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Analytical procedures using SEM/EDX equipment for determining heavy metals contents in fly ash genereated from domestic solid waste incinerators: a casestudy of Soc Son waste to power plant in Ha Noi

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Abstract. The researching and establishing of this process for analyzing fly ash generated from a domestic solid waste treatment plant by Scanning Electron Microscopy Energy Dispersive X-Ray (SEM/EDX) method were investigated. Three sampling series with twelve (12) fly ash samples collected at SocSon waste-to-power plant Ha Noi, Viet Nam were used for: (i) analytical procedure establishment; (ii) standardization; and (iii) test analysis. At each

measurement, a cross-check with one random sample using X-ray fluorescence spectroscopy (XRF) method was carried out to verify the accuracy of the SEM/EDX measurements. As a result, a 6-step measurement procedure has been proposed, including: experimental preparation, sample weighting, liquid/solid phase separation, sample drying, sample measurement and data processing. And for procedure adjustment, liquid/solid phase separation was repeated twice while drying time was doubly increased from 24 to 48 hours to collect all heavy metal contaminated residues. The proposed procedure could provide results in about 3-5 seconds without any requirement of sample digestion while most of the heavy metalspresent in fly ash, including low-content ones such as Ag, Cd and Cr were simultaneously displayed. The standardized measurement procedure was applied to test the hazardous properties of fly ash by comparing with Technical regulations (QCVN) 07:2009/BTNMT – National Technical Regulations on Hazardous waste thresholds. The results identified that fly ash could be primarily considered as hazardous waste with 5/14 heavy metal parameters exceed the limit from 1.7 to 9.5 times and needs to be collected and treated properly. This result is also quite similar to the studies that have been done in China, the US or Viet Nam. Thus, SEM/EDX method could be used for analyzing heavy metal components in fly ash samples of solid waste treatment plants.

Keywords: fly ash, SEM/EDX, analytical process, heavy metals, hazardous waste

Classification numbers: 3.3.2

1. INTRODUCTION

Soc Son waste to power plant started operating in the second quarter of 2022 with a total capacity of 4,000 tons/day. The goal of this plant is treating most of the Municipal Solid Waste (MSW) volume of Ha Noi, Viet Nam [1]. This is the largest waste to power plant in Viet Nam and Southeast Asia and the second one in Viet Nam to come into operation. The process of burning garbage, using limestone and activated carbon to treat flue gas leads to the formation of a certain amount fly ash, which is equivalent to about 1.8 - 2 % of the input solid waste [1, 2].

There have been quite a few studies on the composition of fly ash, mostly in China and some industrialized countries [3 - 8]. According to these previous studies, fly ash has a spherical structure and is classified as hazardous waste as it contains heavy metals, dioxins/furans and some other compounds [4, 5, 7, 8].

For previous years in Viet Nam, most of MSW was treated by landfilling. This mitigation measure, however, is no longer considered as a suitable solution due to its disadvantages such as large area occupation as well as generation of leachate, odor [9]. Currently, a new approach with heat recovery for electricity generation is initially considered more feasible than the landfilling method [10]. However, handling of output waste, including its fly ash is still inadequate for this new approach as it is quite new in Vietnam.

SEM/EDX is an elemental detection and analysis for particles with differential dimensions. The morphology and detail of elemental composition are providing visual results of this method. In addition, it is also possible to compare chemical properties in the form of spectrograms and displaying the elemental distribution map of substances present on the sample. The advantages are the fast time for results, about a few hours and no needing to conduct sample digestion. However, when applied to solid samples in general or fly ash in particular, there is no standard analytical procedure. Most of the analysis operations are automated on measuring instruments therefore, the accuracy of the measurements has not been verified.

For studying the morphology, structure or metal composition of waste incinerator fly ash, ICP, ASS, ICP-MS or ICP-OES and XRF are mostly used [11 - 14]. Each techniques have its certain advantages and limitations to be selected for proper use. When the required limit for quantitative analysis is higher than 1 ppm (μg/g) and when non-destructive analysis of the sample is required, XRF is the optimal choice that should be considered, especially when analyzing solids, powders, fibers, filters and oils. In contrast to ICP and AAS, XRF spectroscopy requires no sample dissolution or treatment, allowing for non-destructive analysis. By avoiding errors due to incomplete dissolution and the large degree of dilution when prepared, the complete analysis work done by XRF helps to ensure the accuracy and reliability of the results [11 - 16].

This study, thus, aims at: (1) establishing a new fly ash analytical procedure using SEM/EDX; (2) Verifying the accuracy of the new measurement procedure through XRF technique for process comparison; and (3) Applying the propose measurement procedure for analysis of heavy metals contained in fly ash of Soc Son waste-to-power plant, Ha Noi, Viet Nam.

2. MATERIAL AND RESEARCH METHODS

2.1. Equipment

Equipment at the laboratory of the Institute of Physics of the Vietnam Academy of Science and Technology include: Hitachi SEM electron microscope model 8100; Bruker SDD Detector (EDX); Centrifugal Rotary Machine; Vibrator; Drying oven; Jewelry scales, etc. This is a method for detecting and analyzing elements for details with microscopic dimensions, providing visual results on morphology and details on elemental composition.

2.2. Sample preparation

Fly ash is a solid specimen. The sampling proceeds are followed TCVN 7538 - 2: 2005 - Soil quality - sampling - Sampling technique manual. Samples were taken at the outlet of the fly ash silo, (i.e., before conveyed into the jumbo) or can be taken in the ash storage. Samples were divided and stored separately in individual zip bags, then arranged in each tray and put in a desiccator to avoid the influences of the external factors.

The number of samples is divided into 03 phases for its purpose. At each phase, four (04) samples corresponding to the vibration frequency of the cloth bag dust filter system were taken. After that, one (01) sample was randomly selected for control by XRF method:

+ Phase 1 (July 2022) Testing and developing the process: sample symbols from FA-1.1 to FA-1.4.

+ Phase 2 (September 2022) Process standardization: sample symbols from FA-2.1 to FA-2.4.

+ Phase 3 (December 2022) Process analysis: sample symbols from FA-3.1 to FA-3.4.

3. RESULTS AND DISCUSSION

3.1. Process setting experiment

According to previous studies, fly ash contains a large amount of soluble salts and alkalis [3 - 5, 18]. In Viet Nam, series of studies by Cao Tien Phu *et al.* also have similar observations when piloting the ash of the MSW incinerator in Can Tho, Viet Nam [18, 19]. Thus, it is necessary to reduce the content of Cl-, dissolved alkali ions in order to accurately assess the heavy metal contents in the fly ash. Based on the SEM/EDX manual, the experimental steps to build the procedure are as follows:

- Step 1: Preparation of the experiment

Use 15 ml plastic test tubes for solid samples, 20 ml capacity glass vials for liquid samples. The vials all have tight-fitting lids and are cleaned to remove impurities.

- Step 2: Weigh the sample

+ Weigh the test tubes and glass jars

+ Weigh about 1 g of fly ash sample and put into a test tube

- Step 3: Separation of liquid - solid phase

+ Add 3 ml of distilled water to the test tube with fly ash for separating liquid and solid phases

+ Shake well in a vibrator for > 1 minute to ensure 100 % dissolution of solids and wash away chloride or alkali radicals.

+ Put the test tubes containing samples + distilled water into the centrifuge for 5 - 7 minutes. This process separates the sample into two phases: a solid phase at the bottom and a liquid phase at the top.

+ Use a pipette to transfer the liquid phase to a glass vial.

+ Solid phase will be used to measure heavy metal composition in fly ash.

- Step 4: Dry the treated sample

+ The tubes containing solids will be put into a medical oven at a temperature of 60 ^{o}C to dry the solids.

+ The drying process will be monitored daily. Maintaining the oven temperature at 60 ^{o}C doesn't require the denaturing of the components in the fly ash.

+ Perform the drying process in 24 hours

- Step 5: Measure the sample

After drying the solid, the fly ash sample was measured using a Hitachi 8100 SEM combined with a Bruker SDD Detector (EDX).

+ Take a small amount of solid for the sample plate with carbon paper.

+ Use a rubber ball to blow off excess dust. Make sure that the sample is clean before putting it into the sample chamber.

+ Insert the sample disc into the measuring device according to the user manual and start the device. Measurements were made using an electron beam with energy of 25 kV at a distance of 1.5 mm.

+ When a high-energy electron beam is shone on the fly ash sample, X-ray spectra with characteristic frequencies are generated and analyzed by an energy dispersive spectrometer thus recording information about the elements as well as the percentage of components in the sample.

+ The instrument will give the result as a table of elements in fly ash with X-ray dispersion spectrum of the fly ash sample.

- Step 6: Process the data

+ Convert fly ash composition data (content %) to ppm (mg/kg).

+ The results of fly ash elemental composition will be processed by statistical mathematical methods with reliability LSD = 0.05.

Table 1. Results of measurement of fly ash composition phase 1 – testing phase, process development (ppm).

Element	SEM/EDX				Gradient speed	XRFFA-1.3
	FA - 1.1	FA - 1.2	FA - 1.3	FA - 1.4		
Mg	18,521	21,010	21,405	19,430	1,350	22,618
Al	18,728	22,090	23,134	21,456	1,881	24,675
Si	19,208	18,129	16,983	16,145	1,337	18,023
P	17,542	15,100	14,870	15,890	1,209	16,244
S	7,255	8,459	8,750	8,734	709	9,623
K	8,754	8,350	9,102	8,012	474	9,930
Ca	229,860	229,830	213,876	248,034	13,959	240,673
Ti	8,341	7,545	7,530	8,535	526	8,429
Fe	24,010	20,360	22,014	23,345	1,611	22,739
Zn	12,509	12,049	12,190	12,098	206	12,602
Pb	2,115	2,404	2,542	2,103	217	2,678
Cr	241	291	283	271	22	295
Ag	180	159	169	176	9.2	182
Cd	91	89	88	88	1.4	91

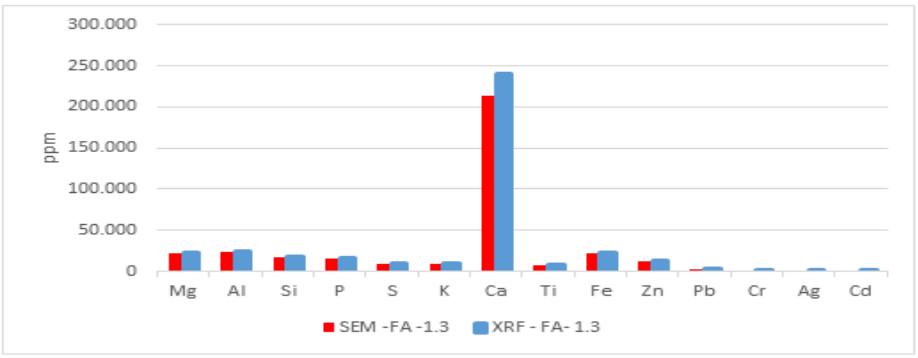

Figure 1. Comparison chart of fly ash composition of Soc Son Waste Power Plant - phase 1 by SEM/EDX and XRF.

+ Compare and cross-check with the results of fly ash measurement by XRF method combined with Silicon drift detector (SDD) (measured at Klamag key laboratory) to verify the accuracy of EDX measurement.

The results of the first phase of sample measurement are shown in Table 1 and Figures 1, 2 below:

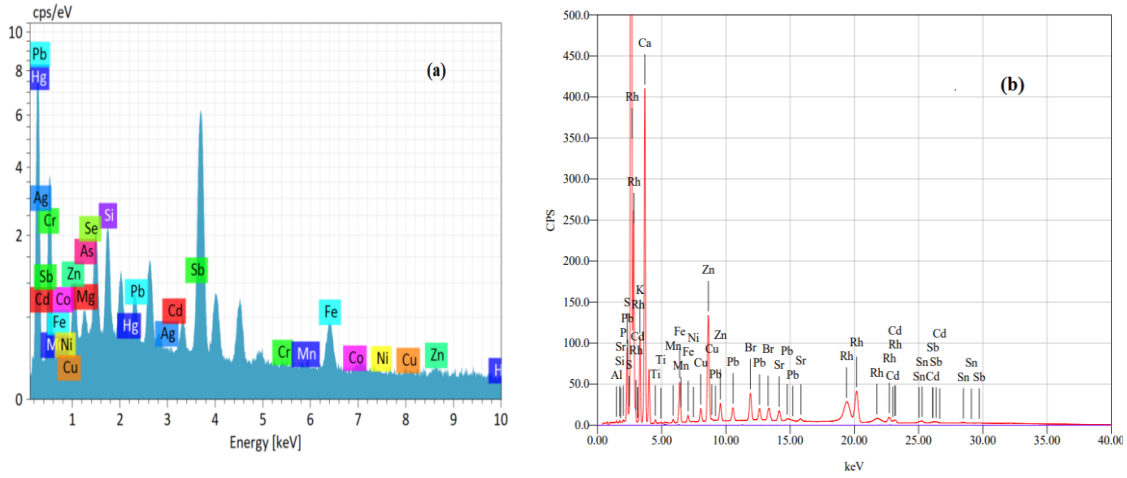

Figure 2. The Energy dispersive X-ray spectrum of the fly ash by SEM/EDX (a) and XRF (b).

Thus, the 1st phase result showed a relative high error rate on analytical values between SEM/EDX and XRF techniques, ranging from 3 - 85 on average for heavy metals such as Mg, Al, Si, Pb, Cr, Ag, Fe, Zn, and Cd and from 8 - 12 % for particular case of others such as P, S, Ca, K, Ti. These relative high error rates require the measurement procedure to be standardized for to lessen the error rate. These relatively high error rates require the measurement procedure to be standardized to lessen the error rate. The reason could be attributed to incomplete washing and separation of liquid/solid phase that leaves a large content of chloride-based salt compounds available in the measured sample. This could be observed through the energy dispersive X-ray spectrum of the fly ash sample when measured by this SEM/EDX method. At the same time, as the measuring chamber of the SEM/EDX is at vacuum, so if the solid sample is not thoroughly dried, it would lead to errors in sample measurement.

According to the results shown in Figure 2, the characteristic X-ray spectra of the elements measured by the XRF method has been background-corrected and measured within the energy range of 0 to 30 KeV with high resolution, while the SEM EDX spectra was only measured within the range of 0 to 10 KeV.

3.2. Measurement and analysis sampling procedure

As previous commented, error in fly ash analysis could be attributed to incomplete separation of liquid-solid phase as well as undried solids with high humidity, leading to inaccuracy of SEM/EDX measurements. Therefore, the liquid/solid phase separation process will be repeated twice in step 3 to thoroughly collect the sediment. Also, the drying time (step 4) is increased from 24 to 48 hours to ensure that the sample is completely dry before performing analysis. The steps of this process are as follows (Figure 3).

After standardizing the process, 02 fly ash samples (batch 2) were included in the control analysis. The results are shown in Figure 4.

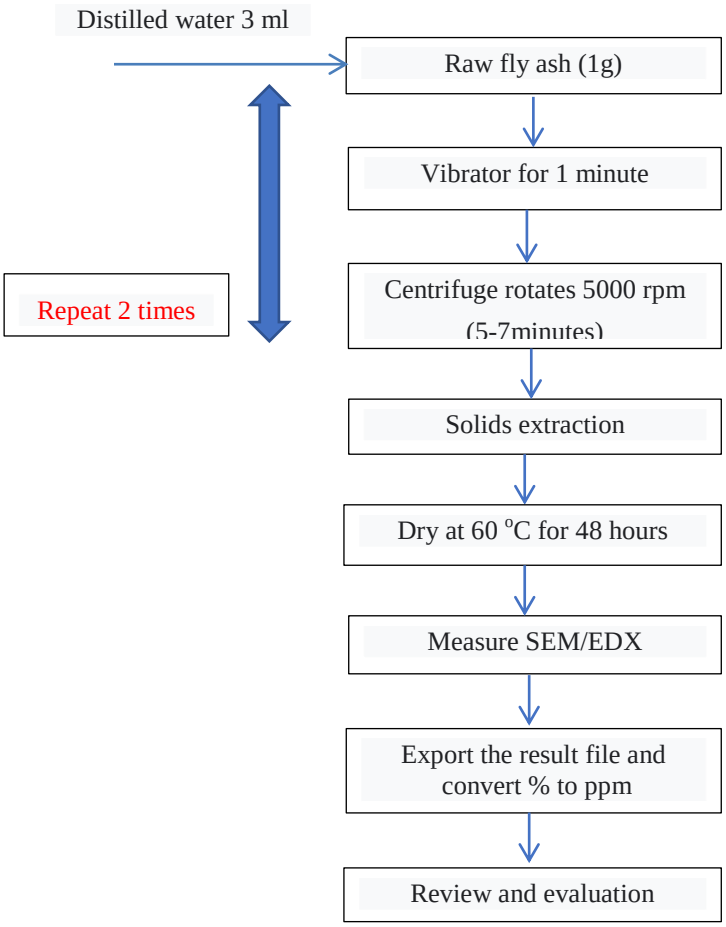

Figure 3. Process of analyzing fly ash composition by SEM/EDX method.

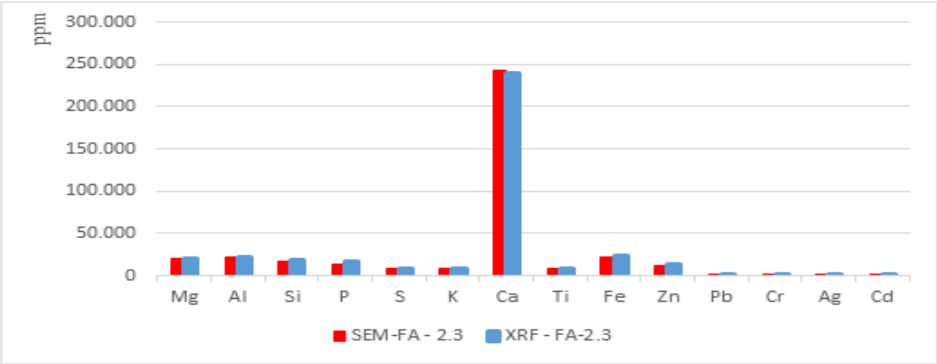

Figure 4. Comparison chart of fly ash composition of Soc Son Waste Power Plant - phase 2 by SEM/EDX and XRF.

The results at this stage show that the error rate between the two measurements SEM/EDX and XRF has decreased significantly and all indicators have the difference value < 5 %. For metal indicators such as Mg, Al, Si, Pb, Cr, Ag, Fe, Zn, Cd, the error rate decreased from 3 - 8 %

to 3 - 4 %. For organic parameters such as P, S, Ca, K, Ti, the error rate decreased from 8 -12 % to 1 - 3 %.

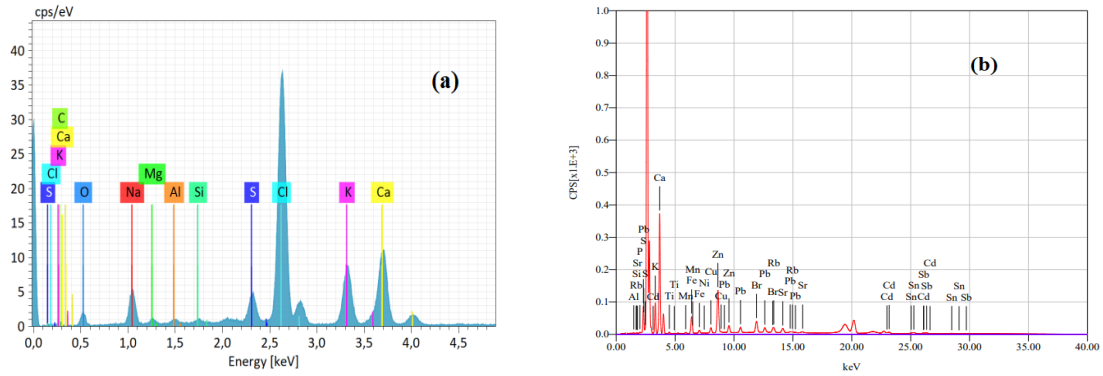

Figure 5. The energy dispersive X-ray spectrum of the fly ash by SEM/EDX (a) and XRF (b).

In general, the above margin of error is acceptable for a measurement that requires fast results without requirement of sample digestion. In the energy dispersive X-ray spectrum of the fly ash measured by SEM/EDX, no chloride radicals were detected. It proved that repeated extraction twice reduced the chloride salt compounds in the fly ash and the normalization of the process measurement is effective.

3.3. Analysis of fly ash samples at Soc Son Waste Power Plant

When applying the established and calibrated process the authors analyzing the 3rd phase to evaluate the hazardous properties of ash according to current technical regulations, as detailed in Table 2.

Table 2. Results of 3rd phase measurement – analysis according to standardized procedures (ppm).

Element	SEM/EDX				Gradient speed	XRF	QCVN 07:2009/ BTNMT
	FA – 3.1	FA – 3.2	FA – 3.3	FA – 3.4		FA 3.3	
Mg	17,476	21,119	21,742	20,284	1,883	22,629	-
Al	21,348	22,054	23,925	22,356	1,088	24,722	-
Si	19,869	18,101	17,164	16,270	1,539	17,822	-
P	16,016	15,292	15,041	15,937	480	15,459	-
S	7,520	8,635	9,310	8,773	751	9,476	-
K	8,738	8,447	9,403	8,281	495	9,674	-
Ca	242,700	235,212	222,831	228,259	8,614	229,773	-
Ti	8,576	7,597	7,618	8,735	608	7,735	-
Fe	24,758	21,222	22,365	24,225	1,640	22,985	-
Zn	12,899	11,859	12,899	12,025	556	13,245	5,000
Pb	2,162	2,434	2,755	2,240	264	2,875	300
Cr	267	298	285	279	13	295	100
Ag	190	170	164	177	11	170	100
Cd	86	97	70	79	11	73	10

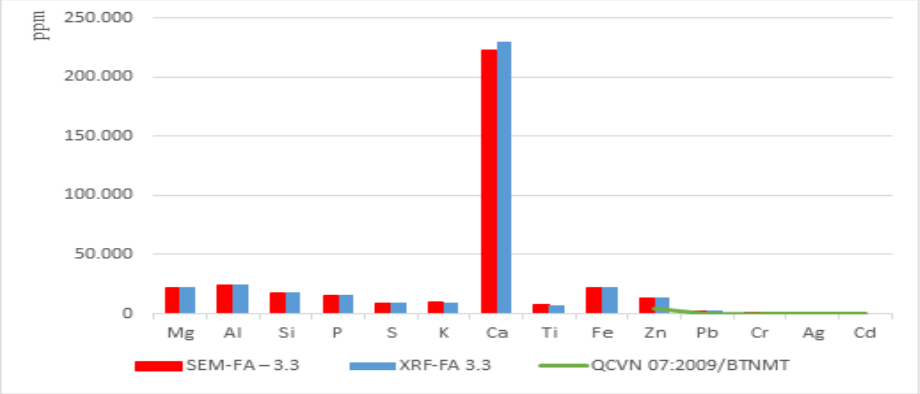

Figure 6. Comparison chart of fly ash composition of Soc Son Waste Power Plant - phase 3.

Comparison of fly ash composition measured by SEM/EDX and XRF methods showed that the results are almost similar. The margin of error between 2nd and 3rd measurements for most indicators remained <5%. Thus, it is possible to put this analysis process into application deployment with a larger number of samples and more indicators.

The application of the process in the 3rd phase sample analysis was compared with QCVN 07:2009/BTNMT, National Technical Regulation on hazardous waste thresholds. The results showed that 5/14 metal indicators specified in the regulation exceeded the allowable limit 1.7-9.5 times (Figure 4). In Cr, Cd indicators particularly hazardous cities, exceeding the standards by 2.95 and 7.3 times, respectively; Pb indicator, as a toxic metal that can cause damage to the nervous system, brain and blood disorders, is exceeding the standard 9.5 times.

Thus, the initial results showed that the fly ash samples from SocSon waste-to-power plant contains high content of heavy metal that could be classified as hazardous waste and needs to be collected and treated properly. This result is also quite similar to the studies conducted in China, the US or Vietnam [4, 5, 7, 8, 20 - 23]. However, when referring to the results of Cao Tien Phu studying fly ash samples at the Can Tho Waste Incineration Plant, only 02 indicators Pb and Cd exceeded the allowable limit [18, 19]. This difference could be attributed to mixing of Can Tho fly ash mixed with chelate for stabilization and solidification before treatment [24].

The experiment through three sampling series with twelve (12) fly ash samples collected at Soc Son waste-to-power plant to propose a new measurement procedure showed that:

- This tool and measurement procedure could be used to analyze heavy metal components in fly ash samples after flue gas treatment step at the solid waste treatment plants. Measurements provides quick results, in about 3-5 seconds without breaking the sample. The measurement can display most of the metals present in fly ash simultaneously, including low-content metals such as Ag, Cd, and Cr.

- In order to cross-check with XRF method, a random sample was sent to the Klamag key laboratory for analysis to ensure the objectivity of the measurement as well as to verify the process. The results showed that after the process standardization (i.e., by performing 02 times of liquid-solid phase separation and increasing the drying time from 24 to 48 hours the error rate between the two measurements significantly reduced from 3 - 12 % to < 5 % for all parameters.

- The results of experimental analysis of 04 fly ash samples at Soc Son Waste to Power Plant showed that fly ash from Soc Son Waste Power Plant is hazardous waste, by containing

some heavy metals that are toxic to the environment. This is the basis for this factory to properly collect and treat in accordance with the Law on Environmental Protection 2020 of Vietnam and current regulations.

4. CONCLUSIONS

The purpose of this study is to develop a process to analyze metal content in fly ash generated from solid waste incinerators using SEM/EDX technique. The target is to provide relatively accurate results without sample digestion required and analysis time < 7 days. Twelve (12) samples of fly ash were taken in the period 7/2022 - 12/2022 during 03 phases: testing, process development and standardized and analyzed according to the established procedure. At each stage, samples were analyzed simultaneously/control with the XRF method at another laboratory to adjust the process and increase measurement accuracy.

The process of analyzing heavy metal components in fly ash by SEM/EDX method has been developed. This process includes 6 steps, from sample preparation to output data processing. Time needed to obtain results is about 3-5 second with small sample volume and without sample digestion.

Experimental results of the 3rd phase of fly ash samples showed that SEM/EDX method indicates most of the metals available. The results of the analytical metal content according to the established measurement procedure are quite similar to the XRF measurement, a proven method with higher accuracy.

SEM/EDX measurement process was applied to test heavy metal content in fly ash of Soc Son Waste to Power Plant with 04 samples (phase 3). Some heavy metal indicators exceed the allowable limit of QCVN 07:2009 /BTNMT from 1.7 to 9.5 times, initially identifying fly ash as hazardous waste. This is similar to the results of previous studies of the US, China and Vietnam [20-23]. Thus, fly ash needs to be treated appropriately according to regulations on hazardous waste before being discharged into the environment.

Therefore, SEM/EDX can be used to determine the heavy metal composition particularly for fly ash in particular and for solid samples in general. However, it is necessary to follow the established procedure to avoid errors that affect the results of the measurement.

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Declaration of competing 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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