

Elemental characterization of coal fly ash using k_0 -based IM-NAA **and EDXRF towards its potential utilization and environmental concern**

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Abstract

India is a major producer of fy ash due to its coal-based thermal power plants. The elemental characterization of the fy ash facilitates its better utilization and safe disposal, which was accomplished using k_0 -based internal mono standard neutron activation analysis and X-ray fuorescence techniques in the present study. The fy ash was mainly composed of useful elements such as Si, Al, Fe, K and Ca. The same was also found to contain the rare earth elements from minor to trace levels in the order of Ce>La>Nd>Sm>Yb>Eu>Tb>Lu and the heavy and toxic elements such as U, Th, As, Pb etc. in minor and trace level.

Keywords Coal fly ash $\cdot k_0$ IM-NAA \cdot EDXRF \cdot In-situ relative detection efficiency \cdot KAMINI reactor

Introduction

Coal is one of the major non-renewable resources that are being used globally for the production of electricity in thermal power plants. Over 39% of the world's electricity is produced through thermal power plants, whereas it is about 75% in India [\[1](#page-7-0)]. Coal fy ash (CFA) is the major industrial by-product that is produced as waste in large quantities from the burning of pulverized coal. The coal that is produced in India is of low grade with the ash content in the range of 30–45%, where as it is 10–15% in the case of imported coal [\[2](#page-7-1)]. Due to the usage of low grade quality of coal, large quantity of fy ash is being generated at the coal based thermal power plants. Hence, its disposal becomes a great liability as it requires a large area for the same [[3\]](#page-7-2). In addition, CFA is also considered to be a major global pollutant due to its surplus amount of generation with hazardous nature [\[4](#page-7-3)].

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As the requirement of electricity keeps on increasing with population and the utilization of the coal and subsequently production of fy ash continuously increased from 1996 to 2019. With the advancement of the techniques and various government policies, Central Electricity Authority (CEA) of India has made the mandatory utilization of CFA towards various applications that would bring down the requirement of the land for the disposal as well as to address the problem of pollutants associated with it. The World Bank reported that India needed as large as 1000 square km area of land to dispose fy ash generated until 2015 [\[5](#page-7-4)]. As per the latest reports by CEA, the maximum utilization of fy ash during the frst half of the year 2018–2019 was carried in cement sector (-27%) , reclamation of low laying area (9.7%) , ash dyke raising (9.2%), bricks and tiles (8.7%) etc. Thus total utilization was accounting nearly 70% of the total fy ash produced, whereas rest of the 30% of fy ash is still unutilized and needs safe disposal because of its toxicity owing to the presence of elements such as U, Th, Cr, Pb, As, Ba etc. $[6-9]$ $[6-9]$.

However, fy ash, at the same time, is a potential alternative source for the recovery of various valuable constituents such as Al, Si, Ca, Mg, Mn, Fe, Cu, Zn and rare earth elements (REEs) [[10,](#page-8-2) [11\]](#page-8-3). Based on their mineralogical and elemental properties, it can be used for the recovery of minerals such as alumina and silica which are being used as the initial raw materials for the preparation of synthetic zeolites.

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As the supply of REEs is getting more and more challenging, fy ash plays a major role as an alternate resource material. Therefore, elemental characterization of fy ash becomes an important task towards their appropriate safe disposal or efective utilization towards the extraction of valuable elements.

For the determination of elemental concentration in solid matrix samples, the conventional analytical techniques such as atomic absorption spectrometry (AAS), inductively coupled plasma optical emission spectrometry (ICP-OES) and inductively coupled plasma-mass spectrometry (ICP-MS) have been used widely, which invariably necessitates pre and post-treatments of the sample for the estimation of its elemental composition [\[4,](#page-7-3) [12](#page-8-4)]. The chemical loss and cross contamination may necessitate their pre and post-treatments that limits the applicability of these methods towards the analysis of solid samples. In contrast, non-destructive assay techniques like neutron activation analysis (NAA) [[13](#page-8-5)], energy dispersive X-ray forescence (EDXRF), particleinduced X-ray emission spectrometry (PIXE) etc. can be attractive alternate analytical tools for establishing the elemental composition of such samples [\[2,](#page-7-1) [14–](#page-8-6)[16\]](#page-8-7).

Among the various standardization methods of NAA [\[17,](#page-8-8) [18](#page-8-9)], the k_0 -based IM-NAA became an attractive technique in the recent past, for the elemental analysis of small to large samples with irregular geometry in various felds. In this method, one of the elements present in the sample itself is used as monostandard and the gamma rays of its activation product were used for the in-situ relative detection efficiency $[19-21]$ $[19-21]$. The neutron irradiation studies were carried at KAlapkkam MINI (KAMINI) reactor, operating with its full power 30 kW thermal energy using 233 U-Al fuel and located at Indira Gandhi Centre for Atomic Research (IGCAR), India [\[22\]](#page-8-12). In EDXRF technique, the elemental concentrations were obtained using comparator method for which the various in-house prepared standards were utilized for the calibration to arrive the absolute concentrations of elements [[23\]](#page-8-13).

The present work describes the quality assurance of k_0 -based IM-NAA method. Elemental characterization of major, minor and trace elements present in the CFA samples received from Interscience Institute of Management & Technology (IIMT), Bhubaneswar, India has been accomplished using the novel approach of k_0 -based IM-NAA and EDXRF techniques.

The radiometric assay of the irradiated samples was accomplished by high resolution gamma spectrometry using a 30%

Experimental

Materials

co-axial High Purity Germanium (HPGe) detector procured from M/s Baltic Scientifc Instruments, Latvia. The detector has a resolution of 1.85 keV at 1332 keV of 60 Co along with an associated 8 k multichannel analyzer (MCA) system of Aptec spectra software. Energy calibration of the detector was established using the standard sources of 241 Am, 137 Cs, $133Ba$ and $152Eu$, which were procured from M/s Amersham, Inc. High resolution bench top EDXRF system, model EX-2600 Genius IF SDD was procured from M/s Xenemetrix, Israel.

Elemental characterization

About 100–200 mg of Coal fy ash samples and a NIST standard reference material (coal fly ash—1633b) were packed in a clean polyethylene sheets and irradiated at pneumatic fast transfer system (PFTS) position of KAMINI reactor at 20 kW power for 6 h. After sufficient cooling $(-15 h)$, all the samples were assayed by HPGe detector at a sample distance of 15 cm in reproducible geometry. A typical gamma spectrum obtained for the irradiated coal fy ash sample is shown in Fig. [1.](#page-2-0)

In the EDXRF analysis, the fy ash samples were thoroughly ground in a mortar and pestle and mixed with known amount of boric acid. The mixtures were further ground and made pellets having a diameter of 25 mm and a thickness of 2 mm by applying a pressure of 2 tons using a hydraulic press. Similarly, preparation of standards for Si, Al, Ca, Fe and Pb were also carried out by mixing known amounts of these compounds with boric acid followed by palletization. The determination of Al, Si, Ca, Fe and Pb in the samples was carried out using a low power Rh-target X-ray tube. The detector with a resolution of 135 ± 10 eV at 5.9 keV of Mn K X-ray equipped with a Be entrance window was used. The intensities of fuorescent X-rays were measured using a PC-based 2 k channel analyzer with nEXt software. The instrument is equipped with 50 W, 50 kV forced- air cooled end window, front anode X-ray tube. Energy calibration of the EDXRF system was carried out using Al, Cu, Mo and Sn foils. A typical EDXRF spectrum of the fy ash sample is presented in Fig. [2](#page-2-1).

Calculation

The ratio of mass (*m*) of an element (*x*) to mass of the internal mono standard element (*y*) in the fy ash was calculated by the following equation in IM-NAA.

$$
\frac{m_x}{m_y} = \frac{(\text{(SDC)}(f + Q_0(\alpha)))_y}{\left((\text{SDC})(f + Q_0(\alpha))\right)_x} \cdot \frac{\text{PA}_x}{\text{PA}_y} \cdot \frac{(\epsilon_y)_y}{(\epsilon_y)_x} \cdot \frac{k_{0,\text{Au}}(y)}{k_{0,\text{Au}}(x)}
$$
(1)

Fig. 1 Gamma spectrum of the irradiated coal fy ash sample

 24 Na

 $\frac{140}{42}$ _{La}

 $\overline{42}K$

3.0

Fig. 2 EDXRF Spectrum of coal fy ash sample

where *S*, *D* and *C* represent saturation, decay correction and counting correction factors respectively, PA is the net peak area, $k_{0,\text{Au}}$ is the literature reported $k_{0,\text{Au}}$ -factors [\[24](#page-8-14), [25](#page-8-15)], ε_{γ} is the detection efficiency, f is the thermal to epicadmium neutron flux ratio and α is neutron flux shape factor. The in-situ relative detection efficiency was obtained using the following equation

$$
f(x_i) = \ln(\varepsilon) = \sum_{i=0}^{n} A_i [\ln(E)]^i
$$
 (2)

where E is the gamma ray energy, A_i is the coefficient of the polynomial and *i* is the order of polynomial. $Q_0(\alpha)$ is the ratio of the resonance integral (I_0) to thermal neutron cross section (σ_0) corrected for α using the following equation.

$$
Q_0(\alpha) = \frac{Q_0 - 0.429}{\overline{E_r^{\alpha}}} + \frac{0.429}{(2\alpha + 1)E_{\text{cd}}^{\alpha}}
$$
(3)

where E_r is the effective resonance energy (eV) and E_{cd} is cadmium cut-off energy (0.55 eV) .

f **and α value of PFTS KAMINI**

The f and α value of PFTS position of KAMINI reactor were established to be 27.8 ± 0.34 and $-4.5 \times 10^{-2} \pm 1.8 \times 10^{-3}$ respectively [\[26](#page-8-16), [27](#page-8-17)]. *f* value indicates that it contains more than 99.3% of thermal neutron component and negative sign of the α values indicates that the neutron spectra at this position was relatively hard compared to the ideal epithermal neutron fux distribution.

In-situ relative detection efficiency: A geometrical independent approach in NAA

The absolute efficiency of the detector is the ratio of detected to emitted gamma rays, which varies with the energy of the gamma rays and source–detector geometry. The absolute efficiency of the detector in the large range can be calculated by using the multi energy gamma-ray of known radioactive sources. The in-situ relative detection efficiency is independent of the source–detector geometry. The gamma rays from activation products are being used for efficiency calibration instead of using the external gamma ray standard. The efficiency ratio of two gamma rays of energies E_1 and E_2 can be calculated by following equation

$$
R_{\rm ee} = \frac{\varepsilon_1}{\varepsilon_2} = \left(\frac{\text{cps}_1}{\text{cps}_2}\right) * \left(\frac{I_2}{I_1}\right) \tag{4}
$$

where cps is counts per second and *I* is gamma intensity.

The gamma energy of the activation products, 152m Eu, 140 La and 24 Na in the coal fly ash sample was used for the in-situ relative detection efficiency. The full energy peak efficiency for the HPGe detector was obtained from 0.122 to 2.754 MeV as shown in Fig. [3.](#page-3-0) The gamma energy with high intensity of the above activation products alone were used for the efficiency calibration, based on the energy of interest needed for the analysis (Table [1](#page-3-1)).

The experimentally measured efficiency ratios (R_{ee}) were obtained by Eq. [4](#page-3-2) in which the ε_1 is the efficiency

Fig. 3 In-situ relative detection efficiency curvefor fly ash sample

Table 1 Gamma energy and intensity of the activation products for in-situ relative detection efficiency calibration

Activation product	Gamma energy (MeV)	Gamma intensity (%)
$^{152\mathrm{m}}\mathrm{Eu}$	0.122	7.0
	0.842	14.2
	0.963	11.7
140 La	0.329	20.8
	0.487	46.1
	0.816	23.7
	1.596	95.4
$^{24} \rm Na$	1.369	99.99
	2.754	99.86

corresponding to the lowest energy of each activation product $[28]$ $[28]$. The equation for full energy peak efficiency calibration was obtained by Eq. (2) (2) .

The fitted efficiency ratios (R_{fe}) were obtained by using the Eq. [\(5\)](#page-3-4) for the same energy as that of the experimental ratios.

$$
R_{\text{fe}} = \left(\frac{\varepsilon_1}{\varepsilon_2}\right) = \exp\left[A_1\left[\ln(E_1) - \ln(E_2)\right]\right] + A_2\left[\left(\ln E_1\right)^2 - \left(\ln E_2\right)^2\right] + \dots + A_n\left[\left(\ln E_1\right)^n - \left(\ln E_2\right)^n\right]\right] (5)
$$

The coefficients $A_1, A_2, A_3, \ldots, A_n$ were obtained by minimizing root mean square deviation (RMSD) value for N number of observations.

$$
RMSD = \sqrt{\left(\frac{1}{N}\right)\left(\sum (R_{ee} - R_{fe})^2\right)}
$$
(6)

Fig. 4 Percentage variation between experimental (R_{ee}) to fitted efficiency ratios (R_f)

The fitted efficiency ratios were obtained using $i = 2, 3, 4, 5$ in the empirical Eq. [2](#page-3-3) and calculated the minimum RMSD value for each.

The variation between experimental to fitted ratios were calculated in each case, which decreases from 10% to less than 1% as the number of coefficients increases from 2 to 5 as shown in Fig. [4](#page-4-0).

Elemental characterization by EDXRF

Molybdenum as the secondary target was used to excite Ca, Fe and Pb in the fy ash samples whereas Al and Si were excited without any flter. The secondary targets were used for particular X-ray energies to minimize the relevant background intensities. The concentrations of Al, Si, Ca, Fe and Pb were calculated by relative method of analysis using the standards. The in-house prepared standards with Al, Si, Ca, Fe and Pb were used to establish the calibration curves by plotting the concentrations with respect to their corresponding peak area as shown in Fig. [5.](#page-4-1) The determination of Al, Si, Ca and Fe in the samples was carried out with their characteristic K X-rays of 1.49, 1.74, 3.69 and 6.4 keV respectively whereas L X-rays 10.55 keV was used for the quantifcation of Pb.

Results and discussion

Quality assurance of *k***0‑based IM‑NAA**

 k_0 -based IM-NAA provides relative concentration of an element with respect to the monostandard by using the in-situ relative detection efficiency, neutron flux parameters such

Fig. 5 Calibration plots for Al and Si in EDXRF

as f and α . The absolute elemental concentration can be obtained from these ratios by the knowledge of the concentration of the monostandard or by mass balance. The internal monostandard nullifes the efect of neutron self-shielding across the sample during the irradiation as the fux will be the same for both monostandard and the element of interest. In-situ relative detection efficiency provides correction for gamma ray self attenuation during counting which makes this method geometrically independent [[29](#page-8-19), [30](#page-8-20)].

In-situ relative detection efficiency, f and α values strongly affect the validity of k_0 -based IM-NAA method. The in-situ relative detection efficiency was measured from 0.282 MeV to 2.754 MeV using gamma rays of 175 Yb, 140 La, ²⁴Na, ⁶⁰Co and ⁴⁶Sc for the irradiated NIST 1633b coal fly ash. The elemental concentration ratios were determined using scandium as monostandard and absolute concentrations were obtained using NIST certifed value of Sc. The reliability between the experimental and certifcate value for all the elements were tested by calculating ξ score defned by the following equation

$$
\xi - \text{score} = \frac{x_{\text{ref}} - x_{\text{expt}}}{\sqrt{s^2 + \sigma^2}} \tag{7}
$$

where x_{ref} and x_{expt} are certificate and experimental elemental concentrations respectively. σ is the uncertainty in certifcate values at 95% confdence level. The uncertainties in experimental values (s) presented in Table [2](#page-5-0) are the standard deviation obtained from replicate experiments. The ξ-score values at 95% confdence level for most of the elements in NIST 1633b CFA were within ± 1 as shown in Fig. [6](#page-5-1) and the negative bias in the ξ-score value may be due to the statistical variation during counting of the sample and reference material by gamma spectrometry. The percentage deviation was within $\pm 5\%$ except Na (9.4%), which assured the quality **Table 2** Comparison of the elemental concentration (mg kg−1) obtained by IM-NAA with the certifcate of NIST SRM 1633b coal fy ash

*Internal monostandard

Fig. 6 Zeta (ξ)–scores for various elements present in NIST 1633b coal fly ash

of the k_0 -based IM-NAA method. Similar methodology followed for analyzing the fly ash samples.

Application of *k***0‑based IM‑NAA**

The elemental concentration ratios of 26 elements were obtained in the fy-ash samples with respect to iron which was chosen as an internal monostandard because of its abundance in fy ash and sensitivity towards the NAA. The absolute concentrations of 5 elements such as Al, Si, Ca, Fe and Pb were estimated by using EDXRF technique as shown in the Table [3.](#page-5-2) These elements except Fe are either less sensitive or short-lived towards their estimation by NAA and hence could not be analyzed by NAA method. However, Fe was estimated by EDXRF and compared with its concentration obtained by relative NAA method, which shows good agreement and used to arrive the absolute concentrations

Table 3 Measured elemental concentration (%) in coal fy ash samples by EDXRF

Elements	Concentration $(\%)$ in the sample			
	$CFA-1$	$CFA-2$	World coal fly ash	
Al	$18.2 + 0.9$	$18.3 + 0.8$	$9.3 - 18.8$	
Ca	$0.34 + 0.11$	$0.32 + 0.09$	$0.4 - 16.5$	
Fe	$3.11 + 0.18$	$4.37 + 0.21$	$1.8 - 11.2$	
$Pb*$	$49.9 + 1.5$	29.8 ± 1.2	$35 - 1075$	
Si	$26.1 + 1.1$	$25.6 + 1.1$	13.3–27.8	

* Concentration in mg kg−1

of the other elements for which concentration ratios were measured with respect to Fe using IM-NAA. The gamma rays of activation products viz. 24 Na, 140 La and 152 mEu were used for the determination of the in-situ relative detection efficiency. Further, the efficiency curve was fitted using Eq. 2 with 5th order polynomial as it corresponds to the minimum RMSD as well as deviation from the experimental values. The typical count rate observed for the activation products in coal fy ash samples were in the range of 0.02 to 8 cps with the associated uncertainty of 0.8 to 5%. The elemental concentration obtained by NAA and EDXRF are compared with the concentration ranges in coal fy ash reported elsewhere [[31\]](#page-8-21) for different countries/regions in the world (Tables [3](#page-5-2)) and [4\)](#page-6-0). The concentrations for most of the elements fall within the range already reported in the literatures [[31\]](#page-8-21) and the concentrations of toxic elements in Indian coal fy ash like As, Pb, Th, U etc., are however of serious environmental concern which were found to be close to the minimum reported values [[31](#page-8-21)].

The composition of fy ash towards the minor and trace elements are extremely variable and depends on the composition of the parent coal. From Tables [3](#page-5-2) and [4](#page-6-0), it is observed that the fy ash samples constituted alkali, alkali

earth, transition as well as rare earths with elemental concentrations varying from mg kg⁻¹ to % levels. Both the samples contain approximately the same concentration of the potentially applicable elements such as Fe, Zn, Na, K, REEs etc. and hazardous elements like U, Th, Cr, As and Pb.

The fly ash samples were mainly composed of Si (-26%) , Al (~18%), Fe (~4%), K (~0.8%) and Ca (~0.3%). These major elements exist in fy ash as oxides and they are considered as cost efective raw materials for the ceramic industry. As these powders are very fne in nature, they can be easily incorporated into the ceramic pastes without further treatment. Fly ash can be used either as a raw material or as an additive in the cement industry, which can be categorized in either cementitious or pozzolanic, based on its CaO content.

The transition metals such as Sc, Mn, Fe, Co, Zn, W etc. are distributed in ash from minor to trace level except Fe (-4%) and is widely used as catalysis agents in various industrial applications. The useful nutrients such as Na, K, Ca, Mn and Zn etc. vary from mg kg⁻¹ to % which would be very useful for the applications in environmental studies like plant growth, improving the soil texture, aeration, percolation and water retention in the treated zone.

The value of fy ash is multiplied because of the presence of the economically valuable constituents i.e. REEs. The present study shows that the concentration of REEs

Fig. 7 Variation of Rare Earth Elements (REEs) in studied coal fy ash samples

Fig. 8 Variation of heavy and toxic elements in studied coal fy ash samples

varies from minor to trace level in the order of $Ce > L$ $a > Nd > Sm > Yb > Eu > Tb > Lu$ with light REEs being dominant as shown in Fig. [7.](#page-6-1) The consumption of REEs is growing in a wide range of commercial and defence applications, and also used widely in various felds such as batteries, catalysts, permanent magnets, metallurgy, energy-saving fluorescent tubes, superconductors and medicines. Thus, utilization of fy ash by extracting these REEs could provide a cost-efective and environmentally friendly method for recycling this waste.

The heavy and toxic elements such as U, Th, As, W, Zn, Ta, Hf, Ba and Pb were also found in minor and trace level which are of serious environmental concern. The distribution of these elements is presented in Fig. [8.](#page-7-5) The presence of U and Th necessitates corrections in concentration of some of elements like La, Ce, Nd, Sm, Eu etc. present in fly ash. This is due to production of fission product which thus interferes with activation product $(^{140}La, ^{141}Ce,$ 147 Nd etc.) of the chemical elements and hence causes over estimation in the analysis. The required corrections were introduced for these elements by estimating the amount of each isotope produced per unit mass of standard uranium and thorium [[11](#page-8-3)].

The presence of valuable and toxic elements makes these fy ash samples potentially useful as well as hazardous thus leading to either extraction of these useful elements or demanding an appropriate disposal protocol. Recycling coal fy ash can be a good alternative to disposal and could achieve signifcant economic and environmental benefts as well. Thus the present study established the potential of k_0 based IM-NAA method for analyzing samples non-destructively.

Conclusion

Elemental concentration of 30 elements including major, minor and trace was established non-destructively by IM-NAA and EDXRF. For the first time, k_0 -based IM-NAA method was applied for the analysis of fy ash samples. Most of the elements with good sensitivity and appropriate half life were calculated by NAA except Si, Pb, Ca and Al. Fly ash samples were found to be mainly composed of silicon, aluminium, iron, potassium and calcium. Utilization of fy ash in cement minimizes the emission of $CO₂$ problem to the extent of its proportion in cement. It can be a valuable resource for potentially applicable REEs and nutrients for the environmental applications. The availability of REEs is very scarce, thus fy ash plays a major role as an alternate resource material. The heavy and toxic elements such as U, Th, As, W, Zn, Ta, Hf, Ba and Pb were found to be in minor and trace levels. Use of coal for power generation results into an increased quantum of fy ash production, which has reached about 100 million tonnes per year. All out efforts are needed to utilize this fy ash not only from environmental considerations, but also to avoid land usage for fy ash dumping. Recycling coal fy ash can be a good alternative to disposal, and could achieve signifcant economic and environmental benefts as well. Further studies would be carried out on the extraction of the valuable elements from the fy ash and also owing to its toxic nature, mobility and leaching studies would be also carried out to investigate the probability of heavy and hazardous elements into the environment.

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