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REE-mineral phases in Indian red mud from the east coast bauxite deposit

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Red mud is often considered a prospective secondary resource of rare earth elements (REE). The Indian red mud samples were characterized using XRD, WD-XRF, ICP-OES and SEM-EDS to study their REE mineralogy and REE content. A major fraction (77%) of the sample was below 45 µm, with total REE content of 433 ppm. There was an enrichment of LREE over HREE, and La, Ce, Nd and Sc were the main contributors to the total REE value. REE-bearing minerals like monazite and zircon occurred as discrete mineral phases in the bauxite residue.

Keywords: Bauxite deposit, monazite, rare earth elements, red mud, zircon.

RED mud or bauxite residue is a solid waste that is produced by aluminium industries during the extraction of alumina from bauxite. It mainly comprises iron oxide, aluminium oxide, silicon oxide and titanium oxide. Generally, it is reddish-brown in colour, and the characteristic red colour is due to the presence of large amounts of iron oxide. The average composition of red mud is Fe₂O₃ (48–54%), Al₂O₃ (17–20%), TiO₂ (3–4%), SiO₂ (4–6%), CaO (1–2%), Na₂O (3–5%)¹. It also contains many trace elements such as Ga, V, Mn, Cr, Ni, Cu, Pb, Zn, Cd, P, Mg, Hf, Zr, Th, U, Nb, Ba, Sr, K and rare earth elements (REEs) in varied proportion^{2,3}. However, the mineralogical and chemical composition of red mud varies widely depending on the source of bauxite and the technological processing conditions applied to recover alumina^{4,5}. It is reported that 0.8–1.5 tonnes of red mud is generated per tonne of alumina produced from bauxite⁶. World inventory of red mud shows that about 60–120 million tonnes are produced annually from the bauxite industry. India is one of the largest producers of red mud, producing about 2 million tonnes⁷. The production of red mud has been rising rapidly^{8–10} and its disposal is one of the major concerns as red mud is highly alkaline and is a potential threat to air, water and land. Hence, red mud is a source of environmental pollution on the one hand^{11,12} and a promising source for obtaining valuable elements on the other¹³. It can be utilized to recover Fe, Al, Ti, Na and REE^{3,14}. It is reported that bauxite residue contains a high amount of scandium (70–260 ppm) that is close to its primary resources^{15,16}. The approximate range of total REE content in red mud is about 500–1700 ppm (ref. 17). Several studies have reported that red mud is a secondary source

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Table 1. Bulk chemical composition of red mud

Oxide	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	Na ₂ O	K ₂ O	CaO	MgO	TiO ₂	Cr ₂ O ₃	MnO	P ₂ O ₅	LOI
Weight %	10.11	17.98	47.93	6.12	0.18	1.26	0.22	3.55	0.09	0.18	0.37	12.01

Table 2. Weight % distribution of different sized fraction of the red mud samples and their REE concentration (ppm)

Size (µm)	+75	-75 + 45	-45
Distribution (wt %)	14.90	8.30	76.80
REE (ppm)			
La	26.5	26	89
Ce	2.2	34.5	151.5
Pr	20.3	14	36.5
Nd	41	37	57
Sm	23.5	24.5	26
Eu	–	–	1
Gd	–	–	–
Tb	7	8	8.5
Dy	–	–	–
Ho	–	–	–
Er	–	–	–
Tm	–	–	–
Yb	5.5	6	6
Lu	8	8.5	7.5
Sc	41.5	42.5	41.5
Y	4.5	5	9
TREE	180	206	433.5
TLREE	113.5	136	361
THREE	66.5	70	72.5
TLREE/THREE	1.7	1.9	5.0

– indicates not detected by ICP-OES.

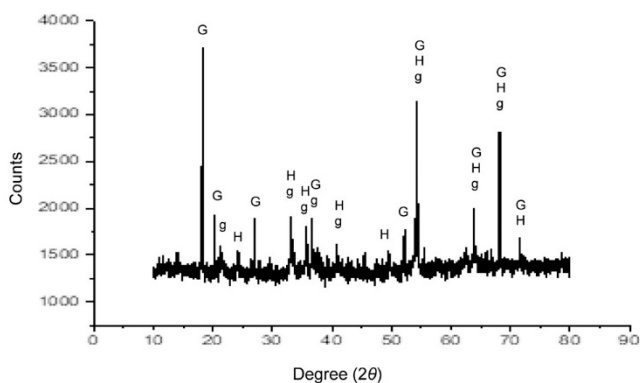


Figure 1. XRD pattern of red mud (G, gibbsite; g, goethite; H, Hematite).

for the recovery of REE^{13,17–19}. As the mineralogy and composition of red mud depend on the source of bauxite ore and the technological process, red mud from India may have some peculiar characteristics, including its REE content²⁰. Though some work has been done on the extraction of REE, mostly La and Sc, from Indian red mud, only a few studies are available on its REE mineralogy. In this present study, we characterize red mud samples from NALCO, Damanjodi, Odisha, India, intending to learn its REE mineralogical characteristics. NALCO is one of the major alu-

mina-producing industries in the country, and its source of bauxite ore is from the East Coast bauxite deposit in southern Odisha. For this study, dry and powdered red mud samples were obtained from NALCO. They were thoroughly mixed and coning, quartering was done to prepare 1 kg of representative sample for characterization. Size classification was done using standard sieves. To ascertain the metal concentration, chemical analysis was performed by WD-XRF (PAN analytical–Zetium) for major oxides and ICP-OES (Perkin, Optical Emission Spectrometer Optima 2100 DV) for REE. The mineralogical study was carried out using XRD (PAN-analytical Model-Xpert PRO) and SEM-EDS (Zeiss EVO18, UK). For the SEM study, polish sections were prepared from the red mud samples by mounting them in epoxy resin. All these studies were carried out at the CSIR-Institute of Minerals and Materials Technology, Bhubaneswar, Odisha.

From size classification, it is found that ~77% of the samples were below 45 µm in size, ~15% above 75 µm, and ~8% of the samples were within 45–75 µm in size. Tables 1 and 2 provide results of the analysis. Table 1 shows that red mud contains 47.93% Fe₂O₃, 17.98% Al₂O₃, 3.55% TiO₂, 10.11% SiO₂, 6.12% Na₂O and other minor elements. Table 2 shows that total REE (TREE) content is more in the finer fractions of the red mud samples. TREE is maximum (433 ppm) in the size fraction of –45 µm followed by 206 ppm in –75 + 45 µm and 180 ppm in +75 µm samples (Table 2). It was also observed that the concentration of LREE was more than the HREE, with the LREE/HREE ratio varying from 1.7 to 5. The LREE value increased from the coarser to the finer fractions, whereas the concentration of HREE did not vary much in different-sized fractions. Major LREE contents of the –45 µm sample were La (89 ppm), Ce (151 ppm) and Nd (57 ppm).

The XRD pattern showed that gibbsite, goethite and hematite were the major mineral phases present in the sample (Figure 1). Using SEM, the other mineral phases such as magnetite, ilmenite, rutile, zircon and monazite were identified along with the major minerals (Figure 2 a, c and e). Monazite and zircon were the major REE-bearing minerals that occurred as discrete grains. Figure 2 b and d shows the electron dispersive spectra along with semi-quantitative analysis of monazite and zircon. Monazite occurred in an irregular shape, whereas zircon was subrounded to rounded. The grain size of the REE phase varied from 10 to 60 µm. Elemental mapping of monazite and zircon, as well as the associated grains, clearly revealed the distribution of REE (La, Ce, Nd, Y) (Figures 3 and 4). The monazite grains contained La – 13%, Ce – 28.2%, Nd – 9.6%, Th – 6.4% and P – 17.2%. The semi-quantitative analysis of all mineral phases measured by SEM-EDX is given in Table 3.

Table 3. Semi-quantitative analysis of different mineral phases present in the red mud samples

Elements/points	1	2	3	4	5	6	7	8
O	28.0	37.6	27.2	35.7	58.7	30.3	31.8	41.9
Si	0.8	15.0		0.6				
Al				3.4	41.3			
Fe			72.8	60.4		29.6	68.2	
Ti						40.1		58.1
Zr		47.3						
P	13.9							
La	12.3							
Ce	28.2							
Nd	10.0							
Th	6.7							
U	0.9							
Mineral	Monazite	Zircon	Magnetite	Goethite	Gibbsite	Ilmenite	Hematite	Rutile

Blank spaces are not detected in the EDAX analysis for a particular mineral at a certain point.

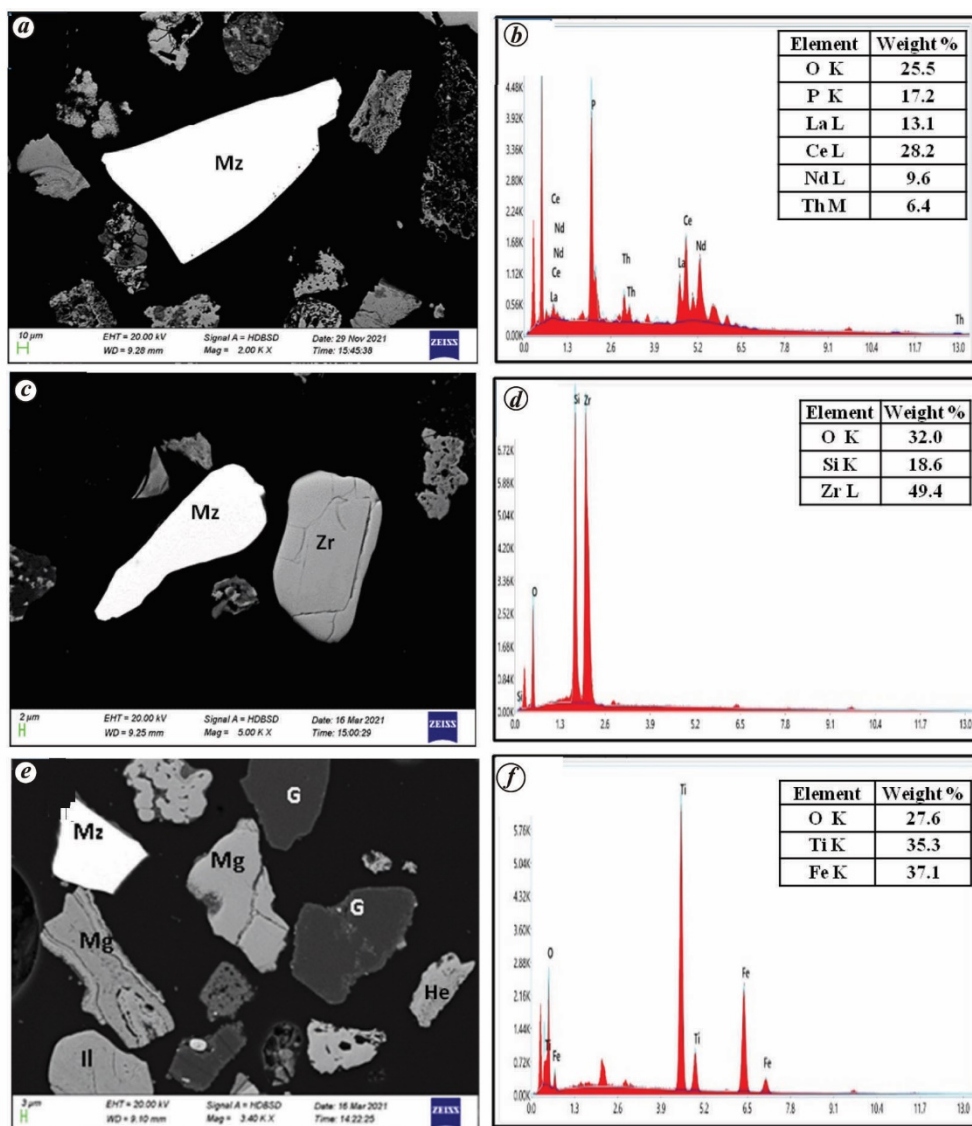


Figure 2. Backscattered electron image of red mud showing mineral phases and their electron dispersive spectra and semi-quantitative analysis. Mz, Monazite; Zr, Zircon; G, Gibbsite; Mg, Magnetite; Il, Ilmenite; H, Hematite. *a*, Monazite grain. *b*, Electron dispersive spectra and semi-quantitative analysis of monazite. *c*, Sub-rounded monazite and zircon grain. *d*, Electron dispersive spectra and semi-quantitative analysis of zircon. *e*, Association of monazite, gibbsite, magnetite, hematite and ilmenite. *f*, Electron dispersive spectra and semi-quantitative analysis of ilmenite.

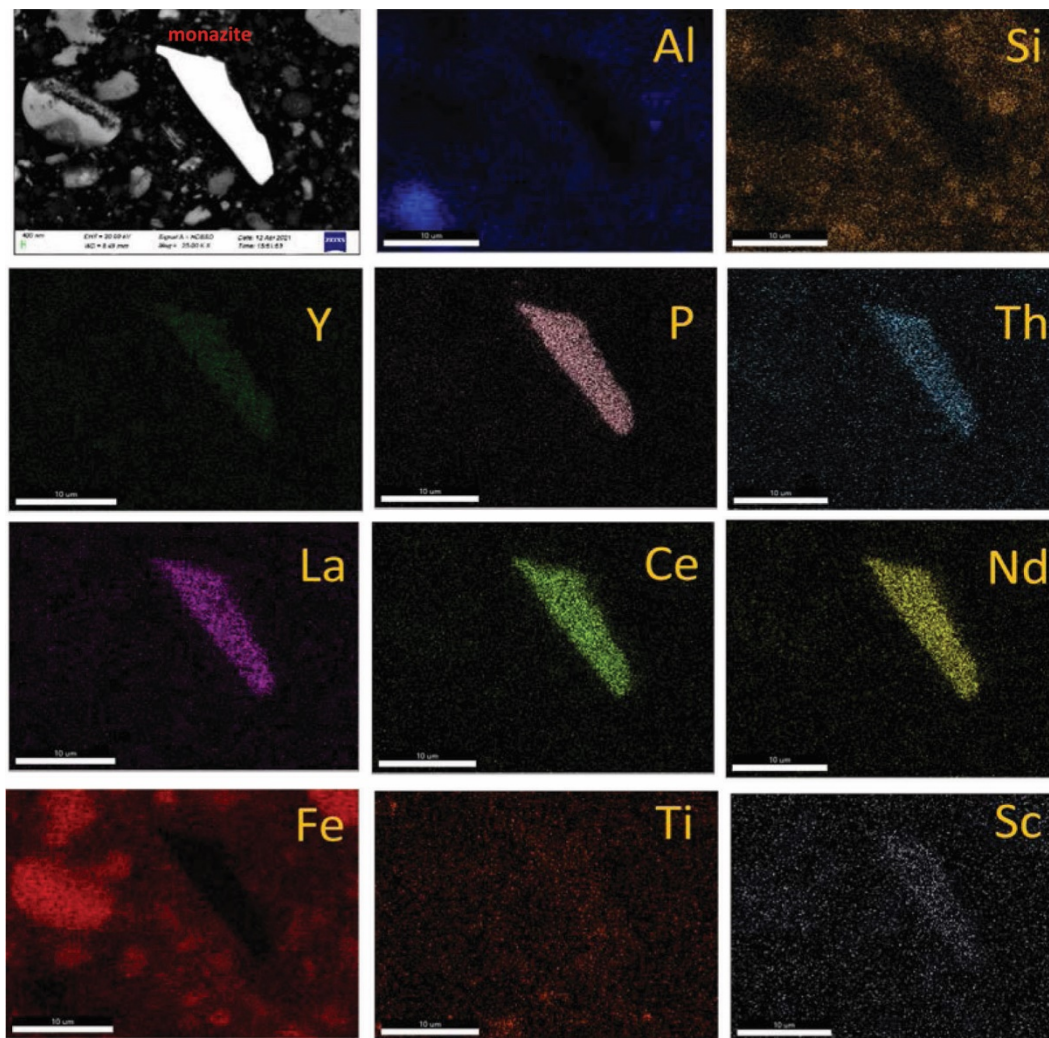


Figure 3. X-ray elemental map of monazite and other associated phases in the red mud samples.

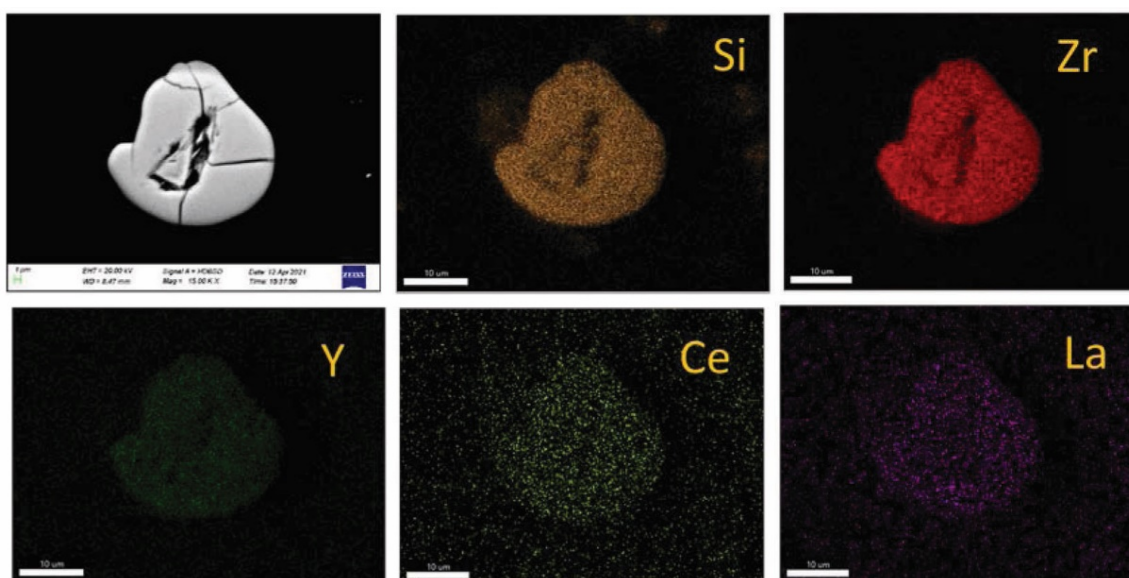


Figure 4. X-ray elemental map of zircon grain showing the presence of REE (La, Ce and Y).

In summary, the red mud samples of NALCO contain about 77% of very fine particles, i.e. below 45 µm, having TREE of 433 ppm. Monazite and zircon are the main REE-mineral phases present as discrete grains along with other minerals such as gibbsite, hematite, goethite, ilmenite, rutile and magnetite. These REE-mineral phases may be concentrated using suitable mineral processing techniques such as floatation and magnetic separation. In view of the current demand and geo-political scenario, there is a need to secure critical resources like REE. Red mud could be a viable feedstock for extracting REE, provided there is a complete understanding of its REE characteristics. This makes the huge accumulation of red mud a resource rather than a waste product. As mineralogy plays a vital role in mineral beneficiation or any route of metal extraction, this study may help in the recovery of REE from Indian red mud in the future.

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Chemical constituents of essential oil from leaves of an invasive weed *Ageratina adenophora* in Central Nepal

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This study aims to identify volatile chemical components in the essential oil of an invasive plant, *Ageratina adenophora*, from Central Nepal. Leaf samples of *A. adenophora* were collected, and the chemical composition

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