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A Case Study for Assessing Coal Flyash in NTPC Power Stations as a Potential Source of Rare Earth Elements

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Abbrevia ions

CCR – Coal combustion residues
CFA – Coal Fly Ash
CREE - critical rare earth elements
EPA – Environmental Protection Agency
EREE - excessive rare earth elements critical
FETV - Fluorination Assisted Electrothermal Vaporization
FGD – Flue gas desulphurization
HREE - heavy rare earth elements
ICP-MS – Inductively coupled plasma mass spectrometry
KED – Kinetic Energy Discrimination
LREE - light rare earth elements
MREE - medium rare earth elements
REEs – Rare Earth Elements
TPPs – Thermal Power Plants
TREE – total rare earth elements
USGS - U.S. Geological Survey
UREE - uncritical rare earth elements

ABSTRACT

Thermal power plants are generating huge quantities of coal fly ash and managing the surplus fly ash has become a global environmental challenge. Coal fly ash is regarded as a promising source of various elements, including rare earth elements (REEs), which are vital components in clean technologies and alternative green energy systems. Due to this widespread usage of REEs, their supply becomes more strained, and finding alternative sources for the production of REEs is increasingly important. This study aimed to measure the concentration of REEs in five different Indian coal fly ash samples and to evaluate their potential for REEs extraction. The concentration of REEs was assessed through inductively coupled plasma mass spectrometry (ICP-MS). Results reveal that cerium (Ce) was the most abundant REEs, with concentrations ranging from 142 to 325 µg/g, while thulium (Tm) and lutetium (Lu) were the least abundant, with concentrations between 2 and 3 µg/g. The TREE content in the analyzed samples varied from 367 to 834 µg/g. Majority of the coal fly ash samples had a critical REEs content of at least 31%, and an outlook coefficient of 0.7 or higher, suggesting that they could be viable for REEs extraction. Our research findings underscore the potential of integrating REEs recovery from coal fly ash. The data presented holds significant value for policymakers and industry stakeholders, offering insights that can illuminate the development of more sustainable and efficacious strategies for coal fly ash management.

Keywords: **Thermal power plants, Coal fly ash, Cerium, ICP-MS, Rare earth elements**

1. INTRODUCTION

India relies significantly on thermal power plants (TPPs) for electricity generation. These plants utilize heat from various fuel sources to produce electricity (Singh et al. 2013). The different types of TPPs in India are (a) Coal-fired (b) Gas-based (c) Liquid fuel (Diesel). India holds the position of the third largest reserves of coal globally, surpassed only by the United States (U.S.) and China. It is also the third largest coal producer globally, with an annual production of around 310 million tonnes and imports nearly 25 million tonnes (Del Rio et al. 2022). Coal plays a pivotal role in contributing to one-third of the nation's comprehensive energy supply, accounting for 55% of the total energy requirements. It continues to be the foremost source of energy for the generation of electricity, with around 75% of the country's electricity being produced through coal-fired or TPPs (Gulagi et al. 2022; Zhao et al. 2022).

During the process of coal combustion, an array of by-products is formed, including boiler slag, fly ash, bottom ash, and residues from flue gas desulfurization (FGD). Indian coal typically contains 30-50% ash content, resulting in significant volumes of coal fly ash (CFA) generated during electricity production (Koech 2015). In the first six months of the 2021-2022 period, approximately 0.13 million kilograms of CFA was produced, with projections that this figure will exceed 1 million kilograms by 2031-32. Due to this huge production of fly ash and its negative environmental impact government introduced stringent guidelines for the complete utilization of CFA (Al-Ghouti et al. 2021). On the other hand, heavy metals and rare earth elements (REEs) leaching from CFA can contaminate soil and water, disrupting aquatic ecosystems and affecting aquatic organisms. Furthermore, REEs can accumulate in plants, animals, and soil, leading to health risks for humans and wildlife. Managing landfills that contain CFA with REEs requires careful consideration of long-term stability, containment, and potential leaching (Spitz & Trudinger 2019). Considering the above serious environmental issues associated with it, there is an imperative requirement to utilize CFA. This strategy can alleviate pollution, preserve natural resources, and lessen the overall environmental impact (Sanchez-Garcia & Hernandez-Sanchez 2021).

Researchers are investigating the possibility of using CFA as a resource for REEs extraction and recovery. Recycling and reusing REEs can reduce the need for mining and minimize environmental impact. By extracting REEs in CFA, we can ensure a balance between resource utilization, risk mitigation, and sustainable management practices (Twiss et al. 2015). REEs also play a crucial role in various aspects of modern society, including the advancement of clean energy technologies. The advancement of sustainable energy technologies, such as wind turbines, electric vehicles, and power-efficient lighting, is heavily reliant on REEs. They improve the functionality of batteries, catalysts, and magnets, which makes them valuable both strategically and economically on a worldwide basis (De Lima & Filho 2015). REEs can be efficiently extracted and recovered from CFA using various methods such as acid leaching, bioleaching, membrane-based separation, oxalate precipitation, and zeolite synthesis (Tkaczyk et al. 2018). These methods are effective, environmentally friendly, and cost-efficient, and contribute to sustainable resource utilization Primary REEs mining can also be reduced by adopting these methods to extract REEs from CFA. In summary, extracting REEs in CFA offers a promising avenue for sustainable resource utilization while addressing global energy and environmental challenges (Stopic & Friedrich 2021).

Recent findings on CFA from western Canada have revealed that Ca-rich CFA have the highest REEs concentration, with nearly 100% recovery using acid leaching technique. Si-rich samples, on the other hand, had lower REEs recovery rates (3-65%) in acid leaches (Swaine 2013). Geochemical data showed that REEs correlated with Al, Si, Ti, Th, and Zr, likely associated with detrital and clay minerals (Bayon et al. 2015; Moldoveanu & Papangelakis 2015). Various extraction methods such as physical separation, hydrometallurgical, and bio-hydrometallurgical techniques are available for extraction of REEs. Researchers have explored membrane-based and electrodeposition-based separation methods for REEs recovery from coal combustion residues (CCR) leachates. However, large-scale REEs extraction faces environmental challenges such as proper waste management, energy consumption, and regulatory compliance. Despite this, ongoing research and successful case studies demonstrate the feasibility of large-scale REEs recovery from CCR, contributing to sustainable resource utilization.

For the determination of REEs, in this study, inductively coupled plasma mass spectrometry (ICP-MS) has been selected among various analytical techniques. This choice is based on the precision, sensitivity, and capability of detecting lower limits offered by ICP-MS, rendering it an optimal technique for identifying the elemental composition of CFA. Two methods related to determining REEs in CFA are as follows: (a) Tetrafluoro boric Acid Digestion: This method eliminates the need for complexing fluoride ions (F^-) with boric acid (H_3BO_3) and provides accurate REEs determination using tetrafluoro boric acid (HBF_4) followed by ICP-MS analysis. This streamlined digestion technique achieved excellent recovery rates. (b) Fluorination Assisted Electrothermal Vaporization (FETV)-ICP-MS: This method involves FETV-ICP-MS and allows for efficient analysis of REEs without extensive sample preparation.

In the present investigation, fly ash samples were collected from five different TPPs of NTPC which are geographically distributed in different locations across India. The present study focuses on the identification of the REEs composition of CFA, which is crucial for understanding their environmental impact and potential for extraction of REEs. Inspite of the huge quantity of CFA generated, there is limited database on the composition of REEs in Indian CFA. A strong data base is required to understand the potential of CFA for commercial extraction of REEs. Addressing this gap, the current study aims to characterize REEs in CFA from various TPPs and evaluate their potential as secondary sources for REEs recovery. The findings will help in identifying suitable CFA for REEs extraction and also will enhance the utilization of hazardous CFA.

2. MATERIAL AND METHODS

2.1 Materials and Reagents

Fly ash were sampled from five silo units in different TPPs of NTPC Ltd. in India viz. Plant A, B, C, D, and E. The selection of different power plants made it possible to obtain CFA samples from various geographical regions throughout India. For subsequent chemical analysis, all samples were dried and kept at ambient temperature in vacuum-sealed plastic bags. In the present study, HNO_3 (65 %, Suprapur grade, Merck), HCl (30 %, Suprapur grade, Merck), HF (40 %, AR grade, Rankem), and ultrapure deionised water (generated using Elga Labwater system) were used. The analysis was performed utilizing a PerkinElmer NexION 2000B ICP-MS system, which had undergone comprehensive calibration procedures before the analyses. Each sample underwent triplicate measurements to ascertain the standard deviation.

2.2 Sample Pre-treatment

A modified Environmental Protection Agency (EPA) 3052 (Remeteiová et al. 2020), USA 67 method was used for sample preparation. Combustion of samples were performed at 800 ^{o}C in the furnace before digestion to eradicate the presence of organic matter. Weighed accurately 0.35 g of ignited sample and added to 100 mL TFM (modified PTFE) vessel, and then 2 mL HNO_3, 6 mL HCl, and 0.5 mL HF were added subsequently. The cocktail in the vessel was then digested using a microwave digestor (Make: Anton Paar, Austria) for 1 h. After digestion, filtered the resulting acidic solution into a 50 mL volumetric flask (using Whatman No.41 filter paper) and made upto the limit with water. Subsequently, the resultant solution was analyzed for determining REEs concentrations utilizing ICP-MS.

2.3 Standard Preparation

For the preparation of the standard stock solution, a pre-prepared multi-element standard solution of 1000 mg/L from Inorganic Ventures containing a mix of 18 elements (10 µg/mL each; Ce, Dy, Er, Eu, Gd, Ho, La, Lu, Nd, Pr, Sc, Sm, Tb, Th, Tm, U, Y, and Yb in 5% v/v HNO_3) was used. Further, 10 mg/L standard stock solution was prepared directly by the dissolution with 2% HNO_3 solution, which was subsequently used to prepare 5 standard concentrations varying between 0.01 to 0.5 mg/L using 2% HNO_3 as diluent, in order to calibrate the system.

2.4 Instrumentation

The determinations were carried out using a ICP-MS instrument equipped in kinetic energy discrimination (KED) mode. For all elements, ultra-pure argon and helium were used as plasma and collision gas respectively. The instrumental parameters typically applied to the determination of REEs in CFA are summarized in Table 1. The operating conditions were optimized for high sensitivity by conducting a daily performance check prior to data collection using NexION Setup Solution. This enhances the signal intensity upto maximum level and decreasing the oxide formation to 2.5% CeO 155.9/Ce 139.905. For all reported element analyses, no correction equations were used.

Table 1: Parameters of ICP–MS employed in the quantitative determination of REEs in various CFA samples

Component/Parameter	Operating conditions
Spray chamber	Baffled glass cyclonic with AMS port
Nebulizer type	Meinhard Type CT Q+
Torch/Injector	Quartz with integrated 2.5mm id injector
RF power	1600 W
Plasma gas flow	15 L min^{-1}
Auxiliary gas flow	1.20 L min^{-1}
Nebulizer gas flow	1.02 L min^{-1}
Collision gas flow	5 mL min^{-1}

2.5 REEs classification

REEs are stratified into three categories: light rare earth elements (LREE – La, Ce, Pr, Nd, and Sm), medium rare earth elements (MREE – Eu, Gd, Tb, Dy, and Y), and heavy rare earth elements (HREE – Ho, Er, Tm, Yb, and Lu) (Dai et al. 2007; Pathapati et al. 2023; Yunus et al. 2018). Moreover, they are delineated as excessive (EREE - Ce, Ho, Tm, Yb, and Lu), critical (CREE - Nd, Eu, Tb, Dy, Y, and Er), and uncritical (UREE - La, Pr, Sm, and Gd) based on their demand and extensive applications across diverse fields (Seredin 2010; Bishop et al. 2023; Sandeep et al. 2023).

3. RESULT AND DISCUSSIONS

From Figure 1 & Table 2, it is evident that the REEs content in CFA exhibits significant and consistent variation. Ce registers the highest abundance of REEs, ranging from 128 to 325 µg/g, while in contrast, Tm and Lu demonstrate the lowest abundances, with levels ranging from 2 to 3 µg/g. Furthermore, notable concentrations of Nd, La, Th, and U are also observed. It is also noteworthy that amongst the plants, Plant C exhibits the highest REEs concentration. The apparent differences in concentration levels among the CFA samples could be attributed to variances in their geographical origins and extracting methodologies.

The total rare earth element (TREE) content of all CFA samples, with the exception from Plant C, was found to be consistent with the literature data (Smolka-Danielowska 2010; Blissett et al. 2014; Franus et al. 2015; Lin et al. 2017; Dai & Finkelman 2018; Wang et al. 2019; Sandeep et al. 2023). Conspicuously, among the critical REEs Nd demonstrated the highest levels. Other CREE, albeit in lower concentrations, exhibited a similar pattern with other REEs.

Table 2: REEs Concentrations (µg/g) in various CFA samples

Element	Plant-A	Plant-B	Plant-C	Plant-D	Plant-E
Ce	142	128	325	130	133
Dy	18	19	25	19	18
Er	12	10	13	11	10
Eu	5	3	8	4	3
Gd	17	16	23	17	18
Ho	4	5	7	5	4
La	40	37	137	36	34
Lu	2	2	3	3	2
Nd	75	67	165	73	69
Pr	19	18	35	17	16
Sc	20	2	12	12	13
Sm	18	17	25	17	17
Tb	3	5	28	4	4
Th	33	30	75	38	35
Tm	2	2	3	3	2
U	53	40	15	42	46
Y	27	28	22	27	36
Yb	9	10	15	9	11

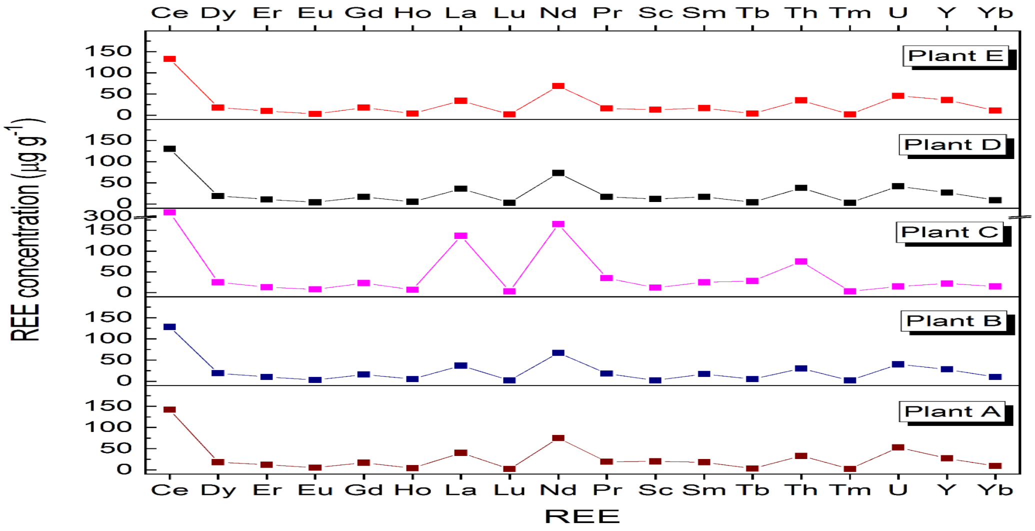

Fig. 1 REEs concentrations in different CFA samples collected from various TPPs

Figure 2 reveals the average individual REEs % contribution to TREE in various CFA samples. The percent average contribution of Ce to TREE was 35.9% and the minimum contribution was apparent for Tm and Lu i.e. 0.55%. In NTPC Ltd. samples of CFA, Nd (18.9%) contributed a greater percentage than Y (6.8%) and Dy (4.5%) among the CREE. Figure 3 shows the distribution of LREE, MREE, and HREE concentrations in Indian CFA, with Plant C showing the maximum respective concentrations of 687, 106, and 41 µg/g. LREE were found to be more concentrated than MREE and HREE in all fly ash samples. The ratio of LREE to HREE ranges between 8.8 and 16.8. The variations in sorption and solubility during the synthesis of REEs in coal may account for the observed variations in the preferential increase of REEs.

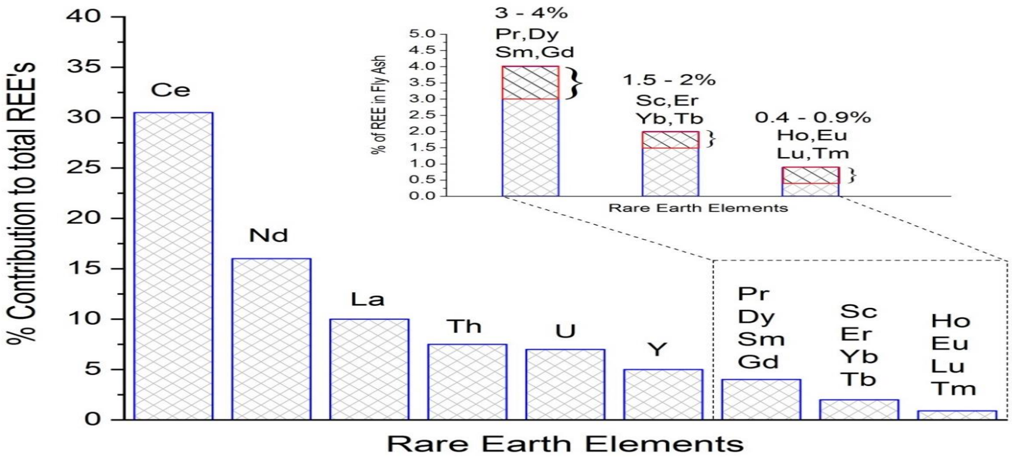

Fig. 2 Average individual REEs % contribution to total REEs in CFA samples

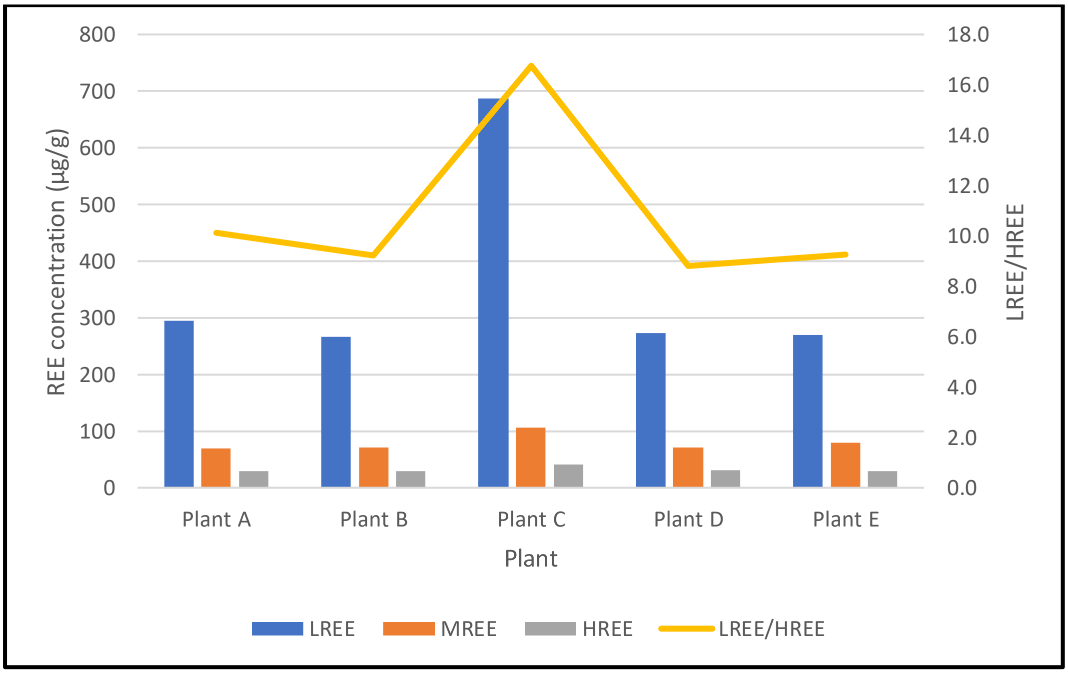

Fig. 3 Distribution of typical REEs concentrations in CFA collected from various power plants

Table 3 presents the comparative data between the REEs content in CFA samples of India and from several other countries. Notable differences are apparent in REEs content of Indian CFA from the global data due to the difference in the geographical origin, kind of coal being used and the combustion processes involved.

Table 3: Comparison of REEs content with global REEs data

REEs	INDIA	INDIA	UK	SPAIN	USA	CHINA	POLAND
Ce	128–325	88.2–218.0	86.6–176.5	64.7–113.0	124–246	266.4	30.7–172.5
Nd	67–165	29.2–66.0	37.2–77.6	58.3–80.0	58–113	114.7	12.7–81.3
La	34–137	28.9–92.7	41.9–85.7	21.0–42.0	57–115	134.4	15.5–81.7
Y	22–36	22.8–48.3	37.3–60.7	95–126	66–132	105.2	17.9–73.2
Pr	16–35	8.0–19.9	9.4–19.3	9.9–15.3	14–29	29.8	3.3–20.5
Sm	17–25	8.9–17.2	7.1–15.4	19.9–22.9	11–24	21.9	2.8–17.0
Gd	16–23	7.0–12.7	7.4–13.4	23.6–30.7	12–25	22.9	2.85–14.65
Dy	18–25	6.9–12.2	6.9–11.1	16.4–25.1	13–26	20.4	2.61–12.18
Tb	3–28	1.1–2.0	1.2–1.9	3.4–4.7	2–4	3.37	0.45–2.40
Er	10–13	3.6–5.7	3.7–6.1	7.6–11.4	7–14	12	1.79–741
Yb	9–15	3.7–5.4	3.4–5.8	5.5–8.3	7–13	11	1.8–6.74
Sc	2–20	21.8–27.7	–	13.6–40.0	83–108	25.9	7–45
Eu	3–8	1.6–2.8	1.7–3.2	4.9–6.3	3–4	4.1	0.56–3.81
Ho	4–7	1.2–2.1	1.4–2.2	2.8–4.5	2–5	3.9	0.59–2.58
Tm	2–3	0.5–0.8	0.5–0.9	0.8–1.5	1–2	1.6	0.27–1.1
Lu	2–3	0.5–0.8	0.5–0.8	0.7–1.2	1–2	1.6	0.3–1.03
Reference	Present Study	Sandeep et al., 2023	Blissett et al., 2014	Dai & Finkelman 2018	Lin et al., 2017	Wang et al., 2019	Franus et al., 2015

Upon further examination, it was revealed from Table 1 that CFA from TPPs contain radioactive elements such as U and Th in the range of 15 to 53 µg/g and 33 to 75 µg/g respectively. The U.S. Geological Survey (USGS) reports that coal typically contains 1-4 µg/g of U, with higher concentrations being rare (Murillo-Tovar 2021; Robertson et al. 2016) and Th concentrations are also within a similar range. The findings of 15-75 µg/g in CFA indicate a significant enrichment of these elements during coal combustion. Research has shown that radioactive elements are more concentrated in CFA compared to coal bottom ash. The reason could be attributed to the finer particle size and the retention of less volatile elements like U and Th in the solid combustion wastes. Thus, future efforts should encompass a thorough examination of the uniformity of REEs and other element contents in the CFA stockpiles. The selection of an appropriate REEs extraction technique will need to be customized based on the properties of individual CFA. Due to the scarcity of information on REEs and other strategically important or potentially harmful element contents in the current fly ash supplies in India (and globally), it is not possible to accurately envisage which extraction process will be the most efficient and which type of forthcoming hazardous waste will be produced. Furthermore, a thorough assessment of the risks to the environment and public health subjected due to these elements needs to be done.

3.1 CFA Potentiality for extracting REEs

In evaluating REEs-rich CFA, two other important parameters, i.e. the outlook coefficient (ratio of critical to excessive elements) and the proportion of critical elements within the overall content, are also used to determine the potential of CFA to extract the REEs (Seredin & Dai 2012; Sandeep et al. 2023; Karadirek 2024). According to Dai et al., a material is deemed to be a promising source for REEs recovery if its outlook coefficient value is ≥ 0.7. In a similar vein, if the percentage of CREE in the material is greater than 30%, it is regarded as a secondary source of REEs (Dai et al. 2017).

Table 4 demonstrates the concentrations of TREE, outlook coefficient, and critical parameters of the CFA. Results reveal that the CFA contained critical elements in the range of 31.29 to 37.14% and had outlook coefficients varying from 0.74 to 0.92. Consequently, based on the aforementioned categorization, they can be deemed as group II potential REEs resources for economic advancement (Seredin & Dai 2012). Sampling from Plant C's CFA may offer a more promising alternative for extracting REEs. Acknowledging the presence of TREE content in fly ashes is crucial, as all samples showed levels that meet or surpass the global average (Ketris & Yudovich 2009; Franus et al. 2015). Several studies on various coal types as potential REEs sources have mostly concentrated on the rare elements in the coal rather than in the associated fly ash. The mineral makeup of coal goes through different changes during and after burning, such as breaking down, turning into vapor, melting, combining, or solidifying. As a result, the levels of REEs in fly ash particles can vary significantly (Jones 1995; Kukier et al. 2003; Izquierdo & Querol 2012;) from coal samples. In addition, it is noteworthy mentioning that in this study, the REEs content obtained from CFA in analyzed sample are assumed to be the same as the REEs content in the entire CFA stock.

Table 4: Classified REEs content (μg/g) in various coal fly ash samples

Element	Plant-A	Plant-B	Plant-C	Plant-D	Plant-E
CREE	140	132	261	138	140
UCREE	94	88	220	87	85
EREE	159	147	353	150	152
TREE	393.0	367.0	834.0	375.0	377.0
CREE/TREE (%)*	35.62	35.97	31.29	36.80	37.14
CREE/EREE	0.88	0.90	0.74	0.92	0.92

**CREE/TREE (%) is the outlook coefficient*

The findings of the current research indicate that Indian CFA contain elevated levels of Ce, Nd and La (Figure 2) in comparison to other REEs, making their economic recovery feasible from Indian fly ash samples. Among these highly abundant REEs obtained from this study, La and Ce play major roles in green energy technologies (Catalysts, Fuel cells and Hydrogen storage). Likewise, Other REEs like Sc (available in moderate concentration in CFA) are extremely important due to the rarity and decline of its direct sources. Because of its high melting point and low density, there is a significant demand for Sc (when combined with Al) in the aerospace sector. In addition to these components, Nd and Dy are commonly employed in the production of magnets. The solvent inventory in a liquid-liquid extraction process would be large, making it not cost-effective. Physical separation process, acid leaching followed by adsorption on to resin polymers is the ideal and economically viable method to recover REEs (especially the HREE) from Indian CFA samples, due to their low concentrations in the lower ppm range (Mondal et al. 2019; Pant et al. 2020).

4. SUMMARY & CONCLUSION

The current research examined the REEs levels in Indian CFA samples from five distinct TPPs of NTPC. Findings reveal that LREEs are predominant in the Indian CFA samples compared to MREE and HREE contents. The difference in REEs levels may be attributed to variations in the quality of coal being utilized and other parameters in coal handling. Out of all the samples analyzed, Ce has the highest abundance among REEs, while Tm and Lu showed the lowest concentrations. CFA from Plant C had the highest overall content of REEs out of the 5 samples, while the CFA from other plants showed a similar trend. This initial study of CFA revealed that every sample tested contained at least 31% CREEs and an outlook coefficient of at least 0.7, indicating they have high potential as valuable sources for REEs recovery. It can be noted that CFA from Plant C contains elevated levels of TREE and, therefore, can be seen as a more effective backup source for REEs extraction. Moreover, the radioactive elements i.e. U and Th, recovered can be possibly used in nuclear power sectors. More research is needed on Indian coal fly ashes to determine the most suitable CFA for use as secondary sources of REEs, despite the promising findings of the current study using ICP-MS.

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CONFLICTS OF INTEREST

The authors declare no conflict of interest with any party that may have seemed to affect the research described in this article.

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