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Microwave Pyrolysis for Efficient Treatment of Radioactive Oil Sludge Waste: Experimental and Machine Learning Approaches

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Abstract Oil sludge waste (OSW) represents a substantial liability for the oil industry, given its composition of water, crude oil (heavier fractions), and sediments, as well as the presence of naturally occurring radioactive materials (NORM). This study presents a novel approach focused on the microwave-based drying of OSW containing NORM, a topic that has received little to no attention in the literature. While microwave pyrolysis has been explored for sludge treatment in general, previous studies have not addressed the specific challenges associated with the safe drying and volume reduction of radioactive OSW. Moreover, to the best of our knowledge, no prior work has applied machine learning algorithms to model and interpret the relationship between OSW composition and process performance during microwave drying of such complex waste. In this study, a dedicated system was developed to selectively remove water and oil from OSW while concentrating the radioactive content in the solid fraction, enabling safer and more compact disposal. The system was designed to take advantage of rapid and selective volumetric heating promoted by microwave irradiation, and to facilitate the capture of volatile compounds and radionuclides at the gas outlet. The process resulted in an average condensate recovery of 71% under microwave power of 993 W and a total treatment time of 15 min. The findings demonstrated that microwave treatment was an effective approach for treating OSW and obtaining dry sediment from samples with higher water and oil content.

Keywords NORM · Petroleum sludge · Radioactive waste · Treatment

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1 Introduction

Naturally Occurring Radioactive Materials (NORM) wastes are produced in a number of industrial contexts, including oil and gas extraction, mining, mineral processing, and certain manufacturing processes (de Araujo et al., 2020). In the oil industry, NORM wastes can manifest as scale deposits within pipelines or as a sludge comprising a mixture of sediment, oil, and water that accumulates at the bottom of separation equipment and storage tanks (Cowie et al., 2008). The radionuclides identified in these wastes are

members of the decay chains of naturally occurring radionuclides, namely ^{232}Th, ^{235}U, and ^{238}U (Casacuberta et al., 2011). The oil is primarily composed of organic compounds, including benzene, toluene, xylene, and heavy hydrocarbons (H. Chen et al., 2024).

The ultimate destination of the oil sludge is contingent upon its radiometric characterization. If the sludge activity exceeds the threshold established in CNEN regulation NN 8.01 (CNEN, 2014), it is classified as radioactive waste, Class 2.2 – radioactive waste from oil and gas production rigs. After screening on site, the waste is packaged and transported to the initial onshore storage facility, where it will await establishment of regulations for final disposal.

The generation of large quantities of radioactive oil sludge and the lack of a comprehensive set of regulations has been a significant concern for the oil exploration companies, for the regulatory bodies, and for researchers in Brazil (Godoy & Mourão, 2024). These are the facts: (i) the presence of long-lived, highly toxic radionuclides (Tessaro et al., 2021); (ii) high sulfur content of the Brazilian oil, which produces corrosive compounds inside waste drums (Xu et al., 2024), (iii) hydrocarbons and sulfur compounds that are responsible for the high chemical toxicity (H. Chen et al., 2024); (iv) lack of regulations ruling the treatment and the final disposal; (v) limited storage space; (vi) oxygenated compounds, such as phenols and carboxylic acids, which are often concentrated in the heavier fractions of crude oil, which can accelerate corrosion and promote gum formation through polymerization and oxidative processes (Czarnocka et al., 2015); (vii) complex and variable composition, rendering the sludge challenging to characterize and to treat through an efficient process; (viii) flammability (Teng et al., 2021).

In Brazil, the NORM waste from oil production rigs is stored in licensed storage facilities, the only currently available option (CNEN, 2014; Lopes et al., 2023). However, the current inventory has reached approximately 20,000 metal drums or polyethylene barrels with a capacity of 200 L each, and as an emergency solution, sludge was exported with authorization from the regulatory bodies of Brazil and the hosting disposal country. In view of the difficulties and risks associated with long term storage, it is of the utmost importance to reduce the volume of oil sludge under storage and to develop effective treatment methods with a view to reducing the volume and to meeting acceptance criteria for disposal of the treated waste. This will assist in reducing operational costs and the risks involved. It is crucial to acknowledge that the NORM issue represents a significant challenge not only for Brazil but also for other nations that adopt a comparable approach. However, it should be noted that the regulations pertaining to these wastes may vary between countries. Consequently, their management may be approached in different ways, depending on the specific legislation of each nation (Godoy & Mourão, 2024). In Brazil, disposal options like injection in plugged and abandoned wells, hydraulic fracturing and other methods practiced in many countries, are all forbidden by law if the concentrations of radioactive substances are above some limiting levels.

A variety of techniques have been described in the literature, many of which are designed to reduce the volume of the material in question by separating the water, degrading the hydrocarbons, and finally, leaching and extracting the radionuclides using different solutions. The use of microwaves is aimed at facilitating the removal of water and promoting the degradation of hydrocarbons (Cho et al., 2020). In microwave treatment, the application of microwave energy causes the molecules to align their dipoles with the electric field (Karami & Hossein Saeedi Dehaghani, 2022). As the oscillating field interacts with polar molecules, these molecules experience a constant rotational motion to align with the electric field creating friction between the molecules (Karman et al., 2022), resulting in localized overheating and rapid dissipation of heat to the surrounding sample (Yoshikawa, 2020). Water molecules are highly capable of converting microwave energy into heat due to their high dielectric constant ($\varepsilon = 76.6$). The organic compounds present in water/oil emulsions, such as asphaltenes ($\varepsilon = 5.5–18.4$), resin fractions ($\varepsilon = 3.8–5.1$), and petroleum ($\varepsilon = 2.1–2.6$), have a much lower polarity than water, so water molecules are mainly responsible for the dielectric heating in oil emulsions (Fortuny et al., 2008).

Microwave heating is a more energy-efficient process, exhibiting faster heating rates than conventional methods and greater temperature homogeneity. This is attributed to the fact that the heating occurs within the sample, rather than through heat transfer. The energy associated with the photons released by

microwave radiation is insufficient to cause the breaking of chemical bonds and the consequent modification of the structure of molecules (Sun et al., 2021). However, the rapid increase in temperature can result in the breaking of heavy hydrocarbon chains. The breaking of heavy hydrocarbon chains into lighter fractions reduces the boiling point of the oil fraction, thus facilitating its evaporation with water and favoring the separation of the solid fraction (Tajik et al., 2024).

Some studies in the literature have employed microwave technology with the primary objective of recovering oil or transforming it into fractions with enhanced added value (Abdulqader et al., 2022; Abualnaja et al., 2021; X. Chen et al., 2023; Y.-R. Chen, 2016; Francis Prashanth et al., 2021; Linhares et al., 2022; Liu et al., 2021; Wang et al., 2012; Xie et al., 2023). It is crucial to acknowledge that the characteristics of oil vary depending on the extraction well, suggesting that its treatments may yield different outcomes for different oil characteristics.

However, despite their proven efficacy in treating oil sludge, microwave-based technologies have not yet been applied to radioactive OSW in a comprehensive manner. Existing studies are either limited to hydrocarbon recovery or have focused on non-radioactive waste matrices as shown in Table S1. Moreover, there is a lack of process optimization and predictive understanding of how sludge composition influences treatment efficiency.

This study aims to fill this gap by investigating the application of microwave pyrolysis for the drying and volume reduction of NORM-contaminated oil sludge. A microwave system was designed specifically to treat this complex waste, including a gas-phase capture unit for volatiles and radionuclides. To understand and optimize the process, we applied machine learning models to link sludge composition (e.g., radionuclide activity, water and oil content) with treatment performance metrics (e.g., solid mass degradation). The research is guided by the following questions: (i) Can microwave drying effectively reduce the volume of NORM-contaminated sludge while safely concentrating radionuclides in the solid phase? (ii) Can predictive models help optimize treatment parameters based on sludge characteristics? Through experimental work, characterization, and data-driven analysis, this study proposes an integrated, efficient, and scalable solution to an urgent waste management challenge, with potential relevance to many countries facing similar regulatory and environmental constraints.

2 Materials and Methods

The primary objective of this research is to assess the efficacy of microwave technology as a means of treating oil sludge waste in an efficient, cost-effective, and environmentally safe manner. This approach aims to facilitate the separation of water and oil from the sediment. The experimental phase can be divided into the following five stages:1) characterization of the raw oil sludge; 2) adaptation of the microwave oven to remove and collect condensable vapors and placement of the absorber; 3) tests with water to adjust the suction flow rate of the vapors and gases; 4) tests with oil sludge samples; 5) characterization of dry sludge, liquid effluents, and activated carbon.

2.1 Characterization of the Sludge

A total of twenty-one samples of oil sludge was received from the oil extraction industry, which were divided into twelve batches, with an approximate weight of 3 kg per batch. Of the samples, nine were comprised of two samples per batch, while the remaining three batches consisted of a single sample each. The samples exhibited varying masses and were received in polyethylene bottles, as listed in Table S2.

2.2 Water Content

The water content was determined using the Dean Stark technique, which is based on the ASTM Standard D 4006-16e1 (ASTM, 2002). This technique relies on the principle of immiscibility between toluene and water solvents. This method was selected due to its cost-effectiveness and suitability for samples containing volatile compounds, with no restrictions on humidity range.

The sample was previously subjected to homogenization, and approximately 10 g were subsequently transferred to a round-bottomed flask with a capacity of 250 mL. A Dean Stark apparatus was affixed to the flask, and a condenser was attached to it, preceded by a connection to the refrigeration system. Subsequently, 60 mL of toluene was introduced via the

condenser. The flask containing the mixture was then heated with a heating mantle until the boiling point of toluene was reached, which occurred at 110.6 °C. The analysis was concluded when no further water was observed, which took approximately 90 min. The quantity of water was then determined on the Dean Stark volumetric scale.

2.3 Sediment

The sediment content was determined by gravimetry (Krug & Rocha, 2016). Approximately 2 g of the homogenized sample was added to a tared porcelain crucible, which had been previously dried in a muffle furnace at 600 °C and cooled in a desiccator to room temperature. The sample was carbonized using a Bunsen burner and incinerated in a muffle furnace at 850 °C for 6 h, cooled in a desiccator, and weighed. The working temperature of the muffle furnace was selected based on the multielemental and complex composition of the sample and the potential presence of long-chain hydrocarbons with a high boiling point (Ferreira & Aquino Neto, 2005). The oil content was determined by mass balance, employing the discrepancy between the total mass and the sum of the masses of water and sediment.

The height, diameter, and the characteristics of the material of the bottles were used to calibrate the gamma spectrometer employed in radiometric characterization. For detailed descriptions of the methodologies employed for radiometric characterization and for the assembled library with the energies and their highest abundances with the radionuclides of interest defined, please refer to Text S1 and Table S3, respectively.

2.3.1 Morphology Characterization

Scanning electron microscopy (SEM) was the device used to examine the micro- and nano-structural attributes of the solid components of the oil sludge waste, connected with an energy dispersive X-ray spectrometry (EDS), for the elemental analysis of the chemical elements distributed on the surface of the material. In this study, the SEM/EDS equipment (Hitachi, model TM4000Plus, Japan) was operated with an acceleration voltage of up to 20 kV.

Thermal gravimetric analysis (TGA) was used to measure the loss of mass due to the evaporation of volatile components, water and organic compounds, with the use of nitrogen as the carrier gas. The TGA equipment (TA Instruments, model SDTQ600, USA) was operated under the following conditions: Injection of the carrier gas, nitrogen, at a flow rate of 50 mL min^{-1}; furnace heating: 20 to 850 °C; heating rate: 20ºC min^{-1}; crucible material: 90 µL alumina crucible without lid; sample mass: approximately 10 mg.

The oil sludge wastes are characterized by a complex and variable composition. The raw samples were classified according to their visual appearance, specifically whether they appeared to have a greater concentration of oil, supernatant oil, or sediment. Table S4 presents images of these samples, which illustrate notable differences between them.

2.3.2 Description of the Microwave System

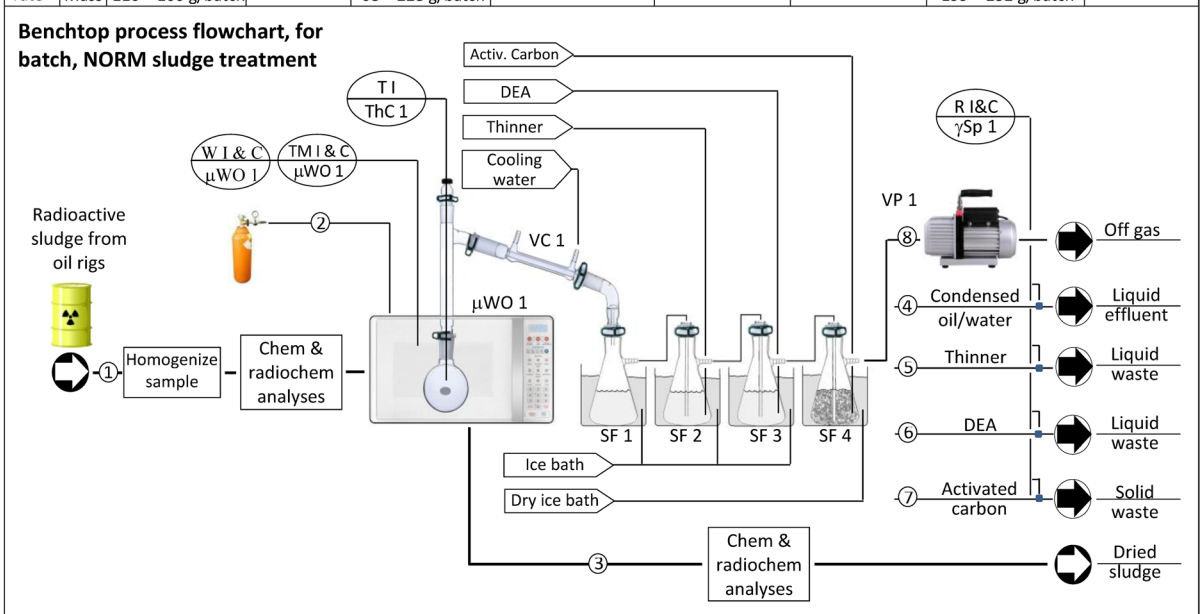
The oil sludge treatment process was carried out in a system consisting of a microwave oven, a glass flask with adapter and condenser, a condensate collection bottle, three gas scrubbers, a glass tube with water coupled to a condenser (microwave absorber) and a steam trap (for the photo of the system, refer to Supplementary Information, Fig. S1).

2.3.3 Microwave Oven

Figure 1 illustrates the process flowchart developed to treat these wastes by using microwave pyrolysis. The samples of raw oil sludge were subjected to a homogenization process, radiometric analysis, and chemical analysis. Thereafter, three batches (8155, 9825, 4579) were selected and samples were subjected to irradiation. In the case of batch 9825, samples from two different recipients (9825–5 and 9825–11) were collected. The condensate was collected in FC-01. In FL-01, thinner was used as a solvent to retain any volatiles. In FL-02, diethanolamine was utilized to retain thermally unstable sulfur compounds. Finally, in FL-03, which was maintained in a dry ice bath, activated carbon was used to retain any radionuclides. Nitrogen flowing was fed into the microwave flask to maintain an inert environment. Chemical analysis was conducted on both the raw and the dried wastes.

Radiometric analysis was conducted on the raw wastes, the dry wastes, the condensate, and the three gas scrubbers. For further information regarding the

Line		1	2	3	4	5	6	7	8
Material		Oil sludge	N_2 gas	Dry sludge	Condensed oil/water	Spent thinner	Diethanolamine	Spent activ. carbon	Off gas
Flow rate	Vol.	-	2 L/batch	-	2 – 101 mL/batch	200 mL/batch	200 mL/batch	-	20 L/min
	Mass	116 – 160 g/batch	-	68 – 228 g/batch	-	-	-	199 – 232 g/batch	-



ThC 1	μWO 1	VC	SF 1	SF 2	SF 3	SF 4	VP 1	γSp 1
Thermocouple 0 – 1100 °C	Microwave oven, 900 W; 30 L	Long glass Alihn condenser	500 mL condensate collection flask	500 mL thinner gas scrubber	500 mL DEA gas scrubber	500 mL cryogenic gas trap	1800 L·h^{-1} rotary vane vacuum pump	HPGe gamma-ray spectrometer

Fig. 1 Flowchart of the oil sludge treatment process using microwaves

methodologies employed for the system, please refer to Text S2, which contains detailed information about the temperature control, purging, condensate collection, materials used for trapping the compounds in the outlet of the system, and cleaning procedures after each experiment.

The experimental work began with some alterations to the household microwave oven (Spicy, model 409284000S, USA). The top was perforated to allow the passage of an adapter tube with three joints for monitoring the temperature, condensate outlet and oxygen purge, as well as a hole for connecting a test tube containing water coupled to a reflux condenser to ensure that it had a microwave absorber and the magnetron was preserved. The diameter of the holes was 3.5 cm for the adapter and 0.5 cm for the microwave absorber connection. Under these conditions, it was possible to monitor the temperature of the experiment.

All experiments were conducted at maximum power, with a 15-min duration. For the readings of the temperature, the processes were temporarily halted to prevent interference from microwave energy on the temperature-measuring equipment, with the temperature of the samples being measured at intervals of 5, 10 and 15 min. The system was operated within a gas exhaust hood. Prior to initiating any experimental trials, the microwave apparatus was calibrated. Calibration is a crucial step to ensure the reproducibility of the heating process and to validate the actual operational power of the device. The output power of the microwave oven was determined in accordance with the Brazilian INMETRO 174/2012 using the same water-load method as described in the IEC 60705, and calculated by using Eq. 1 (Rao et al., 2023).

$$P = \frac{4.187\, m_w\,(T_2 - T_1) + 0.55\, m_c\,(T_2 - T_0)}{t_{total} - t_{aq}} \quad (1)$$

where P is the output microwave power (W), m_w is the mass of water (g), m_c is the mass of the container used (g), T_2 is the final water temperature (ºC), T_1 is the initial water temperature (ºC); T_0 is the ambient

temperature (ºC), t_{total} is the total time (s), t_{taq} is the magnetron heating time.

To this end, 2 L of water in a PET bottle was cooled to a temperature of approximately 10°C. The mass of the empty, dry beaker (316 g) and the beaker filled with drinking water (water: 1 kg) was determined using a precision scale. Subsequently, the temperature of the water was determined using a mercury thermometer, with a final reading of 21°C. The ambient temperature was 26°C. The microwave was set to maximum power, the beaker containing the water was added and heated for one minute. The temperature of the water in the beaker was then measured. The procedure was carried out in duplicate. The average power value of the microwave was calculated to be 993 ± 1 W.

2.3.4 *Microwave Penetration Capacity and Sample Sizing*

It was crucial to ensure that the entire sample was irradiated by selecting an appropriate sample size. This is because microwaves interact with materials in different ways, and their ability to penetrate depends on the frequency and dielectric constant of the material chosen as an absorber. In addition, it is not possible for household microwaves to radiate uniformly. The penetration depth of the microwaves was calculated using Eq. 2 (Tripathi et al., 2015).

$$D_P = \frac{\lambda_0}{2\pi(2\varepsilon')^{1/2}}\left[\left\{1+\left(\frac{\varepsilon''}{\varepsilon'}\right)^2\right\}^{1/2}-1\right]^{-1/2} \quad (2)$$

where D_P is the penetration depth, λ is the wavelength, which can be calculated as $\lambda = c/f$, where c is the speed of light (3×10^{10} cm s^{-1}), f is the frequency (Hz), and ε_r is the dielectric constant.

A penetration depth of 2.217 cm was obtained in the sample using water as a microwave absorber. A 500 mL flat-bottomed flask was used as a reference to size the sample to be treated. The average height found for the spherical part of the flask was 10 cm.

2.3.5 *Accuracy in Radiometric Analysis*

The precision of the radiometric analysis was determined by employing two reference materials that have been certified by the International Atomic Energy Agency (IAEA) (refer to Supplementary Information, Table S5, for the analysis results). The initial reference material was IAEA-RGU-1, Uranium Ore, which contains the following radionuclide: ^{238}U, which is not a gamma emitter. However, the analysis was conducted with consideration of the radioactive balance through the material's descendants, specifically ^{214}Bi with an emission at 609.312 keV and ^{214}Pb with an emission at 351.921 keV. The second reference material was IAEA-RGTh-1, Thorium Ore, which contains the following radionuclide, ^{232}Th, which is also not a gamma emitter. However, it was analyzed considering the radioactive balance through its descendants: ^{212}Pb with emission at 238.632 keV and ^{228}Ac with emission at 911.20 keV.

The reference material was conditioned and its geometry was calibrated efficiently using the In-Situ Object Counting System (ISOCS) calibration software. This software combines the detector characterization produced by the mathematical modelling code called Monte Carlo N-Particle Transport (MCNP), mathematical geometry models, and some parameters of the sample analyzed (Kalb et al., 2000). Equation 3 was employed for the calculation of radionuclides' activity from the reference materials (Thakur et al., 2024).

$$A_\gamma = \frac{N_\gamma}{\varepsilon_{detector} I_\gamma t} \quad (3)$$

where $A\gamma$ represents the activity of the source (Bq), $N\gamma$ denotes the net photopeak area (background-subtracted), $I\gamma$ signifies the gamma-ray emission probability, $\varepsilon_{detector}$ is the detector efficiency, t denotes the time elapsed, considering any potential counting losses associated with the analyzer.

2.3.6 *Machine Learning Approach*

To address the disparate characteristics of the oil sludge samples (radioactive content, composition), as well as the various operating parameters (temperature, time), and responses (removals and condensate generation), the Principal Component Analysis (PCA) was utilized. In essence, PCA is a technique that transforms an initial set of variables into a smaller set by employing linear combinations that represent the majority of the observed variations in the original data set. This process allows for the extraction of

principal components, with the first and second components, PC1 and PC2, accounting for the two largest variations in the data.

To classify and analyze the disparate characteristics of the oil sludge samples, K-Means clustering was employed in conjunction with PCA. K-Means is a well-established unsupervised learning algorithm that groups data based on feature similarity, partitioning observations into clusters in which each observation is assigned to the cluster with the nearest mean. Multiple regression modelling was employed and its prediction power evaluated. Figure 2 shows the ML flowchart employed in this work. For more details on the implementation, refer to Text S3. A generalized linear model (GLM) was chosen to provide interpretable linear relationships between principal components and degradation. In contrast, Random Forest enabled the capture of potential nonlinear dependencies and provided ranked feature importance, offering complementary insight into the process.

3 Results and Discussion

The initial stage of the study entailed the selection of the most representative samples. This was accomplished by measuring the concentrations of ^{228}Th, ^{226}Ra, and ^{228}Ra, as well as the chemical composition of the water, solids, and oil, and by observing and characterizing the visual conditions of each sample in terms of dry, oily, greasy, sandy, and oily compacted. The results are presented in detail in Tables S4, S7, and S8. Figure S2 depicts the correlation matrix for the characteristics of the radioactive oil sludge waste and the dependent variables of the experimental tests. The figure illustrates a number of significant positive correlations, including the relationship between temperature after 15 min and the condition of the waste (e.g., whether it is oily or sandy). Similarly, the removal of water and oil is highly correlated with the percentage of water.

Given the significant correlation values identified, PCA and clustering were employed in an alternating sequence to facilitate the interpretation of the selection process for the desired samples. The data presented in Table S9 is a subset of the variables employed in this study, combining the data shown

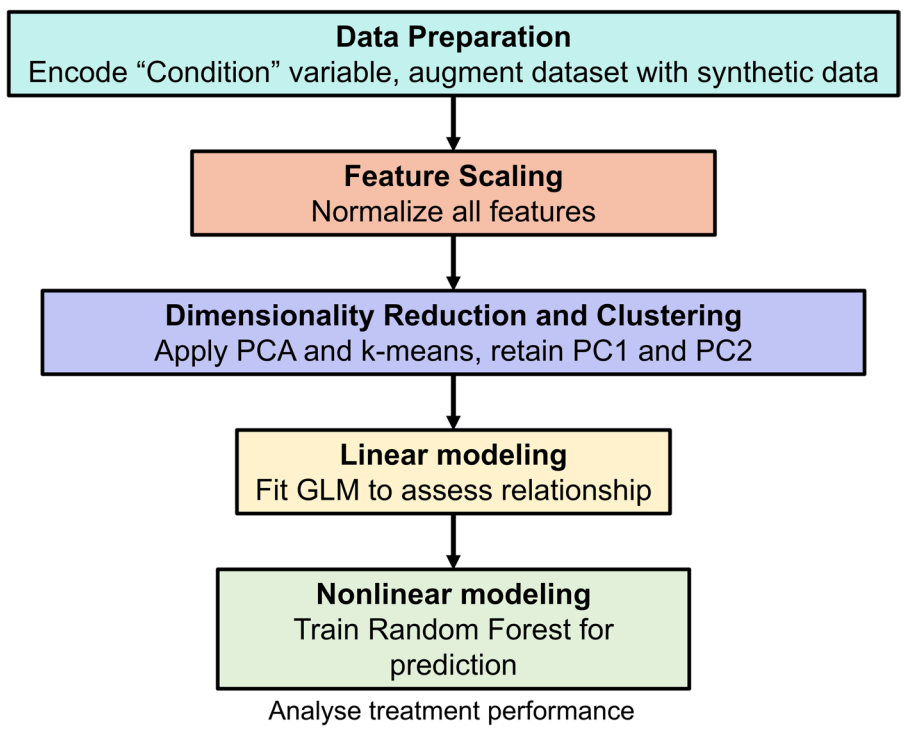


Fig. 2 Flowchart used for the application of the mathematical models. PCA: principal component (PC) analysis; GLM: generalized linear model

in Tables S4, S7, and S8. As illustrated in Fig. S3, the samples can be classified into three distinct clusters. The blue cluster (Cluster = 0) is comprised of samples that are dry and sandy. The orange cluster (Cluster = 1) contains oily samples, and the green cluster (Cluster = 2) includes greasy samples. The application of PC1 and PC2 permitted the representation of six variables in a two-dimensional image, with approximately 61% of the total representation, explaining most of the variance, as shown in Fig. S4. Furthermore, the employment of visual characteristics for clustering enabled an appropriate differentiation of the samples.

The samples selected for further treatment were then chosen based on three criteria: activity concentrations (^{228}Th, ^{226}Ra, ^{228}Ra), composition (% water, solid, oil), and visual characteristics (dry, oily, grease, sandy, oily-compacted), totaling eleven variables. In accordance with these criteria, one representative sample was selected from the dry samples, two from the oily samples, and one from the sandy samples. The selection of the oily samples was based on the use of samples from the same batch for the purpose of checking for homogeneity. This selection permitted the evaluation of disparate samples (clusters 0 and 1, Fig. S3), as well as a comparison of samples that were in close proximity to each other within the same cluster. Furthermore, the application of the ordinary least squares model from the principal components permitted a satisfactory fitting ($R^2 = 0.997$) between observed and predicted values, as illustrated in Fig. S5 and detailed in Table S10. The following equation was generated: $Y_{pred} = 67 + 9.73X_1 + 9.37X_2$, where X_1 and X_2 represent PC1 and PC2, respectively.

3.1 Inter-laboratory Comparison

Samples from four drums were collected and analyzed before and after microwave pyrolysis. Table S6 shows the measurements for ^{212}Pb, ^{214}Pb, ^{214}Bi, and ^{228}Ac for these samples. To ensure the precision and reliability of the results obtained in the radiometric analysis, an inter-laboratory comparison was conducted. The laboratory responsible for conducting the tests was the Radiation Metrology Department (GMA, in Portuguese) from the Nuclear and Energy Research Institute (IPEN, in Portuguese). All gamma spectrometry analyses conducted at the GMA were performed using standard vials with fixed measurements and capacity, with a weight of 100 g per sample. Four samples were selected for comparison: two were obtained directly from the source material, while the other two were prepared by drying the wastes. Upon receipt of the standard bottles, the samples were separated by the laboratory where this research was mainly conducted, the IPEN's Radioactive Waste Management Service (SEGRR, in Portuguese).

The sampling procedure entailed transferring all the samples into a beaker, homogenizing them, and transferring them into a standard bottle. The vial was then closed, labeled with the relevant batch, weight, and date, and sent to the GMA, where it was sealed and stored for 30 days prior to analysis. This was done to ensure accurate measurement of the activity concentration and to maintain equilibrium between the radionuclides of interest. Following the completion of the analysis, the sealed samples were transferred to the SEGRR for further examination and comparison. Figure 3a illustrates the results obtained from the GMA and SEGRR for the same sample, with identical geometry and weight, as well as the SEGRR result for the same sample with a different geometry and weight.

3.1.1 Radiometric Characterization of oil Sludge Wastes from Raw Samples

Radiometric analysis was conducted on all samples due to the lack of knowledge regarding their chemical composition, the absence of established criteria for sample selection, and the necessity to ascertain equilibrium of radionuclides prior to sample opening. As shown in Fig. 3b, there is a significant variation in activity among the batches, which can be attributed to either differences in the characteristics of the geological formations or differences in the extraction methods employed.

3.1.2 Chemical Characterization

Figure 3c depicts the results of the chemical analysis. The percentages of water, solids, and oil were found to range from 1 to 38%, from 29 to 91%, and from 7 to 41%, respectively, demonstrating notable discrepancies among the examined batches. Indeed, the chemical composition of oil sludge waste displays considerable variation across different studies,

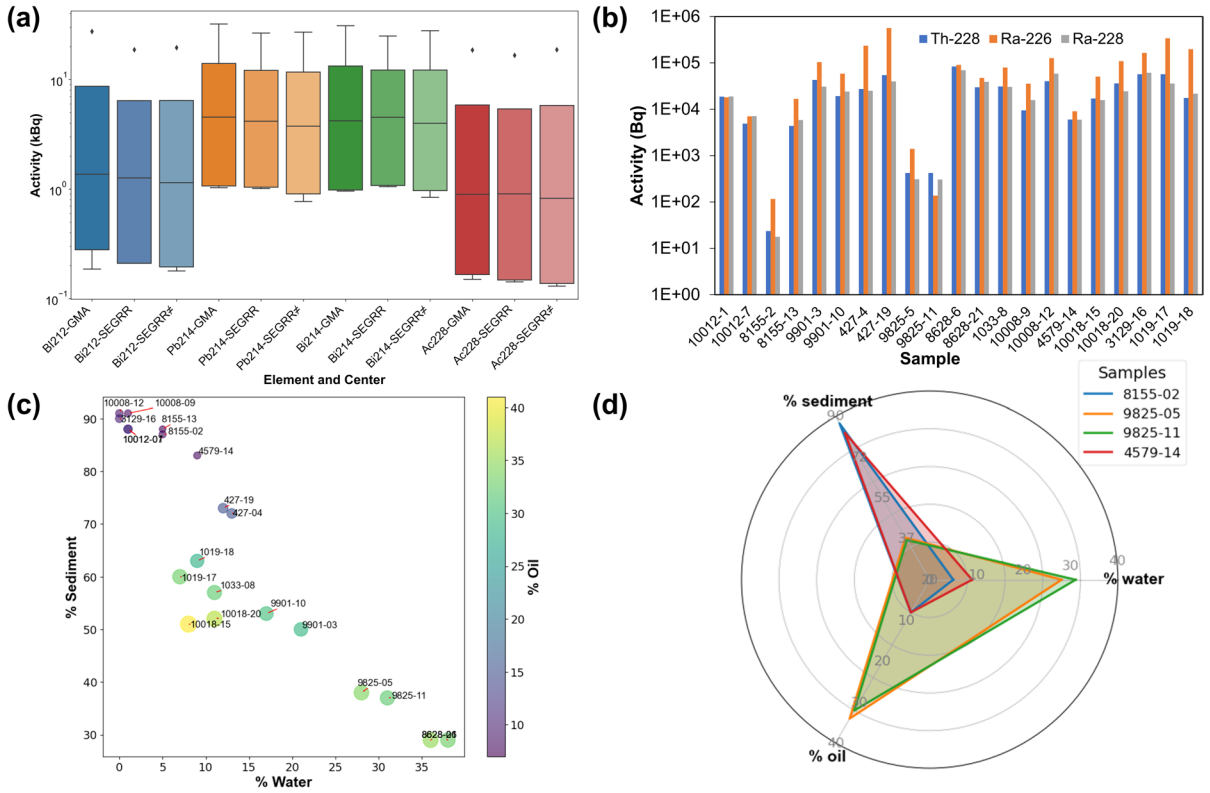


Fig. 3 (**a**) Boxplot of the radiometric analysis for interlaboratory comparison between GMA, SEGRR and SEGRR with same geometry but different weight (≠) in logarithmic scale (for the actual values, refer to Table S6); (**b**) Results of the radiometric characterization of the raw oil sludge wastes in logarithmic scale (Table S7); (**c**) Chemical characterization of raw oil sludge (Table S8); (**d**) Chemical characteristics of the samples chosen for treatment using microwaves

reflecting the inherent variability of such materials in their natural state. In the case of water, the value is considered to be 22% (Oliveira, 2002), 30–85% (Hu et al., 2013), and 17% (Guimarães et al., 2016). With regard to solid, the same authors posit a figure of 62%, 5–46%, and 17%, respectively. With regard to oil, the figures are 17%, 5–86%, and 43%. Additionally, sulfur was previously identified in these waste materials at approximately 1% (Oliveira, 2002).

3.1.3 Treatment of Samples Using Microwaves

The microwave system was tested with four samples, selected based on their high water and oil content or high sediment content, as shown in Fig. 3d. The initial sample subjected to analysis was 8155–2, which exhibited the highest percentage of sediment. The photos of the selected samples are presented in Fig. 4a. As displayed in Fig. 4b, it was demonstrated that the microwave treatment was not an effective method for sludges with low water and oil contents. For sample 8155–2, this treatment resulted in the generation of only 2.5 g of condensate, which corresponds to 9% of the sample's water and oil content (Table S11). We observed that the experiment could have been finished at the four-minute mark, at which point no further condensate was collected.

The second sample to be subjected to the specified treatment was 9825–5. Samples with a higher water and oil content present a greater challenge to the management of radioactive waste, as they also exhibit chemical toxicity, corrosive compounds, and flammability, which can compromise the integrity of the packaging by exposing the wastes. The results for this batch demonstrated a notable variation in temperature, as shown in Fig. 4c, indicating a high degree of heterogeneity, although efforts were made to achieve homogeneity. Despite the observed temperature

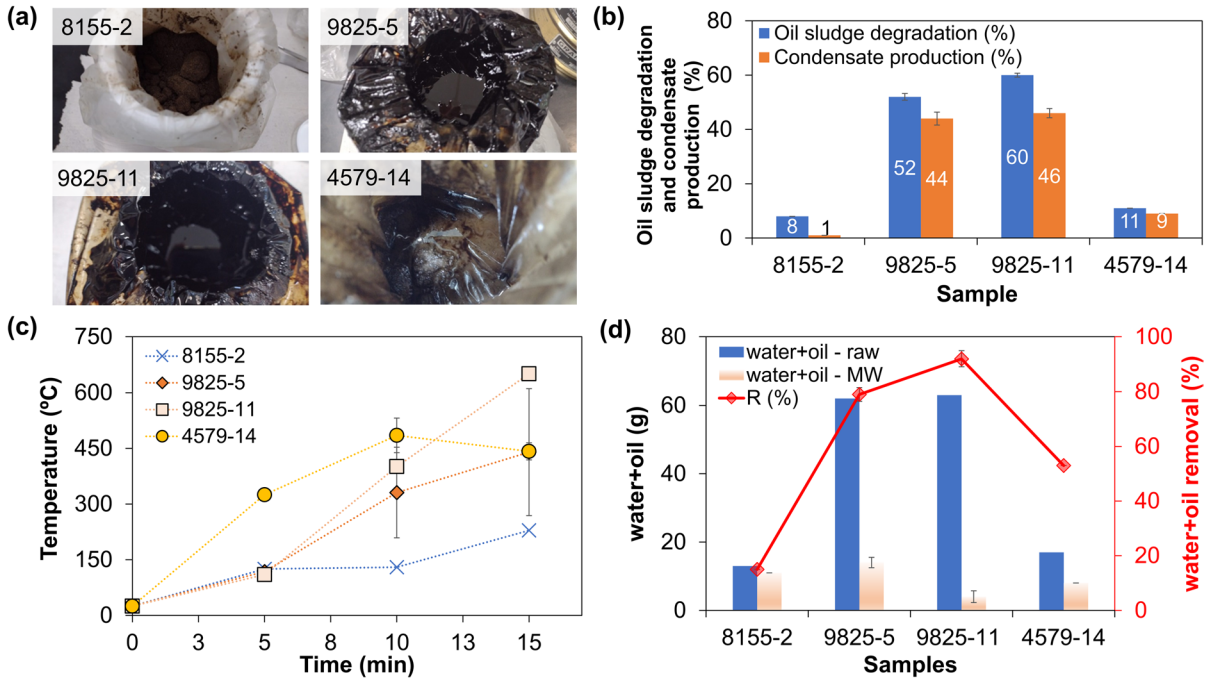


Fig. 4 **a**) Photos of the selected samples; **b**) results of water + oil removal after treatment using microwaves; **c**) temperatures obtained when monitoring the treatment using microwaves; **d**) removal of water + oil by microwaves

variation, the average percentage of water and oil removed reached approximately 80% (Fig. 4d).

In the case of sample 9825–11, the temperatures of the experiments exhibited greater uniformity (Fig. 4c). This phenomenon is likely attributable to the apparent enhanced homogeneity of this particular batch. After nine minutes, the continuation of the treatment without condensate collection resulted in a notable increase in the temperature of the experiment, leading to the generation of brownish vapors that did not condense. This resulted in chemical contamination of the entire system due to the vapors. The quantity of water and oil removed as condensate was the greatest observed in this study, averaging 92% as illustrated in Fig. 4d.

The final sample treated was 4579–14, which exhibited a considerable quantity of sediment, as well as notable differences in characteristics when compared to the other samples. These differences included a higher density, larger granules, and a more transparent liquid phase. The removal rate was significantly lower than that observed in the 9825 batch, with a yield of 53%. The tests could have been terminated at the 9-min mark for 9825–5 and 9825–11, and at the 11-min mark for 4579–14, when no further condensate was collected.

During the microwave treatment, the experiment's temperature exhibited fractional distillation behavior, with well-defined plateaus. This behavior was particularly evident during the first five minutes of testing, where the temperature remained, on average, at 118 °C, likely due to the predominant microwave absorber in the waste being water. Despite the fractional distillation behavior, thermal cracking may have occurred due to the high temperatures reached during the experiment. This is supported by the observation of brownish vapors, which are typically associated with the vaporization of heavier compounds and the condensation of some intermediate products during thermal cracking. Thermal cracking at elevated temperatures promotes the breaking of chemical bonds in smaller molecules, leading to the formation of smaller hydrocarbons, aromatic compounds, and other volatile by-products, which likely contributed to the color of the observed vapors.

A quantitative experimental study on the effect of each separate parameter in this system is not possible given the interaction effects between

the operational variables even in controlled systems. However, the correlation matrix presented in Fig. S2 can give important insights about the results we obtained. For instance, condensate generation (%) is highly correlated with %water, %oil, temperature after 15 min, removal of water and oil, in addition to the waste presenting a visually oily condition, with correlation values above 0.78. As also previously discussed, feature importance studies are useful tools to ranking variables regarding target responses, such as solid mass degradation (%) (Fig. S5).

These machine learning results can serve as a valuable foundation for future process optimization and classification of new oil sludge samples. For instance, the strong correlations and feature importance rankings suggest that pre-characterization of oil content, water content, and visual condition of the waste could help predict key outcomes such as condensate recovery or degradation efficiency, without the need for extensive laboratory tests. Moreover, clustering analysis provides a basis for grouping similar waste types, even under multiple and variable conditions, which often make it difficult to select representative samples. This approach can help tailor treatment strategies accordingly and streamline operational decisions in industrial applications.

3.1.4 Morphology

The objective of the SEM analysis was twofold. Primarily, it aimed to elucidate the intricate and heterogeneous composition of the oil sludge wastes, elucidating the elemental composition and particle size distribution of the sediment fraction. Secondly, it sought to ascertain the feasibility of employing the method as a supplementary technique for identifying the radionuclides of interest in the study. The particle size distribution of the samples significantly varies, as observed in Fig. 5 and Figs. S6-S8. Nevertheless, EDS spectrum was found to be similar for all the analyzed samples, indicating that although particles are somehow different in terms of distribution and characteristics of the solid particles such as size, elemental composition is similar. As observed in Fig. S8, the distribution map for Ra and U for sample 4579–14 is practically identical, given that Ra is a decay product of U. This is the sample that exhibited the highest level of activity during radiometric analysis. However, it was also expected that the

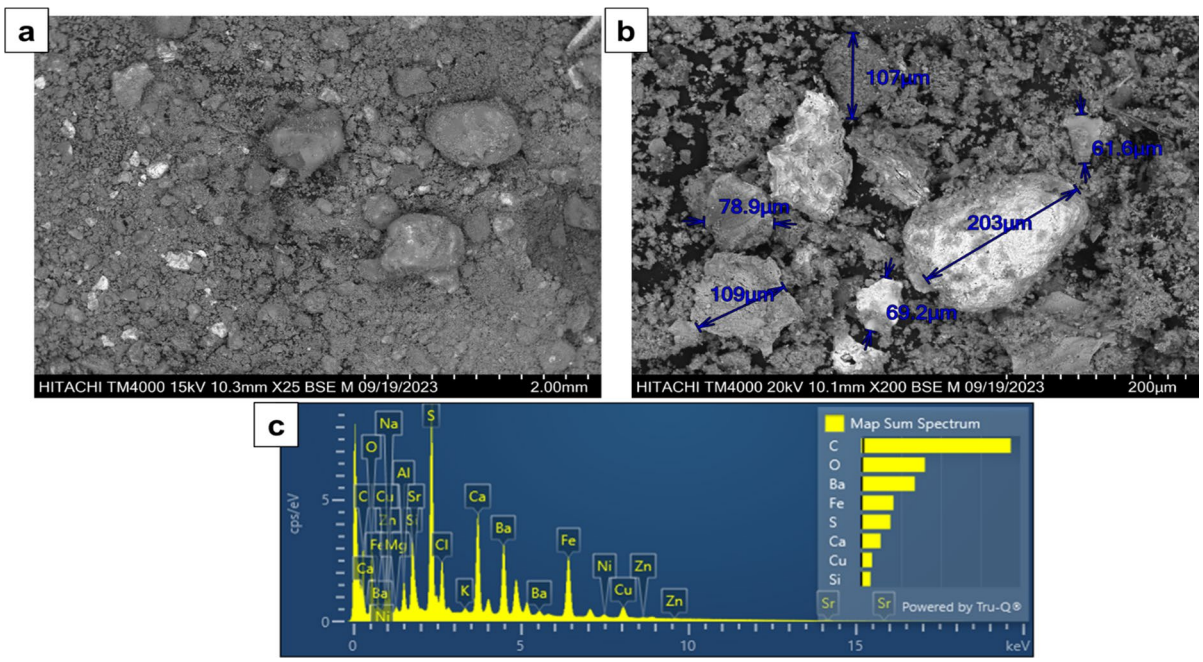


Fig. 5 Morphology of the treated oil sludge from batch 9825–11. SEM image (**a**) 25 × magnification (**b**) 200 × magnification; (**c**) distribution map of the chemical elements Ra and U; (**d**) EDS spectrum showing the elemental composition

isotopes of U, specifically ^{238}U and ^{234}U, would be present in smaller quantities in the oil sludge, remaining in the reservoir rock where extraction takes place. With regard to the Ra isotopes, specifically ^{226}Ra and ^{228}Ra, their presence was anticipated due to leaching and transportation along with the aqueous fraction.

3.1.5 Thermogravimetric Analysis (TGA)

The importance of the TGA analysis is to better understand the composition of the raw sludge and to evaluate the efficiency of the microwave treatment by comparing weight loss between temperature ranges. Since the sample used for the analysis has a very small mass and the wastes are difficult to homogenize, it is expected that the results will be approximate and contain errors. Figure 6a shows the boiling point ranges and compositions of petroleum fractions that can be used to determine the approximate composition of the raw samples and after microwave treatment, as well as the boiling point by carbon range.

The TGA analysis was conducted on both the raw samples and the dry mass resulting from microwave treatment. The variation in mass loss as a function of temperature was observed. The resulting spectra from the TGA analysis are presented in Figs. S10-S12. Figure 6b illustrates the total weight loss percentage for eight samples under TGA conditions. The raw samples 8155–2 and 4579–14, along with their microwave-treated counterparts, exhibited comparable thermal profiles, as evidenced by their mass losses during the temperature increase in the TGA analysis. This similarity in profiles suggests that the treatment is ineffective for samples with low water and oil content.

It is evident that the raw 9825–11 sample undergoes the greatest degree of decomposition when subjected to an increase in temperature, with an approximate loss of 70% (Fig. 6b). With the exception of the initial 9825–11 microwave-treated sample, which also exhibited a notable total weight loss (exceeding 30%), the remaining microwave-treated 9825–11 samples demonstrate a considerable loss when compared to

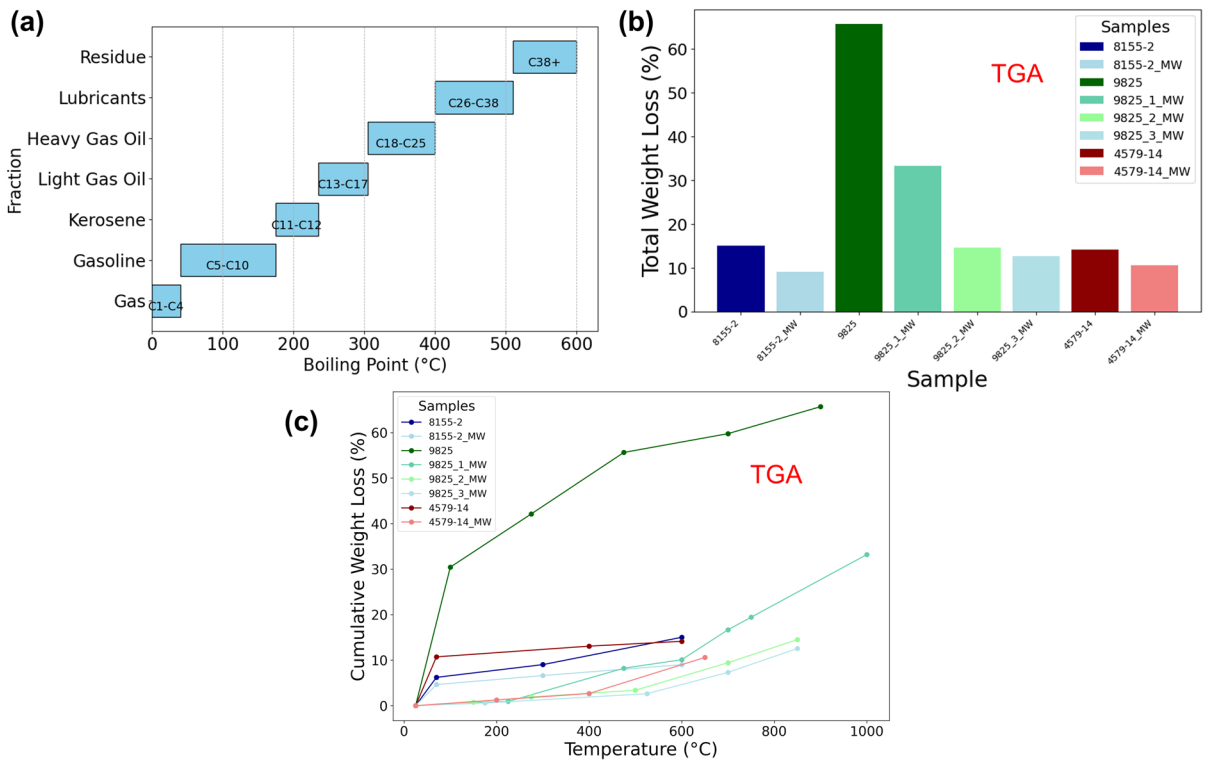


Fig. 6 **a**) Boiling point ranges and compositions of petroleum fractions.; **b**) total weight loss by sample considering the whole temperature range; **c**) cumulative weight loss by temperature range

8155–2 and 4579–14. This suggests that the microwave effectively facilitated the drying of the raw wastes. Indeed, sample 9825–11 exhibited a considerable loss in weight even prior to reaching 100 °C, at approximately 30%. In contrast, the microwave-treated 9825–11 (tests 2 and 3) began to display a significant loss in weight at temperatures exceeding 600°C. A comparison of the thermal profiles of the raw and microwave-treated 9825–11 samples reveals significant differences. The results indicate that heat treatment is an effective method for radioactive oil sludge samples with high water and oil content.

3.1.6 Radiometric Analysis of Different Components of the Reaction System

The activities of the raw oil sludge samples and those subjected to microwave treatment were compared, as illustrated in Fig. 7. Six solutions were analyzed: the raw oil sludge was designated "Raw", the treated solution was designated "Dry", the solutions from the condensate and from the gas scrubbers were designated as such, as were the thinner and DEA solutions. Additionally, the activated carbon was designated "AC". The activities from the condensate, thinner, and DEA were considerably lower (Table S12), and thus are not visible in this graph.

Figure 7 illustrates the retention levels post-treatment, highlighting the variations in component activities (raw, dry, condensate, thinner, DEA, AC). It was determined that the activity of the raw sample is nearly equivalent to the sum of the activities observed in the irradiation products, particularly in the case of the dry waste, indicating that the radionuclides are predominantly retained and concentrated in the dry waste. A reasonable mass balance of the radionuclides was established, although with some approximation, due to the inherent losses associated with the oil waste system. A portion of the oil sludge becomes

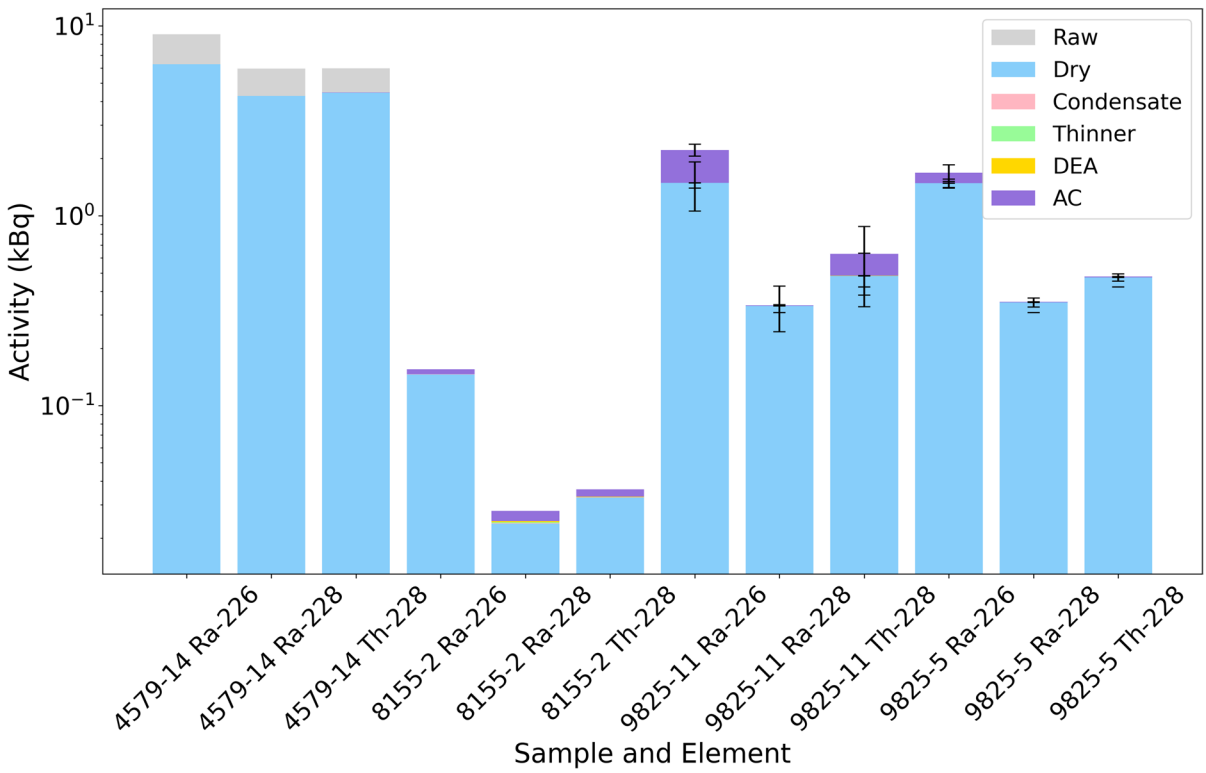


Fig. 7 Distribution of initial and post-treatment activities for each sample and element. The log scale emphasizes variations in component activities (Raw, Dry, Condensate, Thinner, DEA, AC), indicating retention levels post-treatment. The non-visible components imply that the activities were much lower when compared to others. For the detailed calculations on these samples' uncertainty quantification and detection limits, refer to the file sheet (Online Resource 1)

attached to the reactor system, necessitating the use of potent surfactants or acids for effective removal.

Notwithstanding, it was observed that the radionuclides were drawn into the condensate and gas scrubbers, despite the fact that their melting and boiling points are above the maximum temperature of the experiment. As anticipated, the activated carbon in ice bath was capable of retaining a considerable quantity of the radionuclides at the outlet of the system. Notwithstanding the detection, the values for the condensate and gas scrubbers remained below the exemption limit established in national regulations (CNEN, 2014), thus allowing for their disposal as non-NORM waste. The system has demonstrated satisfactory capacity in terms of treatment and capture of the outlet elements. However, improvements are still necessary, including enhanced cleaning procedures and the development of dedicated systems for this specific waste type. These systems should exhibit high efficiency, a higher power output, and more effective microwave harvesting, which could be achieved through optimized reactor geometries, improved microwave distributions, or the integration of alternative reactor types, such as fluidized ones.

4 Outlook of Microwave Pyrolysis For Oil Sludge Treatment

Despite promising laboratory-scale results, microwave pyrolysis faces key challenges for broader application. Although reactor design allows for scale-up by adjusting residence time and temperature (Alam & Khan, 2024), the technology remains at a low technology readiness level, with limited industrial deployment (Motasemi & Afzal, 2013). Energy balances are often neglected (Mutsengerere et al., 2019), but techno-economic studies at the 100 kg/day scale indicate breakeven within four years and lower greenhouse gas emissions than landfilling (Neha et al., 2022).

Safety concerns include the release of volatile organic compounds, including polycyclic aromatic hydrocarbons and trace metals, that may pose potential health and environmental risks if not properly contained and treated (Li et al., 2022; Roslee et al., 2023; Tian et al., 2011). Also, in the case of radioactive wastes, there is a potential volatilization of radionuclides (Prado et al., 2021), demanding robust gas treatment and radiation control systems to prevent airborne contamination.

In Brazil, specific regulations for the disposal of Class 2.2 radioactive waste from NORM sources are still lacking in the CNEN framework. There is a need for clear licensing requirements, safety analysis scenarios, and waste classification protocols, potentially modeled on standards like CNEN-NN-4.01. Law 10.308/2001 allows for delegation of waste management to generators or third parties, but technical guidance remains limited (Godoy & Mourão 2024). Similarly, in the United States, NORM waste is not regulated under the Atomic Energy Act or the Resource Conservation and Recovery Act, and oversight is largely left to individual states (TCEQ, 2025; US EPA, 2021). Disposal typically requires specific licenses, and untreated radioactive materials are banned from conventional landfills.

5 Conclusion

The microwave treatment was found to be an effective method for treating oil sludge waste and obtaining dry sediment from samples with the highest water and oil content. The experiment is entirely dependent on a microwave absorber, and for the purposes of this research, water was considered, thus the estimated temperature of the experiment was 300°C. However, it should be noted that there are other microwave absorbers present in the oil sludge wastes, as evidenced by the temperatures recorded during the course of the experiment, which reached a maximum of 674ºC. The rationale behind characterizing the raw samples was to define a range of water and oil content that was deemed relevant for the research project. The treatment proved ineffective for the sample with a water and oil content of 12%, yielding only 10% recovery in the form of condensate. Conversely, for the sample with a water and oil content of 17%, a recovery of 50% in the form of condensate was observed. The mean recovery for samples with water and oil contents of 62–63% was 71%. Fractional distillation behavior was observed during the microwave treatment, as indicated by the well-defined plateaus. In addition, thermal cracking was identified as a result of the brownish vapors produced during the experiments. This thermal cracking is indicative of the cracking

of heavier compounds and the condensation of intermediate products. The distribution of radionuclides was also evaluated, and it was observed that they were concentrated in the dry wastes. Notably, despite the melting and boiling points of the radionuclides being above the maximum temperature of the experiment, radionuclide drag was observed in the irradiation products, namely the condensate and the gas scrubber. However, although the levels were detected, they were below the exemption limit. To enable regulatory acceptance of microwave pyrolysis, especially for radioactive waste, future work must prioritize (i) validation of radionuclide retention, (ii) standardized treatment protocols, and (iii) engagement with regulatory agencies.

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Data Availability All data generated or analyzed during this study are included in this published article and its Supplementary Information files. Should any raw data files be needed in another format they are available from the corresponding author upon reasonable request.

Declarations

Ethical Approval Not applicable.

Consent to Participate Not applicable.

Consent to Publish Not applicable.

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