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Geochemical Characterisation and Rare Earth Element Enrichment Patterns in Awgu Formation Coals, Middle Benue Trough, Nigeria

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ABSTRACT

Historically, the value of coal has been in its energy potential; nevertheless, it has been increasingly appreciated as a geochemical recorder of past environments and tectonic processes. The present interest lies in its potential for accommodating Rare Earth Elements (REEs), which are essential elements for the current technological revolution. The present research offers new geochemical insights into the exploration of REEs in coals from the Awgu Formation area of the Middle Benue Trough in Nigeria, a region of proven geological significance but yet to be explored. A systematic sampling of twenty-eight coals from the Awgu Formation in the region was carried out and analyzed for the concentration of REEs by inductively coupled plasma mass spectrometry (ICP-MS). The results show moderate to strong enrichment in light REEs (LREEs) and irregular contents of the heavy REEs (HREEs), with possible detrital and diagenetic inputs.

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The observation of positive europium anomalies and shale-normalization of the patterns suggests the presence of felsic rocks as the provenance and low-oxygen conditions during deposition. The study contributes to the resource potential of Nigerian coals and provides a sedimentological outlook for the Cretaceous environments in the Benue Trough. The significance of this study in the present global scenario of resource depletion and the preponderance of Chinese production in the global production of Rare Earths cannot be overemphasized. The coupling of geochemical and stratigraphical parameters adds to the ever-expanding importance of coals beyond fuel and positions them as dual-use geo-resources in the domains of fuel and strategic mineral resources. The present research provides a material contribution to the evolving research in African Rare Earths and sustainable mineral resource policies.

Keywords: Coal, rare earth elements (REE), Awgu formation, Nigeria

1. INTRODUCTION

Coal, though long acknowledged as a fundamental energy resource and industrial raw material, is increasingly recognised as a rich geological archive of ancient Earth systems. The coal that formed from the various biogeochemical processes occurring in prehistoric swampy areas now retains a record of the climatic, environmental, and tectonic processes that got it to be formed the way it is; thus, it serves as both an energy source and the record of the formation history (that is, the stratigraphy) of the coal. The dual nature of coal creates a unique intersection of economic and scientific interests. In recent years, a new dimension has emerged: the occurrence of rare earth elements (REEs) in coal and its derivatives. These 17 metals, including the lanthanides, yttrium, and scandium, are indispensable to modern high-tech applications, ranging from smartphones to missile guidance systems, wind turbines, and electric vehicles [1]. REEs are typically subdivided into light (LREEs: La–Sm±Eu) and heavy (HREEs: Gd–Lu + Y) groups, their distribution controlled by ionic size, redox potential, and volatility [2]. With over 90% of the world's production of coal being produced by China (Department of Energy, 2017), there is a renewed urgency to find alternative sources of coal. Coal seams, as well as combustion products of the burning of coal, are now being examined as a potential source of rare earth elements, and some basins are producing coal with REE concentrations greater than the averages that you would expect to find in the earth's crust [3]. However, the ability to extract REE from coal is closely linked to the mineralogy of the coal in which the REE are hosted (i.e., phosphates, clays, and organic material), as they each have their own set of parameters that impact their extractability [4]. For instance, most LREEs found in bituminous coals are associated with phosphates, while most LREEs found in lower-rank coals are associated with clays. While significant advancements have been made in China, Russia, and the United States [5], much of Africa, particularly Nigeria, remains underexplored. The Middle Benue Trough, a structurally intricate and resource-rich basin in Nigeria, harbours extensive but largely untapped coal reserves. The Awgu Formation coalfields appear to provide a very unique geological setting, yet they have not yet been systematically evaluated for their REE content (a major gap in the knowledge base) (e.g., the methods of formation and deposition of other coal basins containing REE). The lack of systematic evaluations of REE in the Awgu Formation coalfields may be due to the similarity in geologic history between these two coalfields and those of numerous other REE-containing coal basins located worldwide [6]. Additionally, the REE distribution in coal is also indicative of the coal's formation, the chemical environment in which it formed, and the amount of oxygen present during the period of formation. For instance, europium anomalies have been linked to felsic source rocks in basins from Russia to Bulgaria and Nova Scotia [7, 8]. Despite being a leading coal producer, India has only recently begun to systematically assess REE distribution in its Gondwana coals (IEA, 2018).

Besides their economic value, REEs also present environmental challenges during mining and utilisation, underscoring the need for integrated geochemical and ecological assessments [9]. This study, therefore, aims to address a critical gap by unveiling new data on the concentration, distribution, and mineralogical modes of REEs in the Shankodi–Jangwa coals, Middle Benue Trough. Through ICP-MS analysis, it seeks to identify potential REE-bearing phases and inform a broader understanding of resource sustainability within a green economy framework.

2. GEOLOGICAL SETTING OF THE MIDDLE BENUE TROUGH

The Middle Benue Trough, a structurally intricate segment of Nigeria's inland rift system, trends northeastward towards the Bashar–Mutum Biyu axis, marking a stratigraphic transition zone into the younger Gombe and Keri-Keri Formations [10]. Despite its geological richness, this region remains underrepresented in detailed mapping efforts, though select areas south of Bashar have been examined through photogeological surveys [11, 12, 13, 14, 15, 16, 17, 18]. Earlier works by [19], and [20] have established a foundational stratigraphic framework for regions such as Lafia, Keana, and Awe. Stratigraphically, the Middle Benue Trough reveals a dynamic marine–continental succession. The oldest marine sediments, attributed to the Albian Asu River Group, comprise the fossiliferous Arufu, Uomba, and Gboko Formations [16, 17, 21, 22]. These units represent early marine transgressions into the trough, occasionally interrupted by tectonic compressional phases [23]. Overlying these are the Cenomanian–Turonian Awe and Keana Formations, indicative of a major regressive phase [22], succeeded by renewed marine incursions forming the Eze-Aku Group and Awgu Formation [24]. These marine deposits persisted until the Santonian tectonic upheaval, a phase marked by extensive folding and uplift due to global plate reorganisation [25]. Capping this sequence is the Lafia Formation, the only Campanian–Maastrichtian unit in the Middle Benue Trough, deposited before widespread Tertiary volcanism commenced. These stratigraphic and tectonic chronicles not only highlight the basin's complex evolution but also underscore its paleogeographic link between the Tethys Sea and the South Atlantic [26].

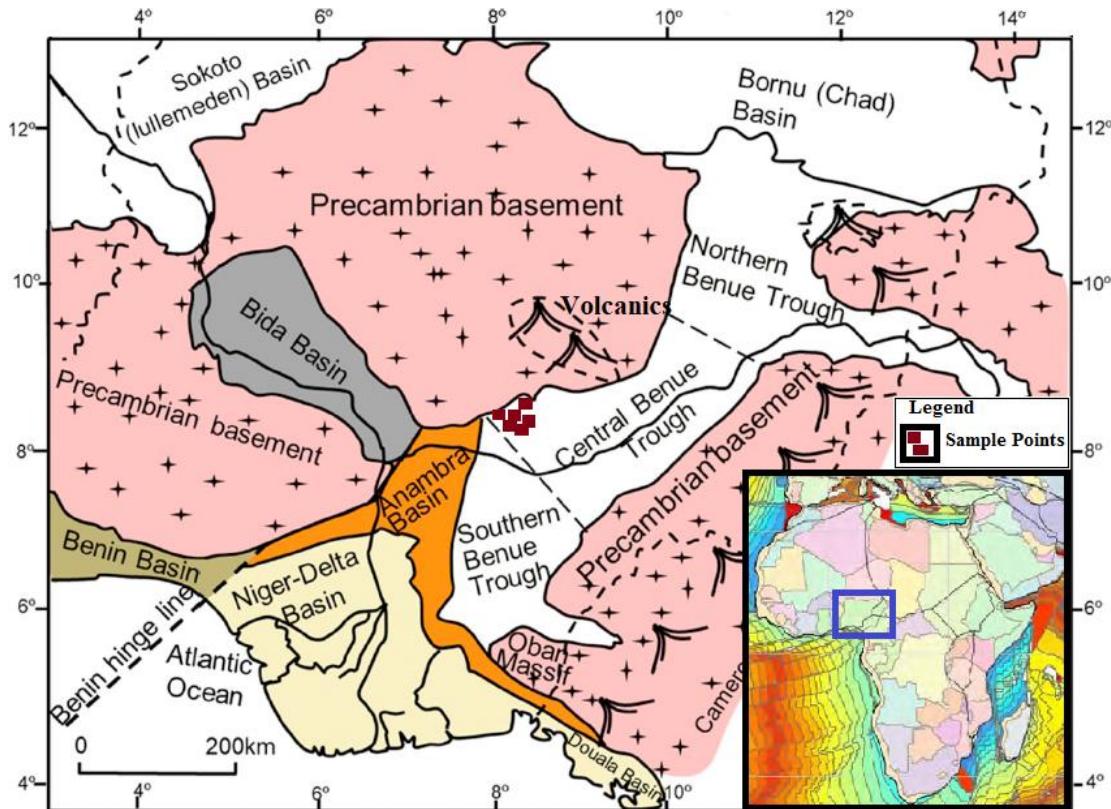


Figure 1. Regional Geological Map of Nigeria showing the study area [27, 28].

SYSTEM		STAGE/ EPOCH	NORTHERN BENUE TROUGH (Carter et al, 1963)	CENTRAL BENUE TROUGH (Ofoegbu, 1984)	SOUTHERN BENUE TROUGH (Peters & Ekweozor, 1982)
QUATERNARY		HOLOCENE			
PLEISTOCENE		Chad Fm			
CENOZOIC	NEO-GENE	PLIOCENE			
		MIOCENE			Benin Fm
	PALEO-GENE	OLIGOCENE			Ogwashi-Asaba Fm
		EOCENE			Ameki Fm/Nanka SS
CRETACEOUS	UPPER CRETACEOUS	PALEOCENE	Kerri Kerri Fm	Volcanics	Imo Sh
		MAASTRICHTIAN	Gombe SS/ Lamja SS	Lafia Fm	Nsukka FM Ajali SS Mamu Fm
		CAMPAIGN	Pindiga Formation	Numanha Sh	Nkporo Sh/ Owe III SS/ Enugu Sh/ Afikpo SS
		SANTONIAN			
	MID-CRETACEOUS	CONIACIAN	Fika Sh/ Sekunle Fm	Awgu Fm	Awgu Fm/ Agbani SS
		TURONIAN	Gongila Fm Jesse Fm/ Dukul Fm	Makurdi Fm	Eze-Aku Sh/ Amasiri SS
		CENOMANIAN	Yolde Fm	Awe/Keana Fm	Odukpani Fm/Agala SS
		ALBIAN	Bima SS 3 Bima SS 2	Eze-Aku Sh Arufu/Uomba Fm (Asu River Group)	Awe Fm Abakaliki Sh
		APTIAN	Bima SS1		Asu River Grp
		PALEOZOIC	PRECAMBRIAN	BASEMENT COMPLEX	Awi FM
		Legend	FM= Formation	SS= Sand stone	Sh=Shale
				=Alluvium Deposit	=Unconformity

Figure 2. The stratigraphic sequence of the Benue Trough of Nigeria; the red boxes indicate the coal bearing formations (modified after [10]).

3. MATERIALS AND METHODS

In this study, twenty-eight coal samples were systematically collected from outcrops of the Awgu Formation exposed along the banks of the River Dep in the Awgu Formation axis near Obi, within the Middle Benue Trough. Due to accessibility constraints and regional insecurity, sample recovery was limited; nonetheless, a proportional stratified random sampling strategy ensured representative coverage across coal seams, following procedures by [29]. Each seam, ranging from 20 to 150 cm in thickness, was sampled based on lithotype band variations. To ensure integrity and freshness, coal surfaces were meticulously scraped before lithological logging and sample extraction. Approximately 200 g of each sample was securely packed in zip-locked polythene bags and transported to the laboratory. Sample preparation adhered strictly to ASTM D-2013 standards (ASTM D2013/D2013M-12, 2012). A hammer mill initially reduced the particle size to <2.36 mm (-8 mesh), followed by riffle splitting into <20-mesh and <60-mesh fractions. The finer 60-mesh fraction was subsequently pulverised for geochemical analysis. The chemical characterisation, particularly the rare earth element (REE) profile, was conducted via Inductively Coupled Plasma Mass Spectrometry (ICP-MS) at ALS Geochemistry Laboratory, Vancouver, Canada. Each sample (50–100 g) was ground using an agate ring mill to prevent contamination. Major element analysis involved flux fusion with $\text{LiBO}_2/\text{Li}_2\text{B}_4\text{O}_7$ at 980°C, followed by dissolution in 5% HNO_3 . Analytical error margins ranged between 1–2% for major elements. For REEs and trace metals, samples were digested over 48 hours using concentrated HF, HNO_3 , and HClO_4 on a hot plate at ~150°C, followed by dilution in 1% HNO_3 . Digestion vessels were pre-cleaned using 55% HNO_3 and 30% H_2O_2 , with leaching completed in two stages. Final analyses were performed using a Perkin Elmer NexION ICP-MS, maintaining accuracy within ±5%.

4. RESULTS AND DISCUSSION

4.1. Results

The Rare Earth Element (REE) distribution patterns in the Awgu Formation coals, based on the provided geochemical data (Tables 1 and 2), reveal a significant enrichment of Light Rare Earth Elements (LREE) compared to Heavy Rare Earth Elements (HREE) (Figs. 3, 4a, and b). The REE distribution follows a characteristic pattern where LREEs (La to Sm) are found in higher concentrations than HREEs (Gd to Lu). The total REE concentrations range from 3.9 ppm to 58.2 ppm, with an average of 15.7 ppm. The distribution pattern showed a negative slope (steep for LREE and flatter for HREE), typical of coal samples enriched in light elements. Sample MBC 07: This sample stands out with the highest Σ REE (58.2 ppm) and highest LREE enrichment, Σ LREE = 52.6 ppm. The coals are characterized by a distinct dominance of LREE over HREE. Average LREE (13.2 ppm), HREE (2.6 ppm). The Σ LREE/ Σ HREE ratio ranges from 2.8 to 9.4 (average 4.1), highlighting the relative enrichment of light rare earth elements. Analysis of the normalized values reveals specific anomalies that provide insight into the depositional environment. Most samples exhibit a negative Cerium anomaly (Ce/Ce^*) (average 0.7), with values generally below 1.0 (ranging from 0.5 to 1.1). A negative anomaly often suggests oxidizing conditions during deposition. The samples show a consistent positive Europium anomaly (Eu/Eu^*) (average 1.7), with values ranging from 1.0 to 2.7. Positive Eu anomalies in coals can sometimes be attributed to the influence of feldspar-rich source rocks or hydrothermal fluids.

Table 1. Rare Earth Elements (ppm) and ratios of some trace and rare earth elements normalized values of Awgu Formation coal in the Middle Benue Trough.

Sample ID N=28	MBC01	MBC 02	MBC 03	MBC 04	MBC 05	MBC 06	MBC 07	Average
Eu	0.1	0.2	0.1	0.3	0.2	0.3	1.5	0.4
Gd	0.3	0.4	0.2	0.4	0.3	0.5	0.6	0.4
Tb	0.1	0.1	0.04	0.2	0.2	0.3	0.5	0.2
Dy	0.3	0.5	0.2	0.5	0.5	0.8	1.02	0.5
Ho	0.1	0.2	0.1	0.2	0.2	0.3	0.6	0.2
Er	0.1	0.2	0.1	0.3	0.2	0.3	0.5	0.2
Tm	0.02	0.02	0.02	0.04	0.03	0.04	0.1	0.03
Yb	0.2	0.3	0.2	0.3	0.3	0.4	0.4	0.3
Lu	0.1	0.3	0.1	0.2	0.3	0.4	0.5	0.3
La	0.7	1.3	0.5	1.5	1.2	2.1	9.6	2.4
Ce	1.6	3.1	1.2	3.3	2.5	4.5	22.9	5.6
Pr	1.0	0.5	0.4	0.5	0.5	0.7	1.7	0.7
Nd	0.3	2.3	0.7	2.0	1.3	2.4	13.7	3.4
Sm	5.5	0.8	0.2	0.7	0.4	0.8	4.7	1.1
Σ REE	4.1	10.3	3.9	10.4	8.0	13.8	58.2	15.7
Σ LREE	1.4	8.0	3.0	8.0	5.9	10.5	52.6	13.2
Σ HREE	1.4	2.3	0.9	2.5	2.1	3.2	5.6	2.6
Σ LREE/ Σ HREE	3.0	3.6	3.2	3.3	2.8	3.3	9.4	4.1
Σ HREE/ Σ LREE	0.3	0.3	0.3	0.3	0.4	0.3	0.1	0.3
Ce/Ce*	0.6	0.8	0.5	0.7	0.7	0.7	1.1	0.7
Eu/Eu*	1.0	1.2	1.7	1.7	1.7	1.4	2.7	1.7

Table 2. Rare Earth Elements (ppm) summation and geochemical anomalies ratios.

Rare Earth Element Parameter	Average Value (ppm/ratio)	Range
Σ REE	15.7	3.9–58.2
Σ LREE	13.2	1.4–52.6
Σ HREE	2.6	0.9–5.6
Σ LREE/ Σ HREE	4.1	2.8–9.4
Ce/Ce*	0.7	0.5–1.1
Eu/Eu*	1.7	1.0–2.7

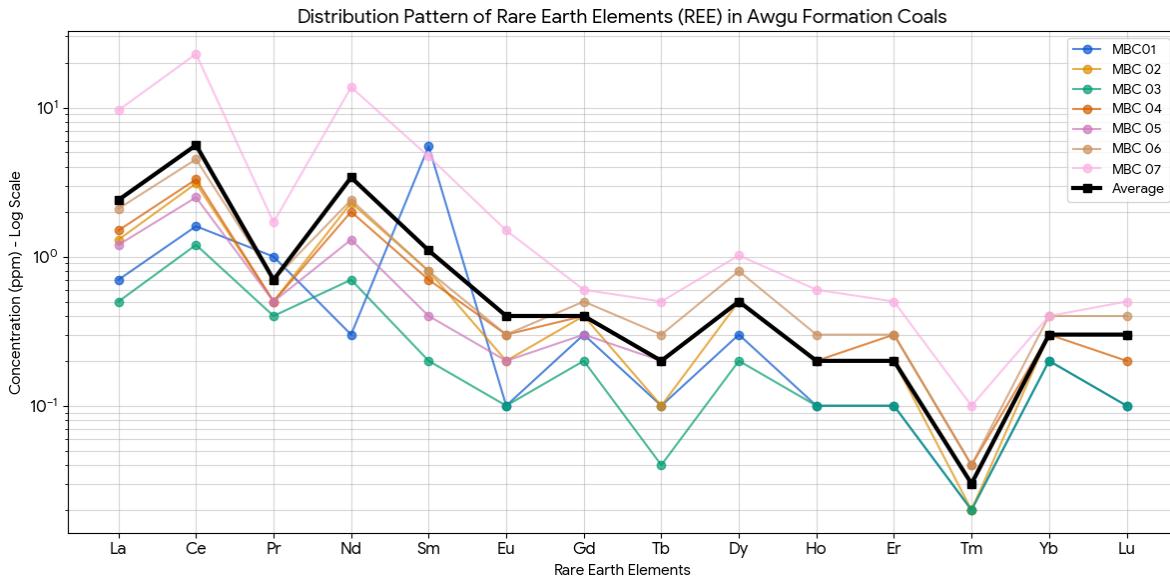


Figure 3. REE distribution patterns spider plot in Awgu Formation coal in the Middle Benue Trough.

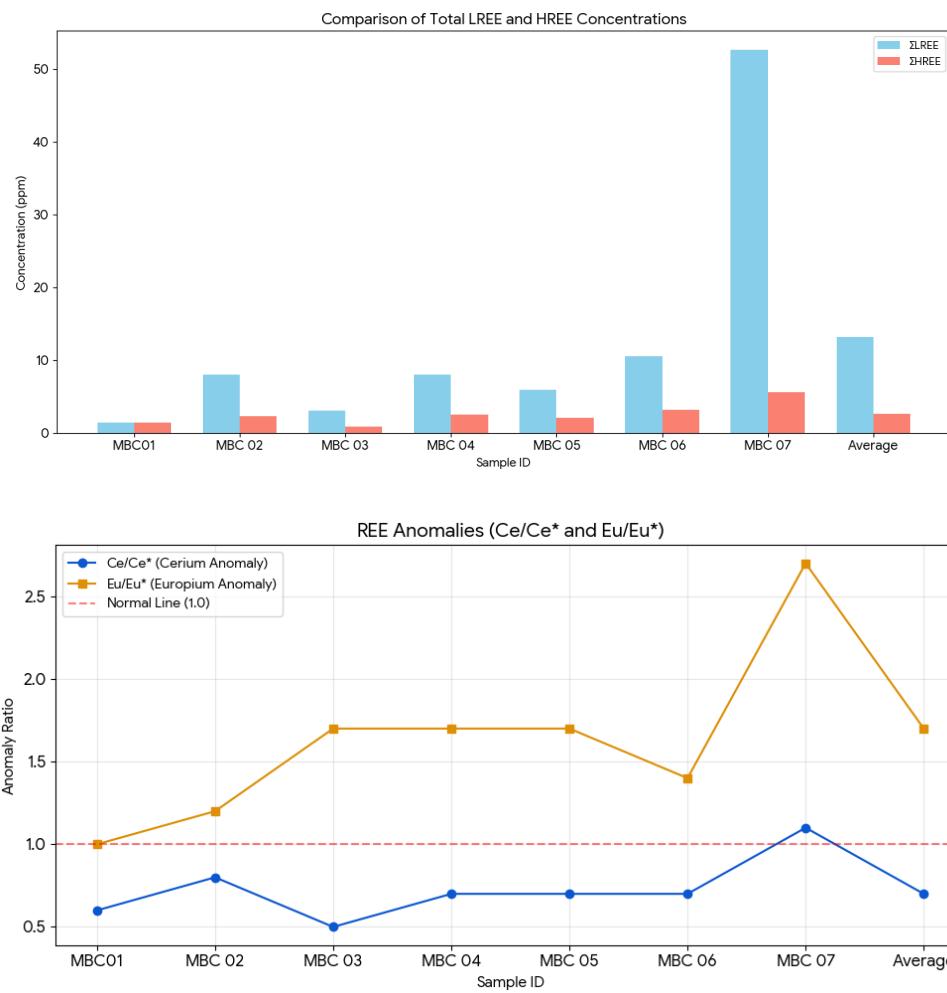


Figure 4. (a) LREE vs. HREE Comparison, (b) Geochemical Anomalies in Awgu Formation Coal.

4.2. Discussion

Rare Earth Elements (REEs), known for their indispensable role in modern technologies, offer valuable insights when investigated in coal. Their geochemical behaviour serves not only as a tracer for depositional and diagenetic processes but also signals potential economic value. The present study evaluates REE concentrations in coal seams of the Awgu Formation, Middle Benue Trough, Nigeria, with a particular focus on the Awgu Formation axis. As shown in Table 1, the total REE (Σ REE) content across all samples averaged 110.01 ppm, higher than the global coal average (68.5 mg/kg) but slightly below Chinese coals (137.9 mg/kg), as previously reported by [30] and [31]. This elevated REE profile is comparable to North American shales, reflecting either a volcanic ash input, continental weathering, or groundwater influence during peat accumulation [32, 33]. Light REEs (LREEs: La, Ce, Nd, Pr, Sm) dominate over Heavy REEs (HREEs: Gd, Tb, Dy, Ho, Er, Tm, Yb, Lu), as seen in most global coal profiles [33]. LREE/HREE ratios range from 2.78 to 9.41 (avg. 4.07), indicative of crustal enrichment patterns, likely due to the greater solubility and abundance of LREEs in natural environments. Compared with the Post-Archaean Australian Shale (PAAS), most samples appear depleted in LREEs, implying a lower input of felsic detritus. Cerium anomalies (Ce/Ce^*) serve as redox indicators. The values observed here ($\text{Ce}/\text{Ce}^* < 1$) suggest a suboxic depositional environment, where Ce is retained in its trivalent state rather than oxidised [34, 35]. Europium anomalies (Eu/Eu^*) ranging between 1.00 and 2.74 (avg. 1.65) - highlight a positive trend, often attributed to detrital feldspar or hydrothermal influx during diagenesis [33]. Figures 3 and 4 show a moderately flat LREE to HREE slope with a pronounced Eu peak, forming a distinct 'Λ'-shaped REE pattern typical of B-type coal REE distribution [36]. This homogeneity implies that the REE-bearing minerals were deposited under similar sedimentological conditions, with minimal post-depositional remobilisation. Understanding the binding states of REEs within coal matrices is fundamental for assessing their recoverability and mobility. The findings here reveal that REEs occur predominantly in the silicate-bound phase, followed by organic associations, with negligible proportions in water-soluble and exchangeable forms (Tables 2, 3, 4). These results align with the modes proposed by [33, 37], who classified REE occurrence in six states: water-soluble, exchangeable, carbonate, organic, silicate, and sulphide. Sequential chemical extraction suggests that detrital mineral inputs, particularly aluminosilicates - are the primary carriers of REEs, with organic matter acting as a secondary host. This supports previous interpretations by [38, 39], highlighting the syngenetic to early diagenetic incorporation of REEs during peat formation. In essence, the Late Turonian-Early Santonian coals of Awgu Formation not only display a distinctive REE fingerprint reflective of their depositional and source rock history but also suggest limited post-depositional alteration. The coherent REE distribution and consistent occurrence modes indicate that these coals hold potential for REE recovery - albeit under environmentally cautious, economically viable frameworks.

Table 3. The chemical occurrences and extraction methods of REEs Number Modes of occurrence.

Number	Mode of occurrence	Extraction
I	Water soluble state	4g coal sample + 30ml water, 25°C, 24hours
II	Exchangeable state	Residue+30ml NH ₄ AC, 25°C, 24hours
III	Carbonate state	Residue of II+1.47g/cm ³ CHCl ₃ , sinking +10ml 0.5%HCl, 20min

IV	Organic state	Residue of II+1.47g/cm ³ CHCl ₃ , floating dried at 40°C, 1:1 HNO ₃ and HClO ₄ added, 5hours
V	Silicate state	Residue of III+2.89g/cm ³ CHBr ₃ , floating +1:1 HNO ₃ and HF, 5h
VI	Sulfide state	Residue of III+2.89g/cm ³ CHBr ₃ , sinking +1:1 HNO ₃ , 5hours

Table 4. Recovery of sequential chemical extraction experiments for a Awgu Formation coal (%).

Modes of occurrence	La	Ce	Nd	Sm	Eu	Tb	Yb	Lu	LREE	HREE	REE
Water soluble state	0.1	0.1	0.1	0.1	1.1	1.2	0.4	2.8	0.2	0.5	0.7
Exchangeable state	0.1	0.1	0.1	0.2	1.2	1.2	0.8	2.8	0.2	0.6	0.8
Carbonate state	3.0	4.4	5.6	7.3	8.0	9.3	31.0	27.8	3.5	8.5	12.0
Organic state	2.8	2.1	3.0	4.2	9.1	14.0	13.1	13.9	2.7	5.1	7.8
Silicate state	75.1	76.2	72.1	70.0	63.7	65.3	51.4	47.3	44.6	20.5	65.1
Sulfide state	19.9	16.3	19.0	18.1	17.2	8.2	4.4	5.7	11.3	2.3	13.6

5. CONCLUSION

The coal of the Awgu Formation, in the Middle Benue Trough of Nigeria, has been studied successfully as a significant strategic mineral resource that has not been well studied before. Using Inductively Coupled Plasma Mass Spectrometry (ICP-MS), it was determined that concentration levels of Rare Earth Elements (REEs) in the coal were found to be higher than the global average, with a maximum value of 58.2 ppm. There are larger amounts of LREEs than there are HREEs, as well as distinct positive europium anomalies that indicate continued use of this geologic material during the Late Turonian–Early Santonian by providing a stable felsic environment that facilitated a suboxic environment during the deposition. The sequential chemical analysis of the coal indicates that the REEs were primarily located in the silicate-bound phases and within the organic matter of the coal. This indicates that the REEs were incorporated into the coal at the time of deposition and that there was little post-depositional re-mobilisation of the REEs. Geochemical stability is a key consideration for any future extractive potential of the host rocks. While providing an academic study of the coal resources in this region, the results further establish the dual-use nature of the Awgu Formation coal as both a traditional fuel and a source of strategic minerals needed for the Transition to a Green Energy Future. As the world moves to diversify supply chains from the current monopolies that exist, the coal resources of Nigeria offer a dormant source of great economic and geopolitical value. In addition to providing the empirical base for formulating national mineral policies to support the technological investment needed to develop these.

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