Applied Radiation and Isotopes 90 (2014) 53–57

Contents lists available at ScienceDirect

Applied Radiation and Isotopes

journal homepage: www.elsevier.com/locate/apradiso

Elemental characterization of coal, fly ash, and bottom ash using an energy dispersive X-ray fluorescence technique

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HIGHLIGHTS

- Concentrations of 18 elements were determined in coal and ash samples using EDXRF.
- Mineral quantification up to 95% was carried out for fly and bottom ash samples.
- Enrichment ratios of elements were calculated in combustion residue with respect to coal.
- Enrichment factor with respect to crustal average was estimated for ash samples.

ARTICLE INFO

Article history:
Received 3 December 2013
Received in revised form
4 February 2014
Accepted 1 March 2014
Available online 12 March 2014

Keywords:
Fly ash
Bottom ash
Fusion point
EDXRF
Enrichment ratio

ABSTRACT

A total of 18 elements viz. Si, Al, Fe, Ca, Mg, K, Na, Sr, V, Zn, Mn, Cr, Cu, Pb, Ni, Co, As and Cd were analyzed in coal, fly ash and bottom ash samples collected across India using an EDXRF technique. Various indices such as element enrichment ratio, enrichment factor (with respect to crustal average) and mineral composition were calculated. Around 95% of mass was reconstructed using the concentration of elements in this study for fly and bottom ash.

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1. Introduction

Coal has many important uses worldwide. The most significant uses of coal are in electricity generation, steel production and in cement manufacturing. According to the World Coal Association around 7.6 billion tons of coal was used worldwide which includes 1 billion tons of brown coal. Global coal consumption has grown faster than any other fuel since 2000. The five largest coal users China, USA, India, Russia and Japan account for 76% of total global coal usage. Electricity production from coal sources in India was 68.56% in 2009. As a fuel, coal refers to all coals and brown coal of both primary (including hard coal and lignite-brown coal) and derived (including patent fuel, coke oven coke, gas coke, coke oven gas, and blast furnace gas) origins. Poor-quality coal with high ash yield results in the generation of large amounts of fly and bottom ash of varying properties when used in coal-fired power plants. Indian coal used in power plants generally has high ash yield (35–45%) and is of low quality (Rajamane, 2003; Mathur et al., 2003; Sarkar et al., 2005).

The major composition of fly ash is qualitatively similar to that of natural earthly materials such as soils and shales. Oxidized compounds of Si, Al, Fe and Ca account for nearly 90% of the composition of fly ash. Other elements such as Mg, K, Na, Ti and S occur as minor constituents and account for a small percentage of the bulk composition. Generally, all other elements occur in the parts per million ranges and, collectively, seldom exceed 1% of the bulk composition. Fly ash is associated with various useful constituents such as Ca, Mg, Mn, Fe, Cu, Zn, B, S and P, along with appreciable amounts of toxic elements such as Cr, Pb, Hg, Ni, V, As and Ba. The concentration of trace elements in ash is extremely variable and depends on the type and composition of the parent fuel, conditions during combustion and efficiency of emission control devices (Dogana and Kobya, 2006). Fly ash also contains radioactive elements which may come in contact with the general public when they are dispersed in air and water or are included in commercial products that contain fly ash (Pandit et al., 2011).

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http://dx.doi.org/10.1016/j.apradiso.2014.03.002
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Fly ash has gained considerable attention from the construction industry as a useful and increasingly important raw material. Once considered as a nuisance waste product with a disposal problem, fly ash is now recognized as a valuable substance which confers certain desirable characteristics in its many applications viz. cement manufacture, ceramics making and wastewater treatment (Diamadopoulos et al., 1993; Panday et al., 1985). The quality of fly ash and bottom ash is determined by the chemical makeup of coal and combustion parameters. So it is important to quantify the elemental composition of coal along with bottom and fly ash, so partitioning of elements can be determined. There are various analytic techniques (ICP-MS/AES, AAS, NAA, DPASV, etc.) capable to characterize the coal, fly ash and bottom ash with their own advantages like lower detection limit, sensitivity, large dynamic ranges, etc. Among simultaneous multi-elements analysis techniques, X-ray fluorescence (XRF) instrumentation has lower capital cost and is cheaper to use than Neutron Activation Analysis (NAA), is faster than ICP-AES and is widely available. XRF techniques require less sample preparation time, cost and also avoid use of hazardous chemicals. In recent years X-ray fluorescence analysis has emerged as a very powerful technique for elemental analysis of environmental samples (Vijayan et al., 1997; Cakir et al., 2003; Budak et al., 2006; Gupta et al., 2013). In view of the potential of fly ash as a useful industrial raw material and a health hazard, as a source for recovery of radioactive and valuable elements, studies of its elemental composition are highly desirable.

In the present study coal, fly ash and bottom ash samples were collected from various coal-fired power plants situated across India. Major (Na, K, Si, Al, Ca, and Mg), heavy and toxic (Zn, V, Cr, Mn, Fe, Co, Ni, Cu, As, Pb, Sr, and Cd) elements in coal, fly ash and bottom ash were analyzed using EDXRF techniques. Voltage and current of X-ray tube and the application of secondary targets were optimized for the analysis of elements of different atomic numbers.

2. Methodology

2.1. Sample collection and processing

The feed coal, fly ash and bottom ash samples were collected from eight coal-fired thermal power plants of India. Most of the power stations under study used Indian coal as the feed material; detail of coal type and their grading is described elsewhere (Sahu et al., 2009). A list of thermal power plants, their capacity and geographical coordinates is presented in Table 1. The samples were collected weekly over a period of one month to obtain true representative samples. The coal samples were obtained in duplicates from samplers located at the coal feeder of the boilers. The samples were further homogenized to obtain a gross sample. Bottom ash samples were collected from the asher of the boiler and fly ash were collected from the hoppers of the electrostatic precipitators. The gross samples were milled and split carefully in accordance with ISO recommendations to obtain a representative subsample of particle size 230 mesh size for further chemical analyses (Bhangare et al., 2011). Each fine sieved (< 63 μm) coal, fly ash and bottom ash samples were homogeneously mixed with cellulose powder (Sigma-Aldrich) in a ratio of 1:1 and pelletized with a hydraulic pressure of 25 tones/cm^2 for three minutes.

2.2. EDXRF set-up

All measurements were carried out under vacuum, using a Xenemetrix EX-6600 EDXRF spectrometer. The instrument consists of an X-ray tube with a Rh anode as the source of X-rays with HVPS 60 kV, 6.6 mA power supply, a LN$_2$ cooled Si(Li) detector with a resolution of 131 eV at, Mn Kα (5.9 keV) X-ray and a 8-sample turret that enables mounting and analyzing 8 samples at a time. Different secondary targets were used to excite the elements in the sample (Singh et al., 2011).The concentrations of 18 elements, namely Na, K, Si, Al, Ca, Zn, V, Cr, Mn, Fe, Co, Ni, Cu, As, Pb, Sr, Mg and Cd were measured. The built-in software was used for the quantitative analysis. To optimize the EDXRF sensitivities for the wide range of elements of interest, three different combinations of EDXRF parameters (including voltage and current) shown in Table 2 are employed for different elements for coal and ash samples. The secondary targets were used for particular line energies to reduce the relevant background intensities. A screenshot of EDXRF spectra produce by bottom ash using Zr as a secondary target is shown in Fig. 1.

2.3. Quality control and quality assurance

Individual and mixture of elements coated on nucleopore aerosol membrane with known deposition per unit area (μg/cm^2) were used for calibration of EDXRF. IAEA-405 and IAEA-433 reference materials were used for to verify calibration. Concentration of elements of interest analyzed in both RMs has shown good agreement with published reference sheet values. Pb and As are

Table 2
Combinations of the parameters and X-ray filters used in the EDXRF setup to find the different elements.

Elements	Voltage (kV)	Current (μA)	Secondary target	Preset time (s)	Range (keV)	Atmosphere
Na, Mg, Al, Si, K, and Ca	27	4800	Ti	800	0–10	Vacuum
V, Cr, Mn, Fe, Co, Ni, Cu, and Zn	40	4000	Ge	300	0–40	Vacuum
As, Sr, Cd, and Pb	50	4800	Zr	500	0–40	Vacuum

Table 1
List of thermal power plants under study with their capacity and geographical locations.

Name of the thermal power plant	Capacity (MW)	Geographical coordinates
Chandrapur Thermal Power Station (Maharashtra)	2340	20°00′24″N 79°17′21″E
Koradi Thermal Power Station (Maharashtra)	620	21°14′52″N 79°05′53″E
Dahanu Thermal Power Station(Maharashtra)	500	19°57′12″N 72°44′54″E
Talcher Super Thermal Power Station (Orissa)	3000	21°05′49″N 85°04′30″E
Jindal Mega Power Plant Tamnar (Chhattisgarh)	1000	22°06′16″N 83°27′04″E
Simhadri Super Thermal Power Plant (Andhra Pradesh)	2000	17°35′42″N 83°05′18″E
Manguru Thermal Power Station (Andhra Pradesh)	90	17°56′14″N 80°49′07″E
Vindhyachal Thermal Power Station (Madhya Pradesh)	3760	24°05′53″N 82°40′18″E

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