Sains Malaysiana 48(8)(2019): 1655–1660
http://dx.doi.org/10.17576/jsm-2019-4808-11

Determination of Heavy Metals and Radionuclides in Coal and Industrial Fly Ash by Neutron Activation Analysis (NAA) and Gamma Spectrometry

(Pengenalpastian Logam Berat dan Radionuklid pada Arang dan Abu Terbang Industri melalui Analisis Pengaktifan Neutron (NAA) dan Spektroskopi Gamma)

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ABSTRACT

Coal-fired power plants and industrial waste (IW) incinerators increasingly dispose large amounts of fly ash that cause environmental contamination by toxic heavy metals. This study aimed to investigate the concentration of heavy metals and measure the specific activity of naturally occurring radioactive materials (NORM) that remain in the fly ash of coal power plants and industrial incinerators. Ash samples were collected from the Kapar and Jana Manjung coal-fired power plants and the Kualiti Alam industrial incinerator in Malaysia. The concentrations of As, Ba, Co, Cr, Fe, Sb, Zn, Th and U were determined via neutron activation analysis (NAA). Results indicated that the highest concentrations of heavy metals in Kapar and Jana Manjung coal fly ash were 27 012 and 57 500 mg/kg for Fe and 494 and 1,119 mg/kg for Ba, respectively, and the lowest concentrations for Sb were at 3.67 and 2.23 mg/kg, respectively. The concentrations of Fe, Zn, Ba and Cr in industrial fly ash were 31 0007, 7 675, 2 760 and 1,029 mg/kg, respectively. The concentrations of As, Ba, Cr, Fe and Zn were higher in industrial fly ash than in coal fly ash. The specific activity concentrations of NORM, namely, ^{40}K, ^{226}Ra, ^{232}Th and ^{238}U determined by gamma spectrometry in Kapar coal fly ash were 321.65, 27.42, 134.41 and 152.71 Bq/kg, respectively. Elemental concentration results indicated that the amounts of heavy metals depended on feed sources and combustion temperatures. Most heavy metal contents and radionuclides in power plant and incinerator fly ash were significantly lower or within the global ranges.

Keywords: Fly ash; heavy metals; naturally occurring radioactive materials (NORM); Neutron Activation Analysis (NAA)

ABSTRAK

Loji janakuasa berasaskan arang batu dan insinerator sisa industri (IW) berterusan melepaskan sejumlah besar abu cerobong yang menyebabkan pencemaran alam sekitar disebabkan kandungan logam berat bertoksik. Kajian ini bertujuan untuk mengkaji kepekatan logam berat dan mengukur aktiviti khusus bahan radioaktif terjadi secara alami (NORM) yang masih bersisa di dalam abu cerobong dari loji janakuasa arang batu dan insinerator industri. Sampel abu telah diambil dari loji janakuasa arang batu Kapar dan Jana Manjung dan insinerator industri Kualiti Alam di Malaysia. Kepekatan As, Ba, Co, Cr, Fe, Sb, Zn, Th dan U ditentukan melalui analisis pengaktifan neutron (NAA). Keputusan menunjukkan bahawa kepekatan tertinggi logam berat dalam sisa abu cerobong di Kapar dan Jana Manjung adalah 27,012 dan 57,500 mg/kg untuk Fe dan 494 dan 1 119 mg/kg untuk Ba, dan kepekatan terendah bagi Sb adalah 3.67 dan 2.23 mg/kg. Kepekatan Fe, Zn, Ba dan Cr dalam industri abu cerobong adalah 31,0007, 7,675, 2,760 dan 1,029 mg/kg. Kepekatan As, Ba, Cr, Fe dan Zn adalah lebih tinggi dalam abu cerobong industri daripada abu cerobong arang batu. Kepekatan aktiviti khusus daripada NORM, iaitu ^{40}K, ^{226}Ra, ^{232}Th dan ^{238}U ditentukan oleh spektroskopi gama di abu cerobong arang batu Kapar masing-masing adalah 321.65, 27.42, 134.41 dan 152.71 Bq/kg. Kepekatan unsur menunjukkan bahawa jumlah logam berat bergantung kepada sumber bekalan dan suhu pembakaran. Kebanyakan kandungan logam berat dan radionuklid di loji janakuasa dan insinerator abu cerobong adalah lebih rendah dengan ketara atau dalam julat global.

Kata kunci: Abu cerobong; analisis pengaktifan neutron (NAA); bahan radioaktif terjadi secara alami (NORM); logam berat

INTRODUCTION

The term 'fly ash' is a generic term applied to all residues produced from the combustion of various materials. These ashes differ from one another because of divergent physical, chemical and consequently toxicological properties. During coal or solid waste combustion, elements are redistributed into bottom ash, electrostatic precipitator fly ash and stack-emitted materials. Fly ash captures most elements, therefore, it is considered as the most important by-product of coal or waste combustion (Ferré-Huguet et al. 2007; Goodarzi 2006; Singh et al. 2016).

Similar to many countries, Malaysia utilises coal as one of its energy resources for electricity generation, and the utilisation of coal likely increases rapidly compared with those of other energy resources (Mohd Annas & Mohd Nor 2005). Coal combustion and burning municipal and industrial solid waste annually produce large

quantities of by-products, such as fly and bottom ash. For instance, the coal ash produced worldwide is estimated to be more than 600 million tons per year (Dwivedi et al. 2008). The estimated amount of fly ash in 2000 that was generated by burning municipal solid waste in the USA, Japan and the European Union reached approximately 25 million tons per year. Ash from coal combustion or industrial waste (IW) incineration is typically discarded in lagoons, settling ponds, landfills or other areas (Reijnders 2005).

Power plant and industrial incinerator fly ash emitted into the environment represents a significant source of potential toxic elements such as As, Cd, Hg, Pb and Se which cause air pollution, and water and soil contaminations. Landfilling is mostly used for the disposal of large quantities of fly ash. Consequently, some heavy metals can leach out of ash ponds and contaminate the soil, surface and ground water (Chen et al. 2014; Dai et al. 2018; Erol et al. 2007; Meij & Winkel 2007; Singh et al. 2016, 2010; Sushil & Batra 2006). Concentration and accumulation of these metals in fly ash depend on feed coal sources, combustion methods and pollution control devices at a plant (Papaefthymiou et al. 2007).

Coal fly ash is a highly heterogeneous material composed of silicon dioxide, aluminium oxide and iron oxide amongst other minerals and may contain significant amount of various elements, such as As, Pb, Ba, Hg, Cd, Cr, Co, Ga, Nb, Ni, Rb, Sr, V, Zr, Th and U, at varying concentrations; some of them are radioactive and hazardous (Goodarzi 2006; Jegadeesan et al. 2008; Malik et al. 2016; Querol et al. 1995; Sijakova-Ivanova et al. 2011). Various analytical techniques, such as atomic absorption spectrometry (Erol et al. 2007), inductively coupled plasma-mass spectroscopy (Goodarzi 2006), X-ray fluorescence (Dogan & Kobya 2006; Malik et al. 2016) and neutron activation analysis (Fardy et al. 1989; Goodarzi 2006; Orvini & Pirico 1995), have been applied to determine heavy metals on milled coal and coal combustion by-products, which may be at major, minor or trace concentrations.

In general, industrial incinerator fly ash consists of alkaline metal oxides and various toxic heavy metals. The characteristics of industrial ash are influenced by incineration parameters, such as furnace type, capacity, furnace temperature and waste input. Previous studies on the characteristics of fly ash focused on heavy metal content and leaching behaviour from municipal solid waste incineration (Chang & Wey 2006; Kida et al. 1996).

This study aimed to investigate heavy metal contents in two coal -fired power plants and industrial incinerator fly ash in Malaysia through instrumental neutron activation analysis (INAA), which is a non-destructive and multi-element analytical technique (Bode et al. 1990; Vance & Ehmann 1991) and to measure the specific activity concentrations of naturally occurring radioactive materials (NORM) in coal fly ash through low-level gamma spectrometry.

MATERIALS AND METHODS

SAMPLING AND SAMPLE PREPARATION

The power plants selected for the study were Kapar coal-fired power plant (KPP) which generates 2420 MW of electricity and contributes about 23% of the country's energy at maximum demand. The total annual feed coal consumption is approximately 3.2 to 3.3 m tones; Jana Manjung coal-fired power plant (JMPP) is produces up to 2,100 MW from its three 700 MW units which uses low sulphur and low bitumen coal to minimise pollution; while Kualiti Alam industrial incinerator (KAI) is the first plant for processing scheduled waste in Malaysia and receives all types of hazardous waste except medical and radioactive wastes.

In preparing the fly ashes samples for INAA, fine-powdered, coal-fired and industrial fly ash were dried in an oven set at 60°C for 3 days to remove the moisture content for accurate dry weight measurement and to produce well-homogenised samples (Al-Areqi et al. 2008). Approximately 100 mg of fly ash samples was placed in clean cylindrical polyethylene vials and sealed by heat sealing. Triplicates were prepared for each sample. The standard reference material (SRM) used for Instrumental Neutron Activation Analysis (INAA) was coal fly ash 1633a which was prepared in the same manner of samples.

SAMPLE IRRADIATION, COUNTING AND MEASUREMENT

The comparative method was used to determine many elements simultaneously by irradiating the standard reference material (coal fly ash 1633a) and fly ash samples in the same neutron flux and counting them in the same geometry. The samples, together with the standard reference material (coal fly ash 1633a), were irradiated for 6 h in a rotary rack facility of TRIGA Mark II reactor of the Malaysian Nuclear Agency. The normal operating power of the reactor was 750 kW, and the neutron thermal flux was approximately 10^{12} cm^{-2} s^{-1}. A summary of the irradiation and counting procedure is presented in Table 1.

All measurements for INAA were performed using HPGe detector with a relative efficiency of 30% and a resolution of 1.8 keV (FWHM) at 1.33 MeV. Photo peaks were analysed using Genie-2000 (Canberra, Inc.). The samples were measured at different cooling times ranging from 2 days to 4 weeks and at 6 cm distances from the detector at NAA laboratory, Malaysian Nuclear Agency.

For direct gamma spectrometry (HPGe), all of the samples were dried, homogenised, filled in a 250 mL cylindrical container with a weighted amount (approximately 250 and 220 g), sealed hermetically and stored for a minimum period of 4 weeks prior to counting to re-establish the secular radioactive equilibrium between ^{226}Ra and its short-lived daughter products due to the possible escape of ^{222}Rn during handling. The same procedure was also performed for the reference material, namely, IAEA-375, which was used as a standard in this present work.

TABLE 1. Summary of INAA conditions used in this study

Procedure	Element	Nuclide	Half-life	γ-ray energies (keV)
Irradiation 6 h Cooling 3-5 days Counting 3600-7200s	As	^{76}As	26.24 h	559
	U	^{239}Np	2.35 d	228, 277.6
	Sb	^{122}Sb	2.70 d	564.24
Irradiation 6 h Cooling 20-30 days Counting 3600-7200s	Ba	^{131}Ba	12 d	496
	Th	^{233}Pa	27.00d	312
	Cr	^{51}Cr	27.80 d	320
	Fe	^{59}Fe	45.1 d	1099, 1293
	Sb	^{124}Sb	60.90 d	603,1690.98
	Zn	^{65}Zn	244.00 d	1115.55
	Co	^{60}Co	5.25 y	1173,1332

The specific activities of the natural radionuclides ^{238}U, ^{226}Ra, ^{232}Th and ^{40}K were measured for 24 h through direct high-resolution γ-ray spectrometry. A Canberra HPGe detector with a relative efficiency of 10% and FWHM of 1.9 keV was used for a ^{60}C-ray energy line at 1,332 keV at Nuclear Science Program, Universiti Kebangsaan Malaysia (UKM). The specific activities of ^{238}U, ^{226}Ra and ^{232}Th were determined from the photopeaks of ^{214}Bi (1,764.49 keV), (186.5 keV) and ^{208}Tl (2,614.53 keV), respectively. ^{40}K was directly determined using the photopeak at 1,460.8 keV.

Results and Discussion

Elemental Concentration

The results of heavy metal concentrations in the fly ash samples are presented in Table 2. The concentrations of heavy metals in fly ash from both power plants were largely different from each other because various types of coal and combustion temperatures were used. In general, the concentrations of most elements, such as As, Ba, Cr, Fe, Th and Zn, were relatively higher in the Manjung coal fly ash samples than in the Kapar coal fly ash samples except Co, Sb and U.

The most abundant elements were Fe and Ba in the Kapar and Manjung coal fly ash samples with concentrations of 27,012 and 57,500 mg/kg for Fe and 494 and 1,119 mg/kg for Ba, respectively. The least abundant element was Sb, and its concentrations were 3.67 and 2.23 mg/kg in the Kapar and Manjung coal fly ash samples, respectively. Considerable variations in the concentrations of the elements in the fly ash samples were related to the initial amounts of these elements in feed coal, their boiling points and the techniques used for handling and storage (Ahmaruzzaman 2010; Nawaz 2013).

Table 2 also indicated that the concentrations of Fe, Zn, Ba and Cr in the ash samples from the incinerator were higher than those of other elements. By comparison, the results between the power plant and industrial fly ashes revealed that As, Ba, Cr, Fe, Sb and Zn were higher in concentration in industrial fly ash than those in coal fly ash because of the high content of various toxic heavy metals in industrial wastes. Toxic heavy metals present in the ash of industrial solid waste are derived from a wide range of products used in industrial facilities, such as plastics, alkalis, batteries, dross and paint/ink/dye solvent lubricants.

Our results showed that two radioactive elements, namely, ^{238}U and ^{232}Th, were found in coal fly ash with concentrations of 7.7 ± 0.29 and 27 ± 0.84 mg/kg, respectively, in the Kapar coal fly ash and 20.05 ± 1.26 and 5.87 ± 0.39 mg/kg, respectively, in the Manjung coal

TABLE 2. The averages concentrations of heavy metals in fly ashes (mg/kg)

Element	Locations		
	KPP Fly ash	JMPP Fly ash	KAI Fly ash
Fe	2.71 ± 0.2 (%)	5.75 ± 0.14 (%)	3.1 ± 0.19 (%)
Zn	*ND	148 ± 29	7675 ±448
Ba	494 ± 90	1119 ± 27	2760 ± 173
Cr	53 ± 6.49	61.73 ± 5.37	1029 ± 39
As	17 ± 0.92	23.58 ± 1.55	54 ± 2.60
Co	41 ± 1.6	35 ± 2.4	35.8 ± 0.70
Sb	3.67 ± 0.59	2.3 ± 0.03	255 ± 17
U	7.7 ± 0.29	20.05 ± 1.26	*ND
Th	27 ± 0.84	5.87 ± 0.39	*ND

* ND: Not Detected

fly ash. Thorium in coal is mostly present as phosphate minerals, such as monazite or apatite, whereas uranium is found in the mineral and organic fractions of coal (Papastefanou 2007). The presence of these heavy metals in fly ash not only represent their existence and concentration as components or contaminants but also indicate the levels of contamination associated with fly ash, especially pollutants released from the stack.

The heavy metals concentrations found in this study and the other values reported from other countries are given in Table 3. The values obtained in our study were within the range of fly ash from Italy except U or lower than elements, such as As, Cr, Fe and Th, in Argentinean fly ash. However, the concentrations of As, Co and U in our study were higher than those in Australian fly ash. Table 3 also shows that some heavy elements, such as Ba, Co, Cr, Fe and Zn, present in industrial fly ash are slightly higher than those in industrial waste (IW) fly ash from Malaysia and between global ranges except Ba and Fe, whose concentrations were higher than those in other countries. Because of the generally low concentration of heavy metals in comparison with global coal power plant ashes, it is not expected that significantly undesirable environmental impacts will arise from fly ashes in Kapar and Jana Manjung power plants or industrial fly ash in Kualiti Alam industrial incinerator to the environment.

NATURAL RADIOACTIVITY

The results of ^{238}U, ^{226}Ra, ^{232}Th and ^{40}K in coal power plant fly ash samples are presented in Table 4. The activity concentrations of ^{40}K in coal fly ash were 321.65 Bq/kg, which were higher than those of ^{238}U (152.71 Bq/kg), ^{232}Th (134.41 Bq/kg) and ^{226}Ra (27.42 Bq/kg). Many researchers showed that the activity concentrations of the NORM are higher in fly ash samples than in coal and bottom ash (Papastefanou 2007). The enrichments of ^{238}U and ^{226}Ra in fly ash are high because fly ash particles are small, and the surface-to-volume ratios are high. The concentrations of ^{238}U, ^{226}Ra and ^{40}K were lower than those in other countries except ^{232}Th.

The results obtained from the determination of ^{238}U and ^{232}Th through INAA were consistent with those determined

TABLE 3. The heavy metals content in fly ashes for various studies (mg/kg)

	This work		Fardy et al. (1989) Australia	Orvini & Pirico (1995) Italy	Marrero et al. (2007) Argentina	This work	Khoo (2006) Malaysia	Landsberger et al. (1993)	Orvini & Pirico (1995)
	KPP-Fly ash	JMPP -Fly ash				KAI Fly ash	IW Fly ash	*MSW Fly ash	City Fly ash
As	17	23.58	2.7-8	5 – 70	24.3	54	70	75.3 - 229	60-230
Ba	494	1119	340-730	1000-5000	390	2760	1.03%	<700- 1300	510-1100
Co	41	35	7.3-28	35 - 55	32.8	35.8	30	4.78 - 37.3	10-35
Cr	53	61.73	32-61	110 - 2900	65.6	1029	930	94.1 - 865	650-1500
Fe (%)	2.71	5.75	0.45-10	6 - 10	4.43	3.1	2.11	0.26 - 2.12	0.9-1.21
Sb	3.67	2.295	1.2-6.4	-	1.98	255	640	618- 1665	380-1100
Th	7.7	20.05	14-29	15 - 80	15.1	*ND	<0.5	0.79 - 9.10	2.3-6.6
U	27	5.87	5.2-9.3	11 - 25	5.5	*ND	<3.0	*ND	50-110
Zn	ND	148	45-180	-	5.5	7675	16000	8690- 8200	6000-71000

*ND: Not Detected. *MSW: Municipal Solid Waste

TABLE 4. Activity concentration (Bq/kg) of natural radionuclides in coal fly ash in this work and other studies

K-40	Ra-226	Th-232	U-238	Reference
321.65±12.73	27.42 ± 2.27	134.41 ± 4.09	152.71±8.55	This work KPP coal fly ash
297 (204-382)	366 (142-605)	-	356 (263-950)	Papastefanou (2007)
545 (523-673)	29.5 (14.3-29.5)	-	-	Van Duong et al. (1995)
170-615	59-110	57-130	64-114	Fardy et al. (1989)
635 (471- 968)	2448 (1442- 3773)	64(43- 124)	2300 (1459- 5198)	Flues et al. (2006)
447	1020	59.1	998	Papaefthymiou et al. (2007)

TABLE 5. Concentrations of ^{238}U and ^{232}Th (mg/kg) in KPP coal fly ash determined by INAA and γ- ray spectrometry techniques

Radionuclide	γ- ray spectrometry (mg/kg)	INAA (mg/kg)
Th-232	32.76 ± 1.00	27 ± 0.84
U-238	12.22 ± 0.69	7.7 ± 0.29

through γ-ray spectrometry (Table 5). The differences between two techniques might be due to the radioactive equilibrium or homogeneity problems or gamma ray interference. From this study, it can be suggested that safe regulations should be taken not only for elements emission but also the utilization and disposal of the ashes.

Conclusion

The concentrations of heavy metals, namely, As, Ba, Co, Cr, Fe, Sb, Th, U and Zn, in power plant and industrial incinerator fly ash samples from two power stations and an incinerator were determined by INAA. The highest concentrations of Fe were detected in Kapar (27,012 mg/kg) and Manjung (57,500 mg/kg) coal ash samples. The lowest concentrations of Sb were 3.67 and 2.23 mg/kg for the Kapar and Manjung coal fly ash samples, respectively. Coal or industrial waste (IW) feed type, burning temperature and pollution control devices influenced the elemental concentrations found in fly ash. The results of NORM in coal fly ash showed that the ^{232}Th concentration was higher than that of global fly ash levels, whereas the ^{238}U, ^{226}Ra and ^{40}K concentrations were less than those of global levels. The concentrations of heavy metals in all types of ashes in this study were within the range or at a lower concentration than those in other countries and it can conclude that no significant environmental impacts of fly ashes if managed properly.

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Received: 24 February 2019
Accepted: 24 May 2019