposition on stainless steel discs. Alpha spectrometry confirmed the presence of U-238, U-235, and U-234 isotopes, while SEM-EDS analysis verified the effective distribution of uranium across the substrates. These findings highlight the reliability of the electrodeposition technique for producing alpha sources. Future studies will extend this work using complementary analytical methods, such as Rutherford Backscattering Spectrometry (RBS), to further investigate the composition and surface properties of the electrodeposited layers, thereby optimizing source performance for alpha spectrometry.

Conflict of Interest

The authors declare no potential conflict of interest regarding the publication of this work.

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