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Analysis of filters in the recovery of Cs, Co, U, and Th isotopes through chemical extraction

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Abstract

This study aims to analyze the recovery yield of radioisotopes using the hot acid extraction method as an alternative to conventional dissolution and calcination techniques. Various filters with different characteristics were evaluated for their potential use in the characterizing radioactive waste removed via ablation probes. The target elements in this research were ^{60}Co, ^{137}Cs, ^{238}U, and ^{232}Th, as these isotopes are commonly found in nuclear facilities. The results indicated that the polyethersulfone membrane filter is effective for the analysis in question.

Keywords Radioactive waste · Laser ablation · Chemical extraction · Filters

Introduction

In recent years, there have been significant developments in the use of nuclear technology across various sectors of society, including scientific research, food preservation, medical diagnosis and treatment, and electricity generation. These applications have brought numerous benefits to society [1]. However, the increasing use of nuclear technology has raised concerns regarding the management of radioactive waste. It is essential to accurately determine the composition of nuclear and radioactive waste—particularly in terms of contamination levels—to ensure they remain low enough not to pose a risk to human health.

The Brazilian National Nuclear Energy Commission (CNEN) establishes criteria for the evaluation and safety of radioactive waste disposal, with waste characterization being the most critical requirement. In the nuclear industry, power plants generate waste that may contain all sort of radionuclides, including gamma emitters such as, ^{60}Co, ^{137}Cs, ^{125}Sb, ^{129}I, etc., alpha emitters such as U, Am, Pu isotopes, and beta emitters such as ^{55}Fe, ^{63}Ni, ^{99}Tc [2]. In addition to compacted tailings, large structures like steam generators, when in disuse, have contaminated surfaces and must therefore also undergo proper characterized [3].

The common characterization methods typically involve chemical and abrasive attack that generate by-products

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which can themselves become additional forms of radioactive waste, particularly in liquid form [4]. One alternative currently being tested for the treatment and characterization of radioactive waste is laser ablation. Researchers at the Nuclear Energy Research Institute (IPEN) are developing a laser ablation probe to decontaminate surfaces. In its current configuration, a laser beam is directed at the contaminated surface, and the resulted ablated material is collected via an exhaust system and retained on paper filters (Fig. 1) [5].

The material retained on the filters serves as a sample for characterizing the radioactive waste, meeting one of the key criteria established for the safe disposal of radioactive material, while maintaining the waste in solid form. For this reason, it became necessary to conduct a study to evaluate the chemical extraction yield of different filters, in order to identify the most suitable filter for use in the laser ablation probe.

The two most commonly used methods for filter analysis are calcination and total acid dissolution. The calcination method is limited by the volatility of some elements, which can lead to significant sample loss during analysis. On the other hand, total dissolution of the filter with acid can sometimes be a labor-intensive process due to the physical resistance of the filters [6].

Different filter membranes exhibit varying behaviors when subjected to acid dissolution. Composite membranes consist of an active layer responsible for selective separation and a support layer made of a thicker, porous substrate that provides mechanical strength without contributing significantly to selectivity during filtration [7]. On the other hand, symmetric membranes have a uniform structure throughout, which performs both mechanical and separation functions to a certain extent.

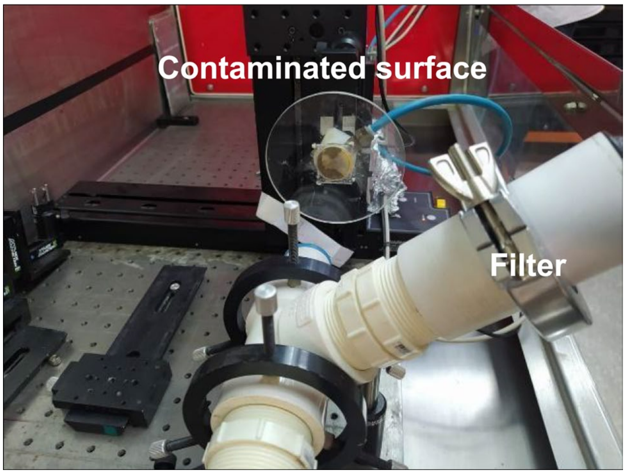


Fig. 1 Assembly diagram of the laser ablation probe, identifying the position of the contaminated surface and the filter that will retain the removed material

The degree of dissolution depends on the chemical composition of the material, with behaviors ranging range from rapid degradation to high resistance against mineral and organic acids. In filters that include a support layer, it may exhibit a greater resistance to acid attacks and may only dissolve in concentrated oxidizing acids, as observed in the case of polysulfone substrates [8].

Determining the total concentration of analytes in a filtration material typically requires acid digestion using concentrated acid or a mixture of concentrated acids, a procedure that is relatively time-consuming. Instead of a total digestion, one alternative is to use a leaching procedure to extract the retained material from the filter. However, this method should be considered an acid leaching rather than a digestion, which cannot replace a determination of the total content of an analyte in all situations [9].

To identify the most suitable filter for use in the laser ablation decontamination device, and to quantify the amount of material retained, this work aimed to evaluate the recovery yield of the radioactive nuclides ^{137}Cs, ^{60}Co, ^{238}U and ^{232}Th using the hot acid extraction method. Various types of filters were tested, and the extraction process was maintained constant using nitric solution (3 mol L^{-1}) at the same temperature and time of extraction in order to obtain total, partial and non-dissolution of the filter.

The radionuclides ^{137}Cs, ^{60}Co, ^{238}U and ^{232}Th were selected because they are commonly found in nuclear waste and can be readily measured using the available infrastructure of the Nuclear Research Center, at the Nuclear and Energy Research Institute, where this study was conducted.

Experimental

Materials

For the extraction tests, standard solutions of radionuclides commonly found in found in radioactive and nuclear installations ^{60}Co, ^{137}Cs, ^{238}U and ^{232}Th were used. The ^{60}Co, ^{137}Cs standards, supplied by the Institute for Radioprotection and Dosimetry (Rio de Janeiro, Brazil) were diluted in nitric acid and had activity concentrations: 789.75 Bq/g, 816.64 Bq/g, respectively. The ^{238}U and ^{232}Th standards, obtained from SPEX CertiPrep (Metuchen, NJ, USA), were diluted in 2% nitric acid and had a total concentration of 1000 µg/mL. Throughout the study, five filters from different manufacturers were tested for their extraction performance. Table 1 shows the specifications for each filter, including composition, porosity, and diameter.

Cellulose and cellulose ester membranes exhibit low resistance in various acidic media, including HNO_3, HCl, and H_2SO_4, whether concentrated or diluted forms. Fiberglass and glass microfiber filters are typically stable in

Table 1 Specifications of the filters used in the chemical extraction tests

Filter	Manufacturer	Porosity (µm)	Diameter (cm)	Composition
Type I	GelmanSciencíens	0.8	25	Cellulose membrane
Type II	Axiva	1.6	47	Fiberglass
Type III	Millipore	0.8	47	Glass microfiber
Type IV	Millipore	0.2	47	Polyethersulfone membrane
Type V	Millipore	3.0	47	Cellulose ester

diluted acid solutions; however, complete dissolution typically requires hydrofluoric acid. Polyethersulfone membranes have demonstrated high stability across a range of acidic environments, including exposure to the concentrated acids mentioned above. Nevertheless, this type of membrane is susceptible to degradation under highly oxidative conditions, such as those found in 70% HNO_3 solutions [10].

Procedure for extraction of ^{137}Cs and ^{60}Co

Twenty microliters of certified ^{137}Cs and ^{60}Co solutions were pipetted onto the different tested filters, which were then dried under an infrared lamp. After drying, the filters were transferred to plastic supports and subjected to gamma spectrometry for the determination of initial activity concentration.

Following this, the filters were transferred to beakers containing 50 mL of 3 mol L^{-1} HNO_3 and subjected to an extraction procedure on a hot plate at 80 °C for 2 h. The temperature was kept constant throughout all experiments. Although temperature effects were not evaluated in this study, it is important to note that certain radionuclides, such as the case of ^{129}I and ^{210}Po, may be lost at higher temperatures, particularly under oxidizing conditions [11, 12].

The extracted material was then filtered and both the beakers and filters were rinsed with Milli-Q water and 3 mol L^{-1} HNO_3 to ensure complete recovery of the analytes. The combined solutions were reduced in volume to approximately 0.5 mL. The final concentrate was transferred onto Whatman filters with the same geometry as the initially used filters, and a second gamma spectrometry analysis was performed to quantify the recovered activity.

Procedure for extraction of ^{238}U and ^{232}Th

In a single 5 mL beaker, 20 µL of certified ^{238}U and ^{232}Th solutions were pipetted and weighed to verify that the dispensed volume corresponded closely to the target concentration. The contents were then transferred onto the different types of filters under evaluation. To ensure complete transfer of the analytes, the beaker was carefully rinsed with a 3 mol L^{-1} HNO_3, and the rinse solution was also applied to the filters.

Next, the filters were dried under infrared light. Once drying, they were placed in beakers containing 50 mL of 3 mol L^{-1} HNO_3. The extraction procedure that followed was identical to the one previously described for ^{137}Cs and ^{60}Co.

Following the extraction procedure and subsequent cooling of the solution, the extract was filtered to retain any residues from the tested filters. The volume of the filtrate was then reduced to approximately 0.5 mL, and the resulting concentrated was transferred onto Whatman filters, which were subsequently dried under an infrared lamp. These filters were then used for neutron activation analysis. To determine the initial activity concentration, a parallel set of filter samples was prepared using the same procedure but without undergoing chemical extraction, and these were also analyzed by neutron activation.

Neutron activation analysis

The filter samples containing ^{238}U and ^{232}Th were irradiated in the core of the IEA-R1 nuclear reactor at IPEN—CNEN/SP for an 8-h cycle alongside filters containing reference standards (SPEX CertiPrep, Metuchen, NJ, USA). In the neutron activation analysis (INAA), both the samples and U and Th standards, each wrapped in polyethylene envelopes, were packed in aluminum foil and then placed in aluminum containers, known as “rabbits”. These aluminum rabbits were then irradiated under a thermal flux of 4.0×10^{12} n cm^{-2} s^{-1} for a duration of 8 h.

The determination of ^{238}U was performed by counting the gamma-ray energy at 277.45 keV, five days after the irradiation. For ^{232}Th the gamma energy at 311.76 keV was counted seven days of the first counting of ^{238}U.

For the measurements, the irradiated samples and reference standards were mounted on stainless steel supports and counted individually by gamma spectrometry. The counting time for gamma spectrometry was 3600 s.

Gamma spectrometry

Gamma spectrometry for all the analyzed filters was performed using EG&G Ortec hyper-pure Ge detectors, calibrated, with resolutions of 0.88 keV and 2.09 keV for the

122 keV and 1332 keV peaks of ^{57}Co and ^{60}Co, respectively. The counting time for each sample was 3600 s. Spectrum analysis was carried out using Gamma Analyst and an in-house software [13].

Scanning electron microscopy

Micrographs of the filters before and after the extraction procedure were obtained using Hitachi TM3000 tabletop scanning electron microscope (SEM) (Hitachi, NJ, USA) which offers a maximum magnification of 30,000X. The instrument is equipped with integrated energy-dispersive spectroscopy (EDS) and a load reduction mode, allowing the analysis of samples without the need for conductive coating. EDS measurements were taken using scanning electron microscopy equipped with a Jeol FEG electron gun, model JSM-IT700HR. The micrographs were obtained with an acceleration voltage of 5 kV and the EDS maps with 10 kV.

Results and discussion

The various manufacturing methods used for membrane filters result in differences in pore structure, morphology, and chemical resistance, particularly in relation to the acids used in dissolution testing [14]. In this study, during the chemical extraction stage, the filters exhibited differed degree of dissolution. The Type I filter dissolved completely, filters II, III and V showed partial dissolution, while filter IV did not dissolve. Examples are shown in Fig. 2.

Cellulose membrane filters (Type I) have been reported to dissolve completely in nitric acid solutions of varying concentrations [15, 16]. Polyethersulfone membranes (Type IV) are known for their excellent mechanical properties, as well as their exceptional hydrolytic, thermal and chemical stability [17]. Fiberglass and glass microfiber filters generally present good resistance to both weak and strong acids [18]. In contrast, the solubility of cellulose ester membranes in acid media depending on the specific type, ranging from fairly to complete dissolution [19].

In Fig. 3 the SEM images of the filter membranes used in the extraction tests are shown. Figure 3A–E correspond to filter Types I, II, III, IV and V, respectively, prior to acid extraction treatment. These images highlight the morphological differences among the microfibers structure and the voids between them, which reflec each filter porosity. The cellulose-based filters (Type I and V) exhibit a characteristic "spongier" morphology with interconnected pores located near the surface, as described by [20]. Fiberglass (type II) and glass microfiber (type III) display similar structure, composed of smooth, cylindrical fibers with no well-defined pore geometry. Filter type IV, shown in Fig. 3D, exhibits a typical microfiltration membrane architecture, featuring a porous surface and a network-like cross-section structure [21].

In Fig. 4 the SEM images of the filter membranes used after the extraction procedure are shown. In Fig. 4A, the image of filter Type III is presented, where only small fragments of the filter remained after exposure to acid. Figure 4B displays filter Type IV. Compared to the unused filters, filter Type III exhibit damages and distortion. In the case of filter Type IV, it appears that only the support layer remained unaffected following exposure to nitric acid at a concentration of 3 mol L^{-1}, while the active layer was thoroughly destroyed or significantly altered.

The EDS results obtained for filters Type I and V, composed of cellulose and cellulose ester, respectively, showed only carbon and oxygen. In filters Type II and III, the presence of sodium, aluminum and silicon were also detected due to fact that both are composed by fiberglass. Figure 5 illustrate the finding observed for filter II. In filter Type IV, composed of polyethersulfone membrane, the presence of sulfur was also identified (Fig. 6).

Although some cobalt has had remained after the extraction processes, especially in filter Type III, the filters did not show the presence of this element. This is justified by the

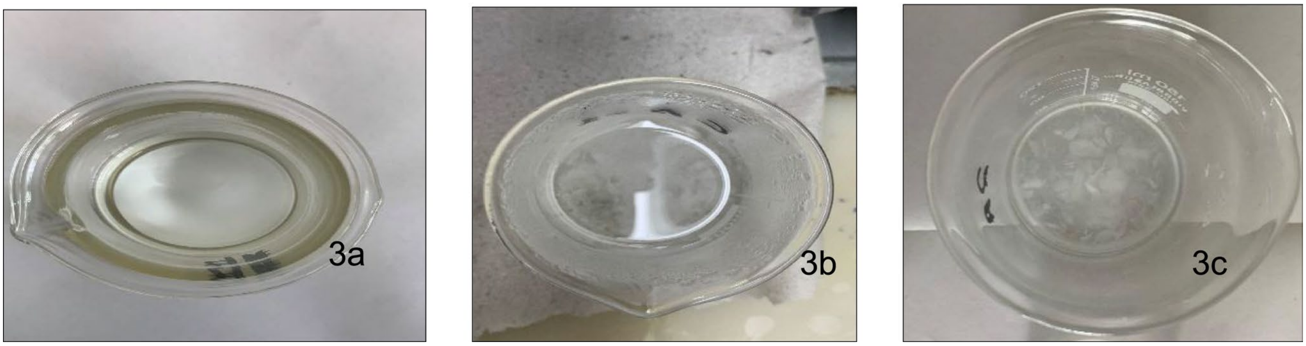
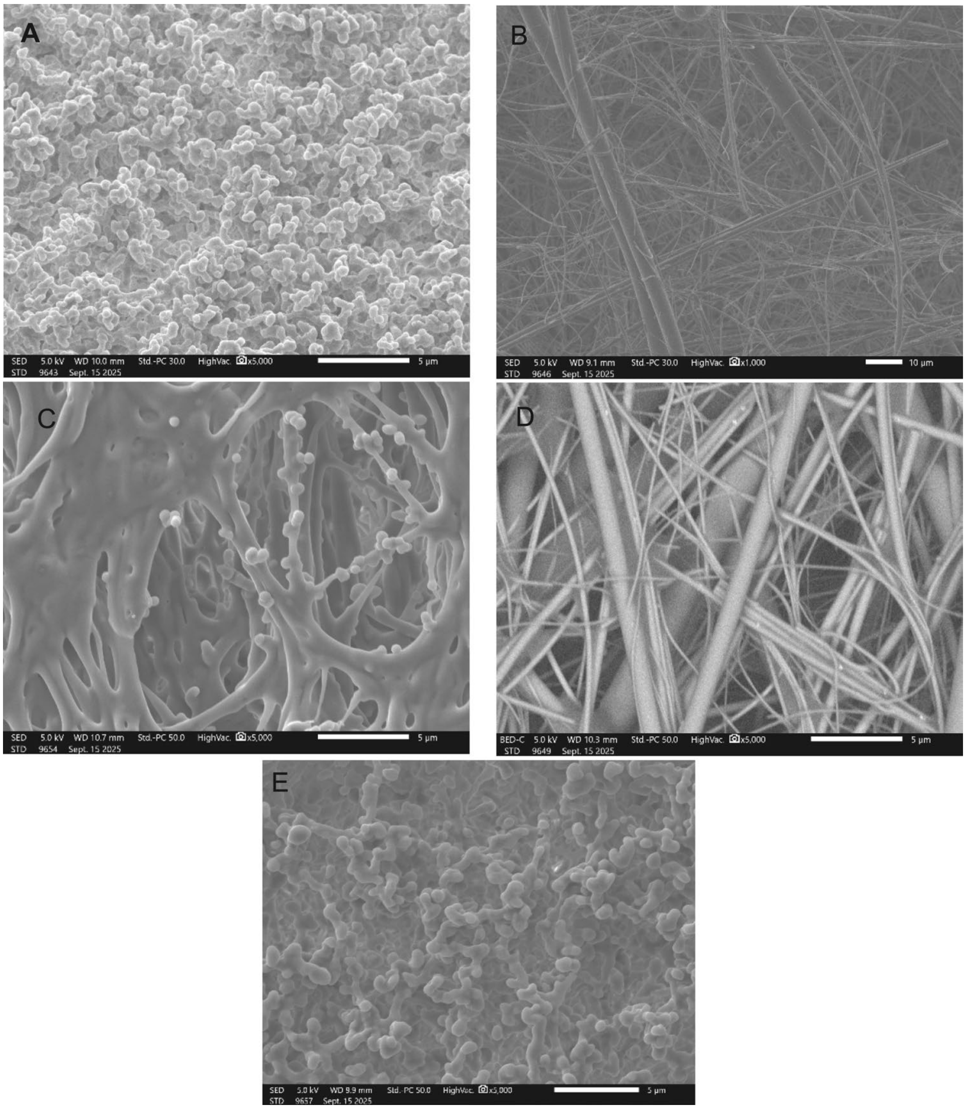


Fig. 2 Filters after the extraction procedure: **a** no dissolution (Type IV), **b** complete dissolution (Type I), and **c** partial dissolution (Type II, III, and V)

Fig. 3 SEM images of the used filters before the extraction process: **A** filter Type I; **B** filter Type II; **C** filter Type III; **D** filter Type IV and, **E** filter Type V



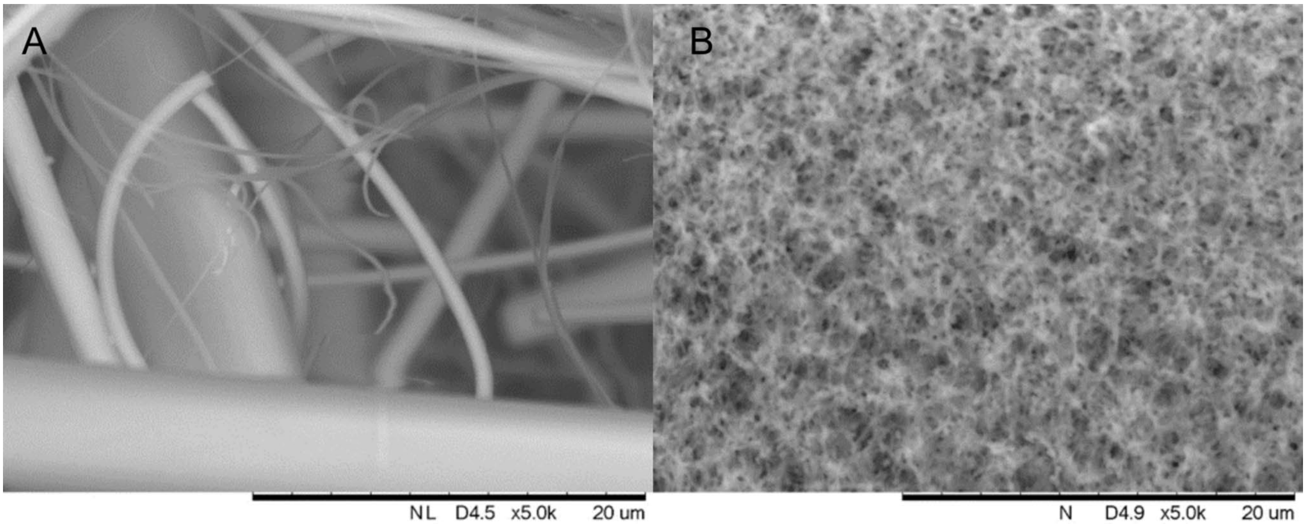


Fig. 4 SEM images of the used filters after treatment: **A** filter type III, that had partial dissolution and, **B** filter type IV, not dissolved

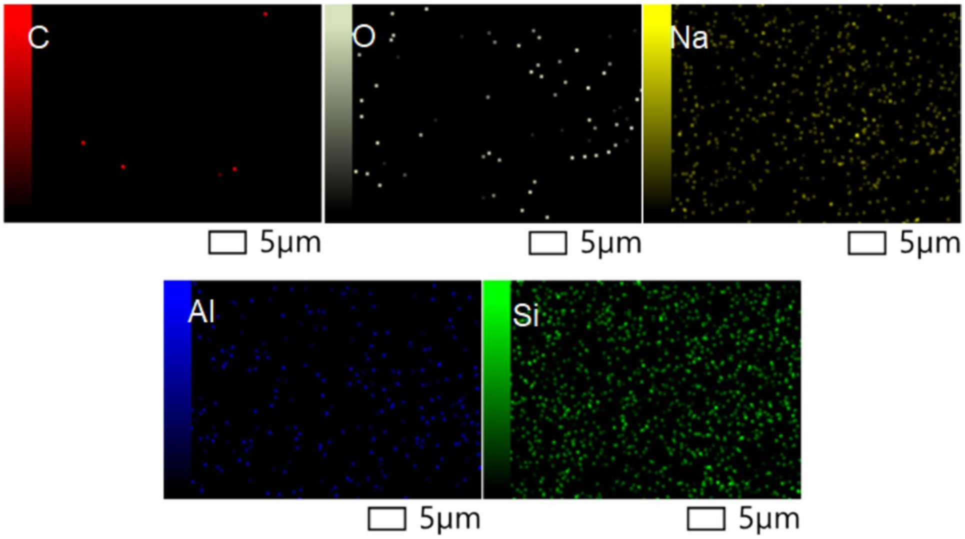


Fig. 5 EDS mappings of initial fiberglass filter membrane (Type II)

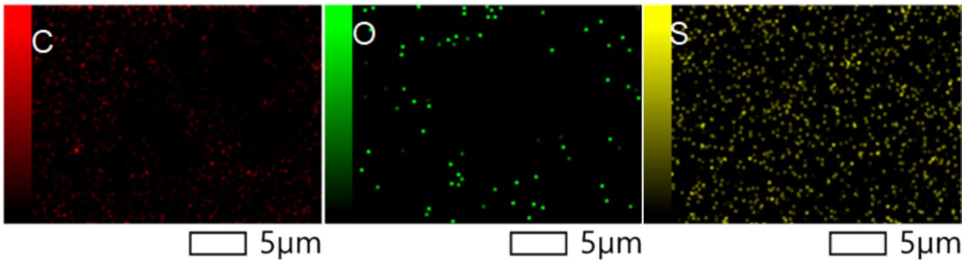


Fig. 6 EDS mappings of initial polyethersulfone membrane (Type IV)

Table 2 Chemical yield in the five filter types tested in hot acid extraction, in % of recovery (n = 3)

Filter	^{60}Co (%)	^{137}Cs (%)	^{238}U (%)	^{232}Th (%)
Type I	87.5 ± 8.6	85.5 ± 9.4	107.3 ± 6.5	97.4 ± 4.9
Type II	80.22 ± 0.04	87.6 ± 2.0	50.5 ± 7.7	50.7 ± 6.2
Type III	87.8 ± 6.3	85.5 ± 0.2	90.9 ± 10.5	82.2 ± 6.3
Type IV	103.9[1]	89.7 ± 7.8	95.1 ± 11.4	83.8 ± 12.0
Type V	87.6 ± 3.6	89.3 ± 0.6	113.3 ± 10.0	88.3 ± 12.7

[1] Only one sample was used, so there is no associated standard deviation

fact that the remaining Co amount must be in the level of picograms that cannot be detected by EDS technique.

There are few studies in the literature that specifically investigate the recovery yield of trace metallic elements and radionuclides from filters. Among the available studies which filters were used as the analytical bases, the most commonly employed methods include calcination, grinding, or complete dissolution of the filter material [22–24].

The results for the recovery yields obtained after the hot acid extraction procedure for each radionuclide are presented in Table 2. The chemical yields represent the arithmetic mean of three independent measurements.

The Type I filter, which dissolves completely, presents an extraction yield around 87% for ^{60}Co and ^{137}Cs, and around 100% for ^{238}U and ^{233}Th. Scofield [24] ported a chemical yield of about 93% for calcined cellulose ester filters, which is comparable to the values obtained in this study. The differences in yields is likely attributed to variations in the extraction techniques employed.

For the Type IV filter, which was not dissolved during the acid extraction procedure, recovery yields ranging from 83.8 to 103.9% was achieved. This filter type is the most recommended for analysis involving hot acid extraction when the objective is partial extraction rather than total dissolution, as it resist degradation under tested conditions. Most studies involving this filter type focus on organic compound analysis [25, 26]. However, Hedberg [27] reported potential issues related to contamination and adsorption when using this type of filter for trace elements analysis.

Filters Type III and V, which were only partially dissolved during the extraction process, showed recovery yields ranging from 82.2 to 107% for all the analyzed radionuclides. Values comparable to those reposted [22]. The lowest recovery yields were obtained for ^{238}U and ^{232}Th when using the filter Type II. A study in the literature reported a recovery yield of 65% for a Type II filter using a H_2SO_4 extraction method over a 6-h duration [28]. A similarly low yield was also observed in the present study, particularly for U and Th. One possible explanation is that the radioactive material adsorbed onto residual silica remaining after extraction process. This may explain why some studies recommend analyzing glass fiber filters only after complete dissolution using a combination of nitric and hydrofluoric acids [29].

The results obtained in this study indicate that acid extraction from different types of filter membranes can lead to analyte losses, with some cases, such as filter Type II for U and Th, showing losses of nearly 50%. Similar losses during filtration procedures and discrepancies in analyte concentrations measured before and after filtration have been reported in other studies [19, 30]. These findings underscore the importance of evaluating recovery efficiency when analyzing materials retained on filters, whether through dissolution or extraction processes.

As previously described, all final solutions were filtered using standard Whatman filter paper after the extraction process. To evaluate the possibility that part of the lost material was retained in this secondary filter during filtration and washing, the filters were analyzed by gamma spectrometry for ^{137}Cs and ^{60}Co only. This measurement was performed on the filters that contained visible residues after the extraction, i.e. filters III, IV, and V, each tested in triplicate. The results showed that no cobalt was detected; however, a mean value of 8 ± 2% of ^{137}Cs was observed, indicating that some material lost may occur at this stage of the process.

Conclusions

This work proposed a simple methodology for analyzing filters using hot acid extraction as an alternative to total dissolution or calcination. The results demonstrated that filters exhibit varying behaviors in response to acid exposure: ranging from complete to partial or no dissolution during the extraction process. Tests conducted with different filter types showed that, for all brands, the chemical recovery yields were higher than 80% for ^{60}Co and ^{137}Cs. However, lower recoveries were observed for U and Th when using fiberglass.

The extraction process for determining activity concentrations in filters containing radionuclides proved to be efficient. However, to ensure accuracy, it is essential to understand the dissolution or extraction characteristics of each filter type, as the proposed method cannot replace total dissolution techniques in all cases. The study also showed that, if not properly evaluated, the filtration process itself can lead to underestimation of radionuclide concentrations.

The proposed method demonstrated practical utility in scenarios where filters are used for contamination monitoring and high-precision results are not required, such as in surface swabbing. For application in the laser ablation apparatus, the Type IV filter proved to be the most suitable for hot acid extraction, as it does not fully dissolve and provided satisfactory recovery yields. The overall recovery may be improved by eliminating the secondary filtration step, an aspect that should be further investigated in future studies.

Research on the effectiveness of laser ablation for decontamination purposes is ongoing and will be presented in future publications.

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Declarations

Conflict of interest The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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