XRF compared to ICP-OES for assessing heavy metal stabilization in medical waste incinerator ash: a cement-based solidification test

Nabaa Q. Kadhem[1], Basim A. Hussain[2], Suroor N. Esmaeel[1] and Itab F. Hussein[1*]

1 Department of Physics, College of Science, Mustansiriyah University, Baghdad, Iraq

2 Scientific Research Commission, Baghdad, Iraq..

E-mail: nabaa.qassim@uomustansiriyah.edu.iq

Abstract

Analyzing the Stabilization of Heavy Metals in Ash from Medical Waste Incinerators with XRF and ICP-OES Techniques Inappropriate disposal of hazardous medical waste presents a major risk to human health and the environment due to the release of toxic heavy metals during combustion. This study examines how well two analytical technique X-ray fluorescence spectroscopy (XRF) and inductively coupled plasma optical emission spectroscopy (ICP-OES) identify and quantify the levels of heavy metals in ash residues from three local medical waste incinerators. Heavy metal concentrations, including Fe, Nd, Ce, Sn, Sb, Ir, Th, and Au, were found to be increased in the results, beyond permissible regulatory levels. A solidification/stabilization (S/S) procedure employing regular Portland cement was used to reduce these concerns; cement ratio were 7% and 25% by weight. Post-treatment analysis showed that XRF provided more consistent and reliable detection across all samples, with treatment efficiency ranging from 89% to 100%. In contrast, ICP-OES results varied significantly, with removal efficiencies between 5% and 100%. For several high-risk elements, the S/S method achieved complete immobilization. The findings suggest that the XRF method offers superior performance in monitoring the stabilization process due to its high sensitivity and precision. The cement-based S/S use the method demonstrated high effectiveness in reducing heavy metal mobility, making the treated ash suitable for safe disposal and potential reuse in construction applications such as roadways and civil infrastructure. These results underscore the importance of selecting appropriate analytical tools for waste assessment and highlight the potential of S/S as a sustainable solution for hazardous medical waste management.

Keywords: Medical Waste Incinerators, Ordinary cement, Toxic elements, XRF, ICP-OES.

1. Introduction

The various components that comprise medical waste include used needles and syringes, body parts, drugs, diagnostic tests, blood, synthetic materials, medical equipment, radioactive materials, and surgical masks, to name a few. One may consider it a subset of all the waste generated in healthcare facilities. Healthcare personnel and the general public are exposed to physical, chemical, or microbial dangers related to the processing, disposal, and treatment of hazardous waste. [1, 2]. One of the most common methods for getting rid of medical waste is burning medical trash. One new type of pollution that is created during combustion is ash. Burying medical waste ashes in waste cemeteries is now the only way to dispose of them. [3–5]. Because the residual ash must be landfilled, even though incineration greatly reduces the amount, the problem cannot be completely remedied [6–8]. Nonetheless, bottom ash is still most commonly disposed of in designated landfills. Bottom ash treatment methods are continuously monitored and controlled to lessen the likelihood of environmental pollution. Because of its toxicity, the bottom ash in this case needs to be exanimated, and every effort should be taken to minimize the quantity of its hazardous components that seep into the environment [9, 10]. In order to prevent medical waste from ending up in landfills, where it could jeopardize the environment and public health, processing and packaging techniques are badly needed [11, 12]. The recommended method for handling fly ash and bottom ash from medical facilities is solidification/stabilization (S/S). This method stabilizes the components and makes them suitable for use in products that are healthy and ecologically beneficial [8, 13, 14]. Both physical and chemical methods are used to stabilize the dangerous heavy metals in cementitious matrices. This is related to the high alkalinity pore and the physical limited permeability of the hardened product. Therefore, there is less chance of pollutants leaking [15–17]. When bottom ash was consolidated by simply and affordably mixing it with cement material, it was

Figure 1. Sample preparation for XRF measurement.

demonstrated to be less hazardous [18]. Stabilization techniques, mixing, and using bottom ash as a material for road construction, structural support, or ceramics are some ways to manage it [16, 19]. To determine the best accurate method for assessing medical waste samples, this study set out to measure and analyze the amounts of hazardous elements using a variety of methods. The study project also intends to test and assess the effectiveness of the (S/S) procedure, which uses varying amounts of ordinary cement to clean up the waste.

Material and Methods

1. The medical waste incinerators represented various hospital tools and equipment kinds and provided samples obtained following the combustion process. The garbage was crushed using non-leakable sacks and a steel hammer. Between 90 and 120 minutes were spent grinding. A 0.7 mm sieve was then used to separate the non-flammable items. Initially, the samples' toxicity and danger were assessed using two methods:

2. **X-ray fluorescence spectroscopy (XRF)**

The XRF measurement procedure is illustrated in the diagram. 0.5 g of boric acid is added to 3 g of pre-prepared samples. This acid fixes the sample in the device mold, which is 1.5 mm wide and 0.5 mm high, when it is inserted into the compression device and a force of 20 MPa is applied. This acid does not change the composition of the sample. The compressed sample is taken and placed in the XRF device as shown in the diagram after the device has been calibrated using a unique reference source. Each sample is then left in the device for fifteen minutes. Figure 1 illustrates the sampling and XRF measurement procedure.
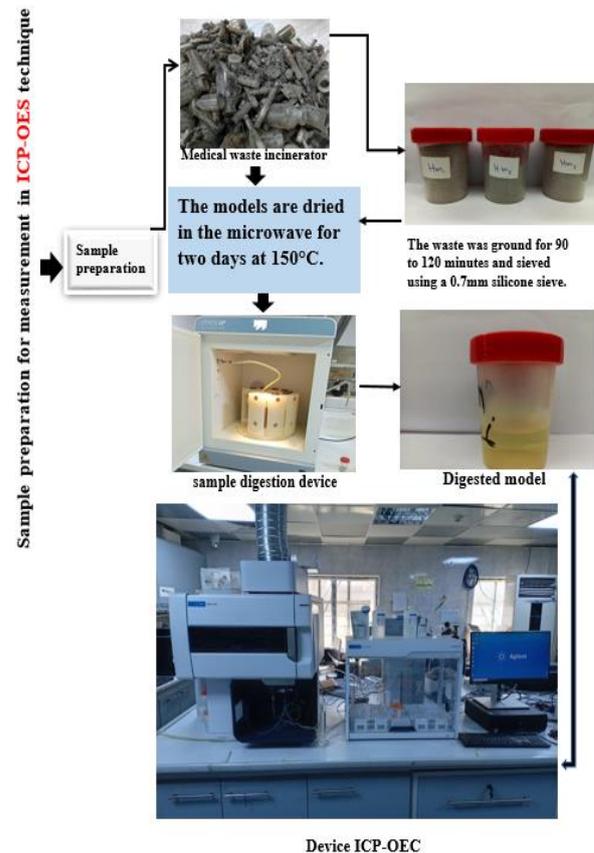

Figure 2. Preparing samples for measurement in the ICP-OES device.

2. Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-OES)

Figure 2 illustrates the sample preparation procedure for analysis using ICP-OES. Three grams of waste weight are taken and dried for two days at 150 °C in an oven. The sample is then collected and placed in the digester vessel. One milliliter of 35% H_2O_2 and nine milliliters of 69% HNO_3 are added. The digester vessel is then tightly closed and placed in the apparatus at temperatures ranging from 25 to 200 °C and high pressures between 10 and 100 bar. The apparatus is turned off after 30 minutes of digestion, and the sample vessel is taken and filtered into a 50 ml bottle. The sample is digested and then sent for measurement in the ICP apparatus after gradually raising the temperature for 15 minutes and then holding it at 200 °C for 15 minutes. The apparatus's glass tube is first acidified with HNO_3 at a rate of no more than 10% to avoid scratches. The plasma is made up of three glass tubes: the sample is kept in the inner tube, the gas that cools the outer plasma is kept in the middle tube, and the coil heats the vibrations to a temperature of 5000–10,000 K, which is followed by the ionization process. Since argon gas is inert and helps cool the inner chamber of the device, it is used. Following this procedure, each element is released through a unique emission technique before passing through a detector that determines the type and concentration of the element.
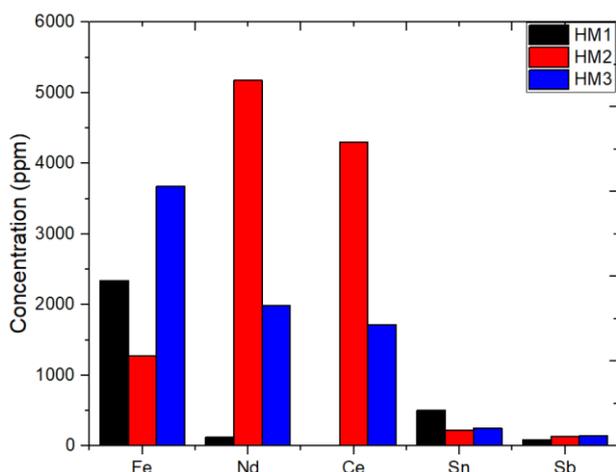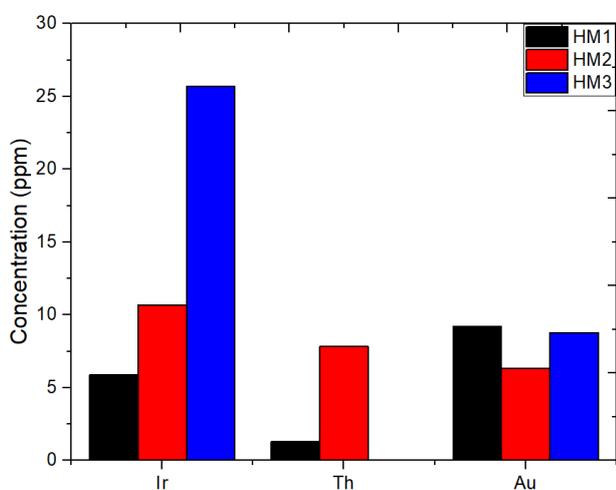
Figure 3. Element measured by XRF (ppm)

Figure 4. Concentration of Ir, Th and Au Measured by XFR (ppm)

The initial technical analysis revealed the presence of numerous heavy elements, among which eight hazardous components were selected for focused treatment. After collecting, grinding, and preparing the samples, the waste was analyzed using two distinct analytical techniques.The pre-treatment results showed that the concentrations of these toxic elements were significantly elevated, exceeding safe environmental thresholds. To mitigate this, a wet stabilization process was applied. The incinerator ash was mixed with ordinary Portland cement and red sand to enhance the binding properties and containment of hazardous substances.Two different quantities of incinerator ash were used, and the mixtures were cast into plastic molds with dimensions of 10×5 cm (length $\times$ width). The molded samples were then left to cure for 28 days under controlled environmental conditions prior to further analysis.

RESULTS AND DISCUSSION

The XRF measuring technique revealed that the three incinerators had a large number of harmful components. A total of eight components were chosen. Fe were the most noticeable components. The second incinerator had the lowest percentage, 1272 ppm, while the third

incinerator had the highest, 3675 ppm. Nd had the lowest percentage in the first incinerator (121 ppm) and the maximum concentration (5176 ppm) in the second incinerator. Ce was absent from the first incinerator and was present in the second and third incinerators at quantities ranging from 1987 to 1718 ppm. The first incinerator had the highest concentration of Sn (509 ppm), whereas the second incinerator had the lowest proportion (220 ppm). In the first incinerator, Sb had the lowest proportion (89.5 ppm) and the greatest percentage (142 ppm). The third incinerator had the highest concentration of Ir (25.7 ppm), while the first incinerator had the lowest concentration % (5.88 ppm). Th was the highest concentration in the second incinerator 7.82 ppm and the lowest percentage in the first incinerator 1.28 ppm. The third incinerator did not contain this ingredient, Au 9.20 ppm was the greatest percentage in the first incinerator, while 6.35 ppm was the lowest in the second. Hospital medical equipment, hospital capacity, manufacturing companies, medical equipment, separation method, and incinerator type all contributed to the variation in concentrations across the three incinerators. One incinerator had one room and an annex, whereas the other two had two rooms and an annex for collecting waste residues after burning. according to figure 3 and 4 and Table 1.

The concentrations of Ir, Th and Au are expressed individually to avoid negative values in the measurement as illustrated in figure 4.

Even though the samples' ICP-OES results showed the same elements assessed by the XRF technique, the concentrations were almost the same as the earlier findings. For instance, the concentration of the element iron was lowest in the second incineration (424 ppm) and greatest in the third incinerator (1225 ppm). nd The greatest percentage, 1725 ppm, was found in the second incinerator, whereas the first incinerator's was 40.3 ppm. Ce was missing from the first incinerator, while it was present in the second and third incinerators at percentages ranging from 572.6 to 662.3 parts per million. Sn had the highest concentration in the first incinerator 169.6ppm and the lowest percentage in the second incinerator 73.3ppm. Sb In the third incinerator, the highest concentration percentage was 47.33ppm and the lowest percentage in the first incinerator29.83ppm. Ir The highest concentration in the third incinerator 8.566ppm and the lowest concentration percentage in the first incinerator 1.32ppm. Th The concentration percentage was very low in the first and second incinerators (0.42-2.60ppm) And it was absent in the third incinerator, Au concentration ranged between (2.11-3.06 ppm). The results of the XRF technique showed the presence of concentrations higher than the permissible limit, even if the concentrations of all components were reduced by different percentages. Additionally, as shown in figure 5 and Table 2, it was shown that there was a correlation between the absence of specific materials in the same incinerators and the existence of large percentages in the same incinerators.
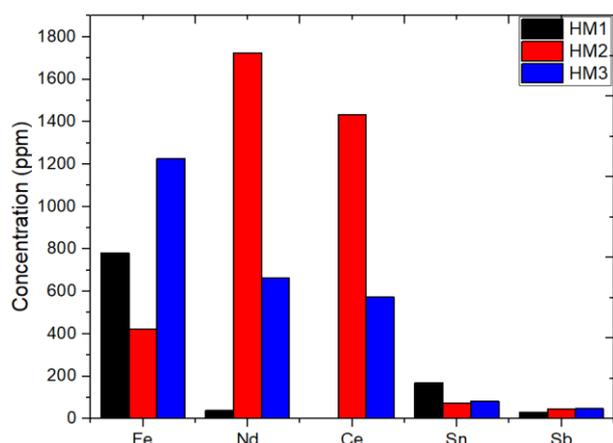
Figure 5. Heavy element measured by ICP-OES(PPM).

The concentrations of Ir, Th, and Au are expressed individually to avoid negative values in the measurement as illustrated in figure 6.
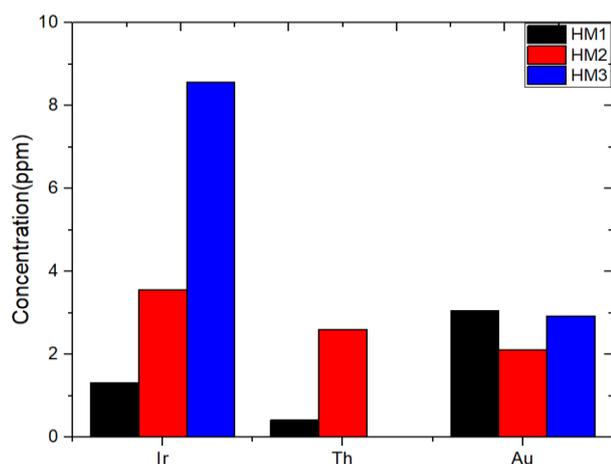

Figure 6. Concentration of Ir, Th and Au Measured by ICP-OES (ppm)

Statistical Analysis

The correlation between the two approaches was calculated for all element concentrations. The results showed a strong correlation and close link between the two elements. They showed that the elements Ce and Nd had a strong bond, reaching 0.99, and that the elements Sb had a strong bond with both Nd and Ce, reaching (0.95-0.96). According to Table (3) in below, a positive correlation between the elements Au, Fe, Ir, and Sb can be observed.

The trash was treated by encapsulating and fixing the elements inside cement concrete made from regular cement after the findings of the two approaches revealed several hazardous poisonous elements over the allowable limit. Red sand and regular cement were combined, and 100 and 300 grams of incinerator ash were added. It was observed that the components' concentrations dropped both to and below the allowable level. According to the data, the XRF approach had a treatment efficiency of 99% for the element Fe, 90% for the element Sn, and 89% for the element Sb. In contrast, each of the elements Nd, Ir,

Th, and Au had a treatment efficiency of 100%. In contrast, ICP-OES data indicated that Fe had a 94% treatment efficiency, Sn had a 9% treatment efficiency, and the Sn element had a 5% treatment efficiency. However, Nd, Ir, Th, and Au all had 100% treatment efficiency, as illustrated in figures 7 and 8 and Table 4. It was demonstrated that a 25% cement ratio produced superior outcomes to a 7% mixture. It was discovered that many dangerous substances were processed and kept using ordinary cement. Furthermore, it was shown that XRF technique was the most effective and precise way to ascertain the concentrations of heavy metals in this type of sample. These results supported the study [16, 20].

Conclusion

According to the study's findings, medical waste incinerators include a range of dangerous heavy elements, with the concentrations of these elements varied based on hospital equipment, operational capacity, and waste disposal procedures. The environment, public health, and workplace safety are all seriously threatened by these factors, particularly when trash is not adequately managed and suitable protective measures are not in place. When the two analytical methods were compared, it was found that X-ray fluorescence (XRF) produced better results than inductively coupled plasma optical emission spectroscopy (ICP-OES). The improved ability of XRF to identify heavy metals in intricate matrices, like incinerator ash, is credited with this superiority. Analysis of the eight most important heavy metals revealed that medical waste incinerator ash is extremely toxic and poses a major environmental risk, with each metal surpassing globally accepted safety levels. This discovery prompted the use of a cement-based encapsulation technique, which effectively immobilized the hazardous substances inside a solid matrix. This method of stabilization and solidification worked very well to restrict the environmental impact of heavy metals, decrease their mobility, and contain them. Furthermore, it was demonstrated that using regular Portland cement offered both excellent treatment efficiency and economic viability. In addition to disposal, the stabilized ash-cement composite shows promise for repurposing in infrastructure projects like land reclamation and bridge building, providing a sustainable method of managing hazardous waste.

Data availability

The data that support the findings of this study can be obtained from corresponding authors upon reasonable request.

Table 1. Heavy element measured by XRF technique

Sample names	Heavy Metal Concentrations(ppm)							
	Fe	**Nd**	**Ce**	**Sn**	**Sb**	**Ir**	**Th**	**Au**
HM1	2344±4.66	121±35.1	-	509±3.07	89.5±1.48	5.88±0.81	1.28±0.42	9.20±0.53
HM2	1272±4.01	5176±43.3	1987±12.9	220±1.96	139±1.54	10.7±0.81	7.82±0.37	6.35±0.533
HM3	3675±6.29	4298±42.5	1718±12.4	252±2.19	142±1.66	25.7±1.17	-	8.79±0.60

Table 2. Heavy element measured by ICP-OES technique

Sample names	Heavy Metal Concentrations(ppm)							
	Fe	**Nd**	**Ce**	**Sn**	**Sb**	**Ir**	**Th**	**Au**
HM1	٧81.3	40.3	-	169.6	29.83	1.32	0.42	3.06
HM2	424	1725	662.3	73.3	46.33	3.56	2.60	2.11
HM3	1225	1432.6	572.6	84	47.33	8.566	-	2.93

Table 3. Comparison of the correlation between the two technologies

	XRF									ICP-OES							
	Fe	**Nd**	**Ce**	**Sn**	**Sb**	**Ir**	**Th**	**Au**		**Fe**	**Nd**	**Ce**	**Sn**	**Sb**	**Ir**	**Th**	**Au**
Fe	1								**Fe**	1							
Nd	-0.010	1							**Nd**	-0.010	1						
Ce	-0.004	0.99	1						**Ce**	-0.004	0.99	1					
Sn	0.0015	-0.99	-0.99	1					**Sn**	0.006	-0.99	-0.99	1				
Sb	0.012	0.95	0.96	-0.97	1				**Sb**	0.012	0.95	0.96	-0.97	1			
Ir	0.58	0.31	0.35	-0.37	0.52	1			**Ir**	0.51	0.38	0.42	-0.44	0.59	1		
Th	-0.82	0.25	0.22	-0.20	0.098	-0.18	1		**Th**	-0.82	0.26	0.22	-0.20	0.1	-0.18	1	
Au	0.56	-0.53	-0.49	0.47	-0.32	0.023	-0.92	1	**Au**	0.57	-0.52	-0.48	0.46	-0.31	0.25	-0.92	1

Table 4. Concentration of element after treatment (ppm)

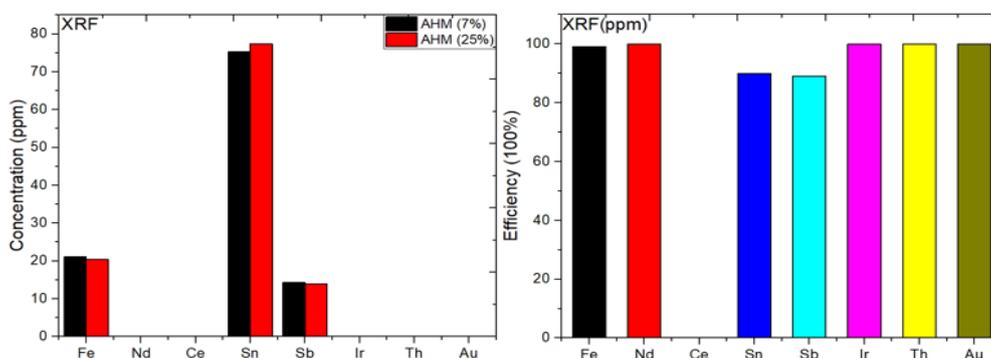| ICP-OES Technique	Concentration of element after treatment (ppm)							
	Fe	**Nd**	**Ce**	**Sn**	**Sb**	**Ir**	**Th**	**Au**
AMW (7%)	7.03	ND	ND	25.1	4.76	ND	ND	ND
AMW (25%)	6.8	ND	ND	25.8	4.66	ND	ND	ND
XRF Technique	**Concentration of element after treatment (ppm)**							
	Fe	**Nd**	**Ce**	**Sn**	**Sb**	**Ir**	**Th**	**Au**
AMW (7%)	21.1	ND	ND	75.3	14.3	ND	ND	ND
AMW (25%)	20.4	ND	ND	77.4	14	ND	ND	ND

Figure 7. Concentration and efficiency of toxic elements after treatment.
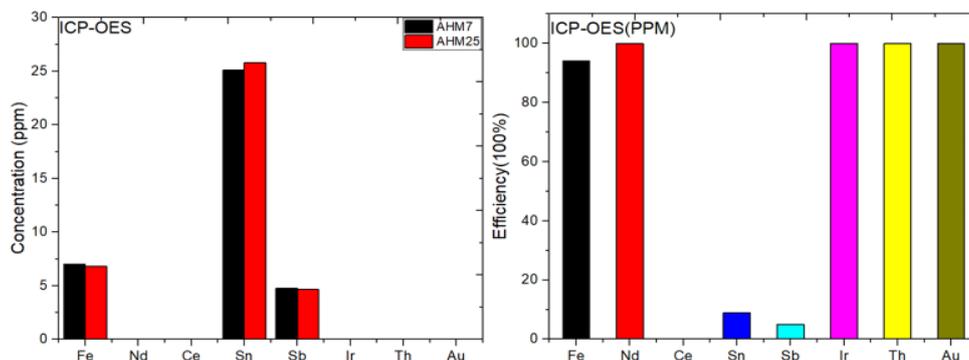

Figure 8. Concentration and efficiency of toxic elements after treatment.

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