



An assessment of Rare Earth Elements in borehole cores from the Ermelo, Witbank and Waterberg Coalfields, South Africa: Focus on mode of occurrence

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Dates:

Received: 29 May 2024

Accepted: 12 Jun. 2024

Published: October 2024

How to cite:

Modiba, D.G. and Wagner, N.J. 2024. An assessment of Rare Earth Elements in borehole cores from the Ermelo, Witbank and Waterberg Coalfields, South Africa: Focus on mode of occurrence. *Journal of the Southern African Institute of Mining and Metallurgy*, vol. 124, no.10, pp. 559–566

DOI ID:

<http://dx.doi.org/10.17159/2411-9717/692/2024>

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This paper is based on a presentation given at the Southern African Rare Earths 2nd International Conference 2024, 19-20 June 2024, Swakopmund Hotel and Entertainment Centre, Swakopmund, Namibia

Abstract

There is currently limited knowledge concerning South African coal deposits as hosts for rare earth elements (REE). This project aims to determine the concentration of REE, including yttrium and scandium (REY+Sc) in various coal seams and adjacent sediments in borehole cores from the Ermelo, Witbank, and Waterberg coalfields in South Africa. Coal petrography (maceral count and vitrinite reflectance), XRD, XRF, sequential chemical extraction procedure (SCEP) before ICP-MS, and mineral liberation analysis (MLA) were conducted on the coal and associated sediment samples. The coals were inertinite-rich, medium-rank bituminous coal, with moderate to high ash content. The two dominant minerals in all the coalfields are kaolinite and quartz, except for sample ZBS2M, where dolomite was dominant. The dominant major oxides are Al₂O₃, SiO₂, Fe₂O₃, and CaO, which concur with the XRD results. The ICP-MS results following SCEP indicated that both coal and the associated sediments were light REY+Sc dominant. There was also an elevation of medium REY+Sc in the Witbank coal's associated sediment samples. Notably, the samples with the highest REY+Sc results were the sediment samples. The Waterberg samples had the lowest REY+Sc from both the coal (less than 2 µg/g to 7 µg/g) and associated sediments (2 µg/g to 15 µg/g). While the highest REY+Sc was yielded from the Witbank-associated sediments (500 µg/g to 2,500 µg/g). The carbonate fraction step proved to be the most successful leaching step, as most REY+Sc was recovered in that step for all coalfields. In addition to the carbonate fraction, the silicates and sulfide fraction in the Witbank samples, and the ion-exchangeable fraction for the Waterberg-associated sediments samples proved successful in leaching out the REY+Sc. MLA determined that monazite and xenotime in all the coal samples were the REY+Sc-bearing minerals.

Keywords

REY+Sc, coal, South Africa, sequential chemical extraction procedure, ICP-MS

Introduction

There is an increase in the demand for critical elements such as lithium and rare earth elements globally with the advent of the fourth industrial revolution, green energy, and the need to reduce global greenhouse gas emissions (Bauer et al., 2022; Dai and Finkelman, 2018; Hower et al., 2018). Rare earth elements (REE) Sc, Y, La, Ce, Pr, Nd, Pm, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, and Yb, including yttrium and scandium (REY+Sc), are relatively abundant in the upper continental crust, despite being referred to as rare, which is a misnomer. The challenge is the separation of the elements from each other. According to the International Union for Pure and Applied Chemistry (IUPAC), the REE are 17 elements found on the periodic table, comprising 15 elements in the lanthanide group, plus two transition metal elements, namely yttrium and scandium, collectively referred to as REY+Sc for the purpose of this paper.

According to Zhou et al. (2017), there are 178 REY+Sc deposits globally and as of the year 2017, they were reported to amount to 478Mt REY+Sc, either from primary or secondary sources. Balaram (2019) further elaborated that the primary sources of REY+Sc are from magmatic, hydrothermal, and metamorphic processes, while the secondary source is from weathering and erosion of the primary sources. The global demand has led to a need to broaden extraction from both primary mineable ore deposits, and to expand extraction from alternative, non-conventional sources, such as coal and combustion ash (Dai and Finkelman, 2018; Seredin and Dai, 2012). The increased interest in investigating REY+Sc in coal bears its challenges, such as gaining a clear understanding of the element's host minerals and potential extraction techniques.

To determine the best technique for REY+Sc extraction in coal, there needs to be an understanding of the modes of occurrence in which these elements occur. Internationally, many studies have been dedicated to this topic, including work conducted by Arnold (2023), Balaram (2019), Borzykowski (2019), Dai et al.

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(2004), Dai et al. (2017), Fu et al. (2022), Hower et al. (2018), Li et al. (2008), Li et al. (2023), Serendin and Dai (2012), and Yang et al. (2023). These studies proposed that REY+Sc may be associated with either the organic or inorganic component of coal material. A stronger organic association is mainly observed in low-rank coals. REY+Sc may be associated with the inorganic i.e., mineral fractions of coal in authigenic and clastic minerals, for example, in phosphate minerals such as monazite and xenotime, carbonate minerals like bastnaesite, and accessory minerals such as zircon and apatite (Arnold, 2023; Bauer et al., 2022; Seredin and Dai, 2012). There are two classifications of REY+Sc associated with coal, according to Seredin and Dai (2012). In the first classification, the elements are classified geochemically into Light REE (La, Ce, Pr, Nd, Pm, Sm), Medium REE (Eu, Gd, Tb, Dy), and Heavy REE (Ho, Er, Tm, Tb, Lu). The second classification is based on their market or economic importance and is classified into whether they are critical (Nd, Eu, Tb, Dy, Y, Er), not critical (La, Pr, Sm, Gd), or excessive (Ce, Ho, Tb, Yb, Lu).

Despite the traction globally, there is limited knowledge regarding REY+Sc in South African coals. Wagner and Matiane (2018) focused on coal and coal ash from 3 power stations Eterigho-Ikelegbe et al. (2021) reviewed the enrichment and mode of occurrence of REY+Sc in coal and coal discards. Mokoena et al. (2022) investigated the effects of acid concentration on the recovery of REE from coal fly ash. More recently, Chitlango et al. (2023) reported on REE in density-fractionated coal samples from the Waterberg coalfield. The knowledge gap in South Africa brought about the inception of the current research, intending to determine the concentration of REY+ Sc in a variety of South African coals and their associated sediments in borehole cores from the Ermelo, Witbank, and Waterberg coalfields. In this paper, we discuss coal characterization and the sequential analytical technique used for leaching, prior to the inductively coupled plasma mass spectrometry

Table I
Coal sample names per coalfield

Coalfield	Sample name
Ermelo	UNI2 A, UNI2 B, UNI2 C, UNI2 D, UNI2 E, UNI2 F, UNI2 G, UNI2 H, UNI2 I, UNI2 J, UNI2 K, UNI2 L, UNI2 M, UNI2 N, UNI2 O
Witbank	ZBSDR1, ZBS5T, ZBS5M, ZBS5B, ZBSHF1, ZBSDR2, ZBS4T, ZBS4M, ZBS4B, ZBSYF1, ZBSDR3, ZBS3, ZBSYF2, ZBSDR4, ZBS2Y, ZBS2M, ZBS2B, ZBDIF
Waterberg	12A, 11A, 10A, 9B, 8A, 8B, 8H, 8J, 8K, 7B, 6A, 5A, 4A, 4B, 3A, 3B, 3C, 2A, 2B

(ICP-MS) analysis to determine the association and concentration of REY+Sc in coal and associated sediment samples

Methodology

Sample collection

The Ermelo and Witbank coalfields are situated within the Mpumalanga province, over 200 km and 130 km east of Johannesburg, respectively. The Waterberg coalfield is in the Limpopo province, 297 km north of Johannesburg (Figure 1). We logged full borehole cores from the study areas and sampled both coal and lithologies found immediately above and below the coal seams. We obtained fifteen, eighteen and twenty coal and associated sediment samples from the Ermelo, Witbank, and Waterberg cores, respectively (Table I).

Sample preparation and characterization

All coal samples and associated sediments were crushed and milled at the University of Johannesburg (UJ), using the Retsch ZM200

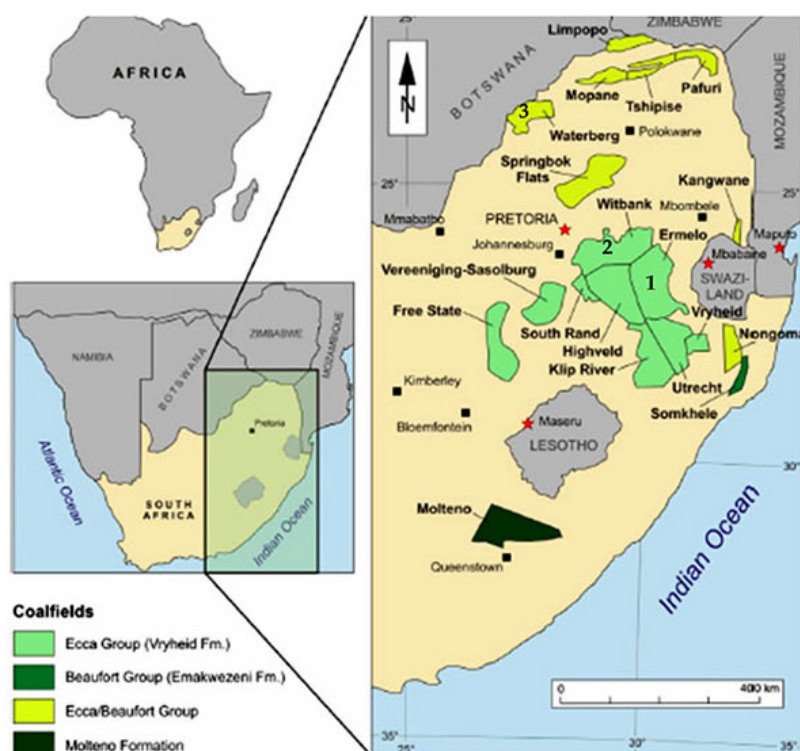


Figure 1—South African coalfields with the Ermelo (1), Witbank (2) and Waterberg (3) coalfields (modified from Hancox and Gotz, 2014)

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ultra-centrifugal mill to achieve homogenous -1 mm samples. A riffler was used to split each sample into three sub-samples. Sub-sample one (-1 mm) was retained for petrography. Coal petrographic analysis is based on 500-point counts per sample, categorizing the components observed as either vitrinite, liptinite, inertinite, or one of five mineral groups (i.e., clay, quartz, sulfides, carbonates, and other minerals). This is based on the South African National Standard/International Organization for Standardization (SANS/ ISO) 7404-3 (2016).

Sub-sample 2 was milled to -212 μm for X-ray diffraction (XRD), X-ray fluorescence (XRF), proximate and total sulfur analyses. The proximate analysis was conducted following SANS/ ISO standard number 17246 (2011). The mineral content was determined using XRD and the bulk chemistry (as major element oxides) in the samples were determined using whole rock analysis by XRF. Sub-sample 3 was further milled, using a pestle and mortar, and passing a screen of 106 μm in preparation for the sequential chemical extraction procedure (SCEP), a chemical leaching process, prior to inductively coupled plasma mass spectrometry (ICP-MS) analysis. All the coal samples used in the SCEP and subsequently analysed using ICP-MS are fresh coal samples. Samples with the highest REY+Sc values, following ICP-MS analysis, were re-sampled and ashed for further analysis using elemental mapping measurement mode on the mineral liberation analyser (MLA).

X-ray diffraction analysis

XRD analysis was outsourced to XRD Analytical and Consulting, Pretoria, South Africa. Samples were prepared for XRD analysis using a backloading preparation method. Diffractograms were obtained using a Malvern Panalytical AERIS diffractometer with a PIXcel detector and fixed slits with Fe-filtered Co-K α radiation. The phases were identified using X'Pert Highscore Plus software. The Rietveld refinement method estimated the relative phase amounts (weight %).

Whole rock geochemistry - X-ray Fluorescence (XRF)

XRF analysis was outsourced to Bureau Veritas Testing and Inspection South Africa, Centurion, South Africa. A 0.7 g of a pulverized sample was added to 7.0 g of 66:33 LiT, LiM (LiI) flux and fused using a Claisse M4 fusion instrument. The resulting fusion discs were measured on a PANalytical Epsilon 3-XL EDXRF spectrometer equipped with a 50 kW 3mA Ag-tube, 10 sample carousel, He-purge chamber, and silicon drift detector.

Sequential chemical extraction procedures (SCEP) and ICP-MS

SCEP was conducted at UJ, Spectra labs. This procedure has been adapted from Dai et al. (2004). SCEP entails the chemical leaching of samples by different lixiviants. One sample is leached in six steps depending on which lixiviant is used. The steps are water-soluble, ion-exchangeable, carbonates, organically bound, silicates, and sulfides. Each sample derived from the respective step is analysed in the ICP-MS to determine the REY+Sc concentration. A PerkinElmer NexION 300X ICP-MS, housed at UJ was used for element determination in the coal and associated rock samples.

Mineral liberation analysis (MLA)

For MLA analysis, coal samples with the highest REY+Sc content were selected from each coalfield (UNI2M, ZBS2B, and 11A). The sample preparation for MLA was outsourced to SJT MetMin Services, South Africa. These samples were ashed at 450°C for eight hours, to remove all volatile matter, and then mounted in

30 mm diameter epoxy resin blocks and cured. The prepared resin blocks were analysed at the Spectrum MLA laboratory, UJ. A scanning electron microscope FEI QUANTA 650 FEG instrument was operated under a vacuum to ensure no interactions with air particles occurred. A back-scattered electron detector (BSE) was used at 25 kilovolts so that the elemental contrast could be used to locate the rare earth-containing carriers of interest. The BSE images were captured using MLA processing software. The results presented in this paper indicate which minerals host the respective REY+Sc. The results are presented as BSE images with their respective spectral plots.

Results and discussion

General characterization

The coal samples consist mainly of coaly-shales, dull-banded coal, and bright coal. The associated sediments are mainly clastic material consisting of carbonaceous shale, mudstone, sandstone, siltstone, and diamictite. All coal samples have moderate to high ash content. Samples from the Witbank coalfield have the highest average volatile matter of 25%. All samples have low sulfur content, the highest value is from the Witbank coalfields, sample ZBS4T, with 5.9% sulfur content.

Petrographic results are shown in Figure 2 and Figure 3. Ermelo coal samples are classified as high inertinite, medium-rank D/C bituminous coal, while the Witbank and Waterberg coal samples are also high inertinite, medium-rank C bituminous coal. The dominant minerals, according to the petrographic analysis, are clay, quartz, calcite, and pyrite. These results agree with studies conducted by Mahooana (2022), Moroeng et al. (2024), and Wagner and Matiane (2018). Figure 3 illustrates the presence of calcite and clay in photomicrographs.

Mineralogy: XRD

The mineralogy results are represented in Figure 4a-c per coalfield. The two dominant minerals in all the coalfields are kaolinite and quartz, except sample ZBS2M where dolomite was dominant. These are followed by muscovite and microcline in the Ermelo and Witbank samples. Trace to minor amounts of calcite and pyrite were detected in samples from all three coalfields, while dolomite was only detected in samples from the Witbank and Waterberg coalfields. These findings were corroborated by the petrography results.

Whole rock geochemistry - X-ray Fluorescence (XRF)

The dominant major element oxides are Al₂O₃, SiO₂, Fe₂O₃ and CaO, as indicated in Table II. These values reconcile with the mineralogy results discussed above and with work published by Bauer et al. (2022), Dai and Finkelman (2018), and Moroeng et al. (2024).

SCEP and ICP-MS analysis results

The SCEP results are indicated in Figures 5 to 7 a for the coal samples and b for the associated sediment samples). All the coal and associated sediment samples indicate that there is an enrichment in LREY+Sc. There is also an elevation of MREY+Sc for the Witbank-associated sediments (Figure 6b). Both coal and associated sediment samples display an affinity for the carbonate fraction. This agrees with the findings discussed by Moroeng et al. (2024). There is more REY+Sc recovered from the associated sediments compared to the coal samples for all the study areas.

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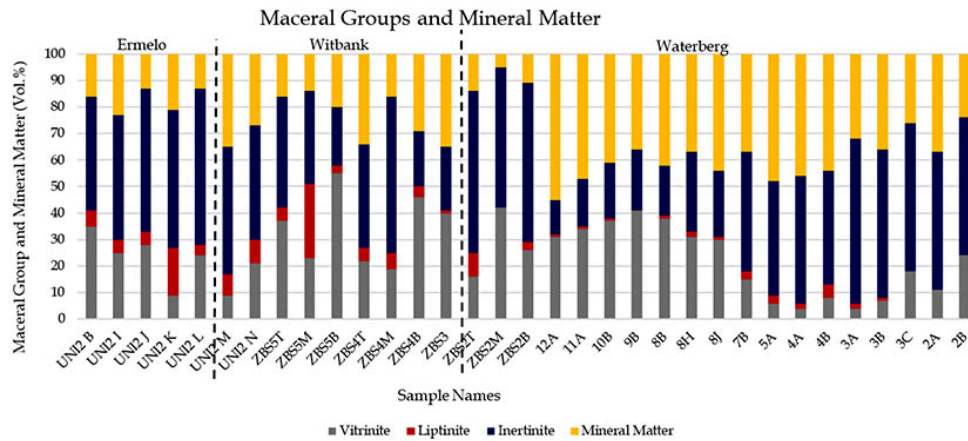


Figure 2—Maceral groups and mineral matter for all coal samples (vol.%)

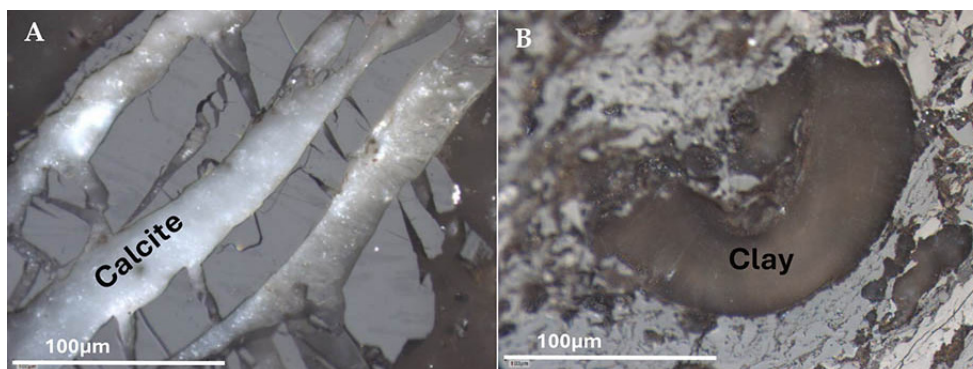


Figure 3—Photomicrographs of reflected white light with immersion oil; total magnification x500. (A) Calcite veins truncated by vitrinite particles, indicating an epigenetic association. (B) Vermicular clay embedded into dominant inertinite

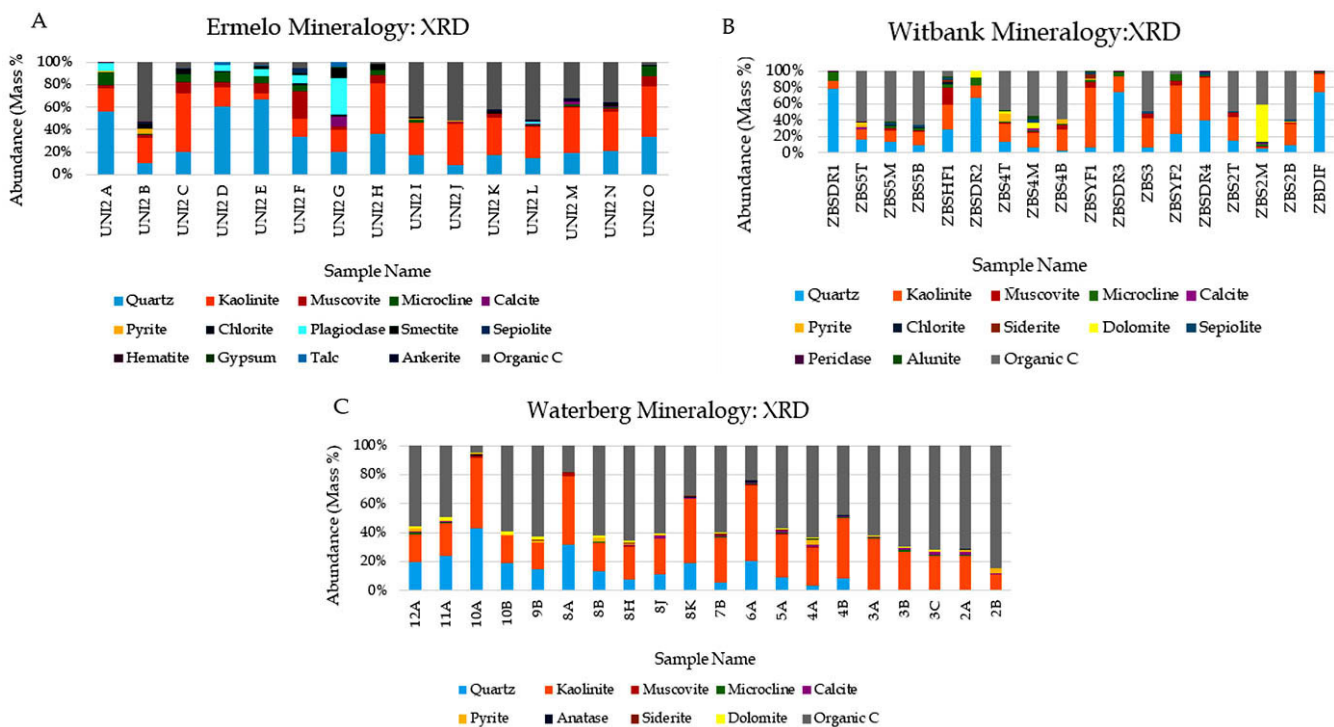


Figure 4a–c—Abundance of minerals from coal and associated sediment samples

REY+Sc for the carbonate fraction in the Ermelo coal samples range from below 2 µg/g for the medium and heavy REE to ~16 µg/g and 7 µg/g for Ce and La, respectively (Figure 5a). While the associated sediments, indicated by Figure 5b, range from

~3 µg/g on average for the medium and heavy REE. Up to a maximum of ~35 µg/g and 18 µg/g for Ce and Nd, respectively. A similar pattern is observed in the Witbank coal samples, as indicated in Figure 6a. The medium and heavy REE are below 2 µg/g The light

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Table II

Average major oxides per coalfield (Weight %)

	Al ₂ O ₃	SiO ₂	Fe ₂ O ₃	CaO	Cr ₂ O ₃	K ₂ O	MgO	MnO	Na ₂ O	P ₂ O ₅	TiO ₂	V ₂ O ₅	ZrO ₂	BaO	SrO	ZnO	SO ₃
Ermelo	20.4	63.3	5.6	3,2	0.0	2.3	1.4	0.0	0.5	0.2	1.1	0.0	0.1	0.1	0,1	0.0	1.6
Witbank	22.0	61.4	5.3	4.2	0.0	1.5	1.5	0.0	0.3	0.2	1.1	0.0	0.1	0.1	0.1	0.0	2.2
Waterberg	27.6	55.6	6.2	4.2	0.0	0.9	0.6	0.0	0.1	0.5	1.5	0.0	0.1	0.1	0.1	0.0	2.3

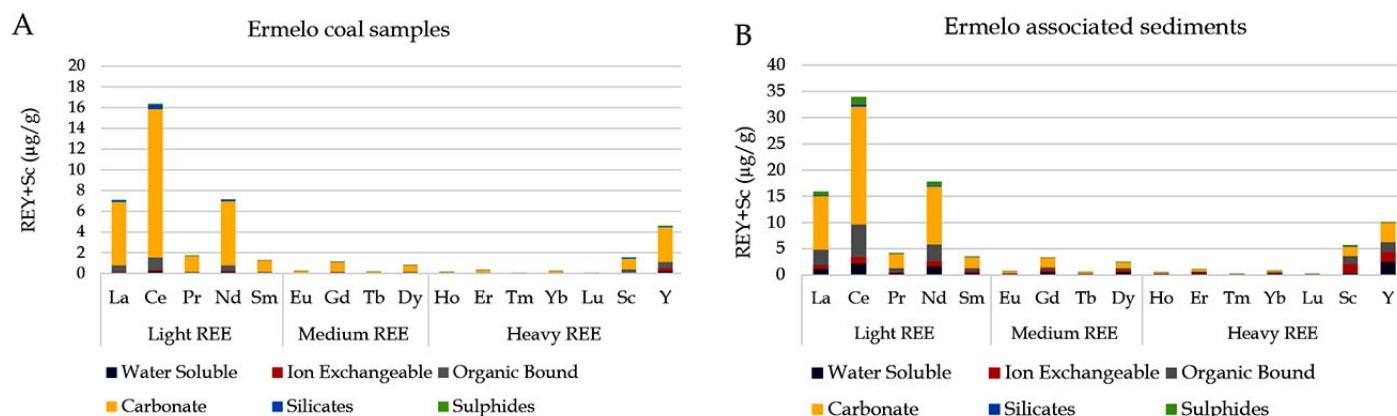


Figure 5a,b—Ermelo SCEP REY+Sc fractions

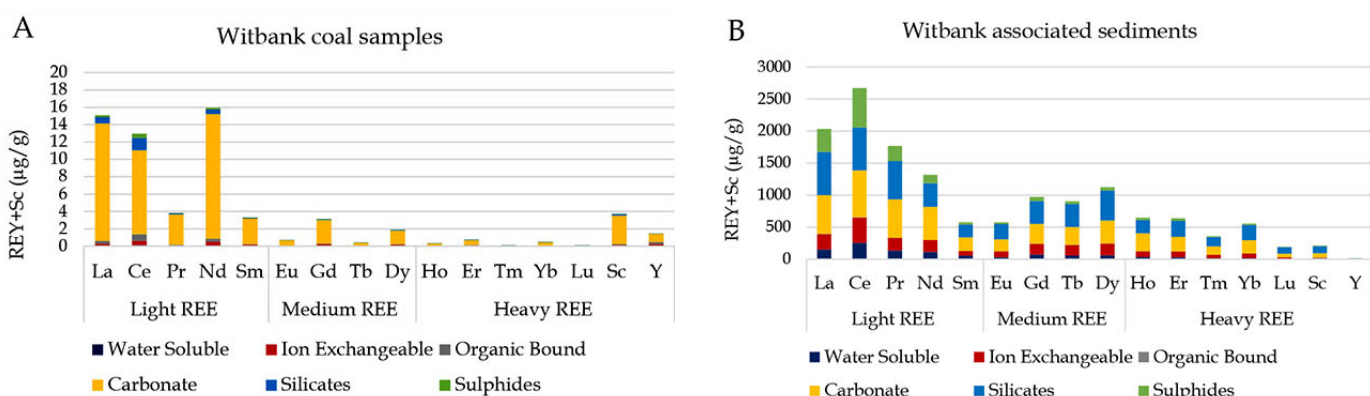


Figure 6a,b—Witbank SCEP REY+Sc fractions

REE displayed Ce amounting to 16 µg/g, followed by Nd and La at ~7 µg/g. The carbonates fraction yielded the most results in the Witbank coal samples. The carbonate, silicates, and sulfide fraction results from the Witbank coalfield-associated sediments yielded the highest REY+Sc from the SCEP process, ranging from ~200 µg/g to 600µg/g for HREE. Followed by ~500 µg/g to over ~1,000 µg/g for MREE. The LREE values ranged from ~500 µg/g to over ~2,500 µg/g for Ce, La, being the highest. Research conducted by Dai et al. (2004), Li et al. (2008), and Yang et al. (2023) also discussed REY+Sc associated with silicates and alumino-silicates and clays.

The Waterberg coal and associated samples yielded the lowest concentration for all fractions and compared to the samples from the other two coalfields. The coal samples have the highest concentrations in the carbonates and silicates fractions. This ranges from less than ~ 2 µg/g to ~ 7 µg/g, indicated by Figure 7a. The Waterberg-associated samples indicated by Figure 7B have the highest concentration in both carbonates and ion-exchangeable fractions. This ranges from less than ~ 2 µg/g to ~ 15 µg/g. The REY+Sc associated with the ion-exchangeable fraction are also

discussed in the work conducted by Bauer et al. (2023). The SCEP results also imply that the highest concentrations of REY+Sc in a particular fraction indicate the highest leaching potential of the lixiviant used in that step.

MLA

Monazite and xenotime particles were detected in all three samples (Figures 8a and 9a). In one example, monazite is locked in kaolinite (Figure 8a). Figure 8b indicates that the main rare earth elements present are La, Ce, Pr, Nd and Gd. Figure 9a depicts a xenotime grain attached to kaolinite. The main rare earth elements detected in the xenotime grain are Y, Gd, Tb, Dy, Ho and Er.

Conclusions

The borehole core samples examined are medium-rank C bituminous coals with moderately high ash content, low volatile matter and low sulfur content. The minerals present in all coal samples were mainly kaolinite and quartz, followed by muscovite, microcline, dolomite and trace amounts of calcite and pyrite. Both coal and associated sediment samples are enriched with LREY+Sc, with an elevation of MREY+Sc in the Witbank associated sediment

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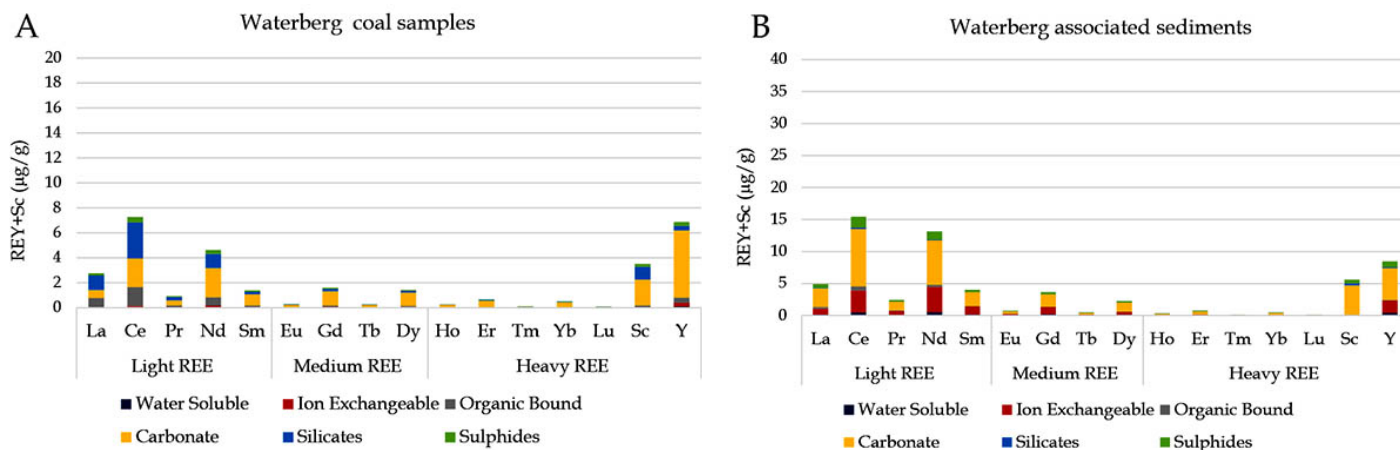


Figure 7a,b—Waterberg SCEP REY+Sc fractions

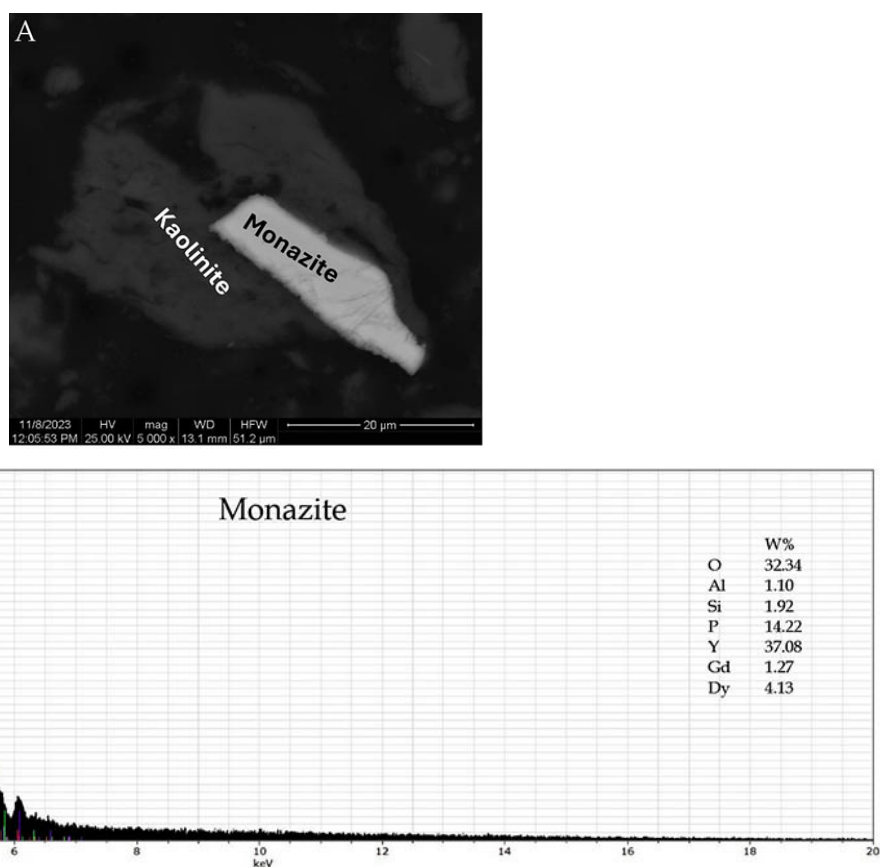


Figure 8—Back-scattered electron image of a monazite grain locked in kaolinite (a), MLA spectra taken from the monazite grain (b)

samples. There is more REY+Sc recovered from the associated sediments compared to coals following the SCEP. The Waterberg has the lowest REY+Sc in both the coal and associated sediment samples, while the highest REY+Sc was yielded from the Witbank associated sediments. The carbonate fraction step proved to be the most successful leaching step, as most REY+Sc were recovered on that step. This may also indicate an affinity for the carbonate minerals present, indicated by dolomite and calcite. However, there is also an affinity for silicates, sulfide fraction in the Witbank samples, and the ion-exchangeable fraction for the Waterberg associated sediments samples. MLA determined that monazite and xenotime in all the coal samples were the REY+Sc-bearing minerals and most are locked in kaolinite.

Acknowledgements

The authors are grateful for the support from the Centre of Excellence for Integrated Mineral and Energy Resources Analysis (DSI-NRF CIMERA) at the University of Johannesburg and the University of Johannesburg Spectrum Analysis Facility. This work is based on research financially supported by the National Research Foundation (NRF) of South Africa under Grant Number 118564. Any opinion, findings, conclusions, or recommendations expressed in this material are those of the author(s) and therefore, the NRF does not accept any liability in regard thereto. The authors also thank Dr TV Makhubela for his support in conducting the study.

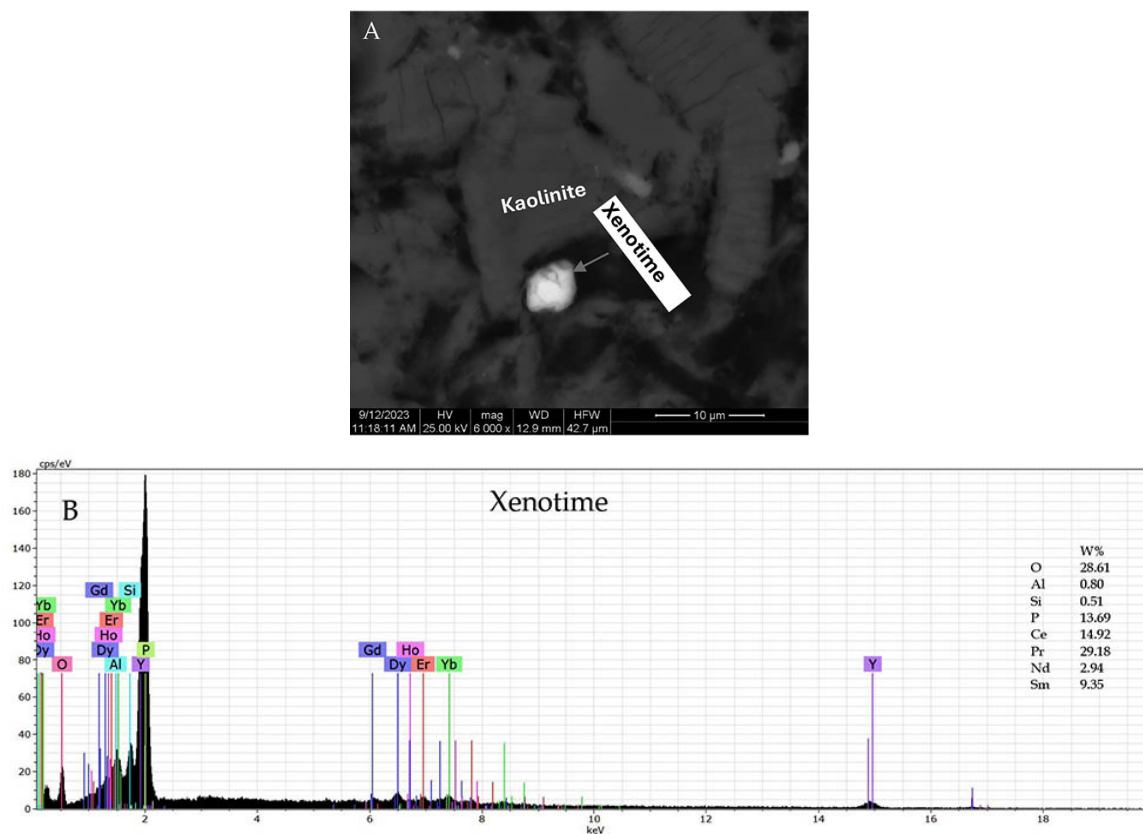


Figure 9—Back-scattered electron image of a xenotime grain locked in kaolinite (a), MLA spectra taken from the xenotime grain (b)

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CONFERENCE CENTRE, STELLENBOSCH

OBJECTIVES

- To expose delegates to issues relating to the generation and handling of sulphur, sulphuric acid, and SO₂ abatement in the metallurgical and other industries
- Provide an opportunity to producers and consumers of sulphur and sulphuric acid and related products to be introduced to new technologies and equipment in the field
- Enable participants to share information about and experience in the application of such technologies
- Provide an opportunity for role players in the industry to discuss common problems and their solutions.

EXHIBITION AND SPONSORSHIP

There are a number of sponsorship opportunities available. Companies wishing to sponsor or exhibit should contact the Conferences and Events Co-Ordinator.

ECSA Validated CPD Activity,
Credits = 0.1 points per hour attended



FOR FURTHER INFORMATION, CONTACT:
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