Proceedings of the 11th World Congress on New Technologies (NewTech'25)
Paris, France - August, 2025
Paper No. ICEPR 160
DOI: 10.11159/icepr25.160

Evaluation of Natural Clay Minerals for Heavy Metal Adsorption from Lead-Acid Battery Recycling Waste

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Abstract –The escalating demand for sustainable energy storage underscores the critical role of lead-acid batteries (LABs), yet their recycling generates hazardous wastes laden with toxic heavy metals. This study evaluates natural kaolinite and bentonite clays as low-cost adsorbents for immobilizing lead (Pb) and arsenic (As) in LAB recycling wastes from South Africa. Mineralogical characterization revealed complex slag compositions dominated by sodium sulfates, lead oxides (litharge), and sulfides (galena). Leaching tests (SPLP: pH 4.2; water leaching: pH neutral) demonstrated significant mobilization of Pb (981 ppm) and As (153 ppm) from slags, with Pb release amplified under neutral conditions. Adsorption experiments showed bentonite reduced Pb by 99.4% (981 → 5.91 ppm) in recycled slag leachate, outperforming kaolinite (95.5% reduction). For As, bentonite achieved partial reduction (153 → 107 ppm), while kaolinite increased As concentrations due to competitive ion effects. Elemental releases (Al, Si, Na) post-treatment indicated clay dissolution, highlighting practical limitations. Results affirm bentonite's superior efficacy for Pb immobilization but stress the need for enhanced As adsorption strategies. These clays offer a viable, locally sourced solution for mitigating LAB waste impacts in developing regions, advancing circular economy goals in battery recycling.

Keywords**:** Lead-acid battery waste, Clay adsorption, Heavy metal leaching, Bentonite remediation, Kaolinite treatment, Sustainable waste management, Adsorption isotherms, Environmental remediation

1 INTRODUCTION

The global shift towards sustainable energy systems has intensified the demand for reliable and cost-effective energy storage technologies. Lead-acid batteries (LABs) remain essential due to their affordability, recyclability, and dependable performance in automotive, industrial, and renewable energy applications. Approximately 85% of global lead consumption is devoted to battery production, underscoring the critical role of LABs in modern energy infrastructure [1].

Despite their benefits, the recycling of LABs produces hazardous waste streams—including slags, sludge, and metal-laden effluents—that contain toxic heavy metals such as lead (Pb), cadmium (Cd), zinc (Zn), and arsenic (As). Improper disposal or treatment of these wastes poses serious risks to human health and the environment [2]. In many developing countries, including South Africa, insufficient waste management infrastructure often results in the co-disposal of LAB waste in unregulated landfills, contributing to groundwater contamination and environmental degradation [3].

While LAB recycling reduces reliance on primary lead mining—which is both energy-intensive and environmentally detrimental—current waste treatment practices remain inadequate. Existing containment strategies, such as synthetic geomembranes, tend to be costly and vulnerable to mechanical damage or chemical degradation [4]. Therefore, there is an urgent need for low-cost, sustainable alternatives capable of stabilizing heavy metals in waste streams under realistic environmental conditions.

Natural clay minerals, especially bentonite and kaolinite, have emerged as promising adsorbents for environmental remediation. These layered aluminosilicates possess high cation exchange capacity (CEC), large specific surface area, and negatively charged surfaces, all of which enhance their affinity for heavy metal ions [5]. Bentonite, rich in montmorillonite, is characterized by its swelling capacity and interlayer ion exchange, while kaolinite offers structural stability and can be chemically modified to improve adsorption [6].

Despite encouraging laboratory studies, practical applications of clay-based adsorbents for treating LAB recycling waste are limited. Most research does not fully account for complex industrial conditions, such as mixed contaminant presence and fluctuating pH, which affect adsorption efficiency. Furthermore, the long-term structural integrity and reusability of clays after metal adsorption remain insufficiently understood, representing a critical knowledge gap in waste management [7].

This study therefore focuses on evaluating the potential of natural clay minerals, specifically bentonite and kaolinite, as sustainable and cost-effective adsorbents for mitigating heavy metal contamination in hazardous waste generated during lead-acid battery (LAB) recycling processes. It aims to address critical gaps in understanding the performance of these clays in immobilizing heavy metals under real-world conditions, investigating their structural stability, adsorption efficiency, and reuse potential. By leveraging the abundant availability of clay minerals in South Africa and integrating mineralogical, chemical, and environmental assessments, this study aims at developing a useful approach to hazardous waste management, promoting sustainable practices that are both technically effective and economically feasible for the battery recycling industry.

2 METHODS

2.1 Sample collection

Five soda ash slags and one gypsum sludge sample were collected from a battery recycling facility in Vereeniging, South Africa. Samples were hand-grabbed near the blast furnace to ensure they were representative of actual processing conditions. Kaolinite (Grahamstown, Eastern Cape) and Eccabond bentonite (Heidelberg, Western Cape) were sourced from a local supplier. These clays are known for their high cation exchange capacities, low permeability, and strong adsorption properties.

2.2 Characterisation techniques

2.2.1 Bulk mineralogy

Battery waste and clay samples were pulverised (if not already in powder form) and subjected to X-ray diffraction (XRD) using a Bruker D8 Advance X-ray diffractometer. Rietveld refinement and the fundamental parameters approach were applied for phase quantification. Clay identification included glycolation and heat treatments (400°C and 550°C) per USGS protocols.

2.2.2 Phase composition

Electron probe microanalysis (EPMA) was performed using a JEOL JXA-8230 electron microprobe to quantitatively assess phase compositions at 20 kV and 30 nA, whereas scanning electron microscopy with energy-dispersive spectrometry (SEM-EDS) was performed on carbon-coated epoxy-mounted samples using a Zeiss EVO SEM and Bruker EDS to identify elemental associations.

2.2.3 Bulk chemical composition

Bulk chemistry was determined via X-ray fluorescence spectrometry (XRF), while major and trace elements were quantified using inductively-coupled plasma-optical emission- and mass spectrometry (ICP-OES and ICP-MS). ICP-MS was preferred for elements at trace levels, such as Pb, Cd, and As.

2.3 Leaching tests

Leaching tests were conducted using two methods to assess elemental release under different environmental conditions. The Synthetic Precipitation Leaching Procedure (SPLP), following EPA Method 1312, involved leaching 100 g of sample with an acidic extraction fluid composed of H_2SO_4 and HNO_3 at pH 4.2 ± 0.05 for 18 hours, followed by filtration. To simulate natural weathering under neutral conditions, 200 g of slag was mixed with 500 mL of deionised water at a liquid-to-solid ratio of 2.5:1, stirred continuously for 24 hours.

Leachates from both methods were analysed for elemental concentrations using a combination of techniques: XRF for major elements, ICP-OES for moderate concentrations and ICP-MS for trace metals. Detection limits were carefully considered, especially for Pb, where more sensitive methods may be necessary for precise quantification.

2.4 Adsorption tests

The adsorption capacities of kaolinite and bentonite clays were evaluated using leachates from water leaching experiments, specifically from recycle slag and brown slag samples, which contained the highest concentrations of Pb and As, respectively. In each test, 5 g of clay were mixed with 100 mL of leachate in 250 mL glass bottles and agitated at 200 rpm on a mechanical shaker for 24 hours at the natural pH of the solutions to simulate realistic environmental conditions. After filtration through 0.45 µm membranes, the residual metal concentrations were measured by ICP-OES and ICP-MS. Adsorption efficiencies were calculated from the difference in metal concentrations before and after treatment, allowing for a comparative assessment of the two clays' potential as low-cost, effective adsorbents for heavy metal removal from battery waste leachates.

3 RESULTS

3.1 Physical Appearance and Chemical Composition

The slag and sludge samples exhibited a wide range of physical appearances, with distinct colours and textures reflecting their varying compositions and origins. The gypsum sludge was light cream in colour and fine in texture, while the five slag samples ranged from dark grey (Grey slag), to reddish-brown (Red-brown slag), to a white-grey hue (White slag). These visual distinctions corresponded to compositional differences revealed by chemical analysis.

Pre-leaching chemical analyses (Table 1) showed that the slag materials were primarily composed of sodium (Na), sulfur (S), and Pb, while the gypsum sludge was dominated by calcium (Ca) and S. Kaolinite and bentonite clays were found to be rich in silica (shown by Si) and aluminium (Al), with minor constituents including magnesium (Mg), iron (Fe), and Ca. X-ray diffraction confirmed that the kaolin sample consisted predominantly of kaolinite, with minor mica and quartz, while the bentonite sample was primarily composed of smectite (montmorillonite) and quartz. Clay identification tests, including glycolation and thermal treatment, verified the swelling characteristics of bentonite and the non-swelling nature of kaolinite.

Table 1: Chemical composition of the sludge and slag expressed in wt%

Elements	Recycle slag	Brown slag	Red-brown slag	Grey slag	White slag	Gypsum Sludge	Kaolinite	Bentonite
Na	4.5	34.5	2.1	20.5	33.8	0.3	-	-
Al	<0.05	0.1	0.9	0.9	<0.05	0.1	11.4	8.1
Ca	<0.05	1.5	0.9	2	<0.05	24.1	<0.05	0.4
Co	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	-	-
Cr	<0.07	<0.05	<0.05	<0.05	<0.05	<0.05	-	-
Cu	0.1	<0.05	<0.05	<0.05	<0.05	<0.05	-	-
Fe	0.6	0.1	1	1.7	<0.05	0.4	0.4	1.8
Mg	<0.08	<0.05	0.3	0.3	<0.05	0.7	0.2	1.8
Mn	<0.05	<0.05	<0.05	0.1	<0.05	0.4	<0.05	<0.05
Ni	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
Pb	94.7	1.5	59.5	37.5	1	0.5	<0.05	<0.05
Si	<0.05	0.8	6.5	3.3	<0.05	0.7	30.0	27.5
Ti	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	0.5	0.1
V	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
Zn	<0.05	<0.05	0.1	<0.05	<0.05	<0.05	<0.05	<0.05
As	12.8	489	<10	237	<10	<10	<10	<10
Carbonate (CO_3)	1.9	3.5	13.7	4.1	0.1	11	-	-
Carbon (organic)	0	3.6	0	1.2	0	0	-	-
Total C	0.4	0	2.8	2	0	2.2	-	-
S	0.1	12	11.2	6.1	17.8	14.5	-	-

3.2 Mineralogical Characterization

Quantitative XRD analysis of the battery waste samples identified several major mineral phases (Figure 1), including halite (NaCl), thenardite (Na_2SO_4), burkeite ($Na_6(CO_3)(SO_4)_2$), litharge (PbO), galena (PbS), gypsum ($CaSO_4{\cdot}2H_2O$), natrite (Na_2CO_3), and calcite ($CaCO_3$). Halite and thenardite were dominant in the White, Brown, and Red-brown slags, whereas galena and natrite were more prevalent in the Grey slag. Litharge and metallic lead were observed in several samples, indicating the presence of both soluble and insoluble lead-bearing phases.

Scanning electron microscopy coupled with EDS provided microstructural confirmation of the XRD results. Backscattered electron imaging (Figure 2) revealed distinct phase boundaries, with lead-bearing phases such as litharge, galena, and lead metal embedded within a heterogeneous matrix. The Red-brown and Brown slags showed well-formed thenardite and burkeite crystals, whereas the Grey slag featured discrete galena grains and sodium-bearing silicates.

Electron probe microanalysis precisely quantified elemental distributions, confirming and refining XRD and SEM-EDS findings. A summary of mineralogical and microstructural variations across the samples is shown in Table 2. Sodium-calcium sulfates (burkeite, thenardite) showed Na_2O from 40–51 wt% and SO_3 from 35–50 wt%, with minor PbO, mainly in White, Brown and Recycle slags. Lead phases are key in these wastes, with anglesite ($PbSO_4$) common in White, Grey, and Recycle slags, exhibiting PbO >60 wt% and intergrown with other sulfates and halite. The Recycle slag uniquely contains litharge (PbO >90 wt%), indicating oxidized lead species from pyrometallurgical treatment and redox variability. Grey slag iron oxides, such as magnetite, contain FeO, reflecting an oxidized environment. The gypsum sludge is distinct, composed almost entirely of gypsum (CaO ~32.5 wt%, SO_3 ~46.7 wt%), confirming its secondary, hydrated nature.

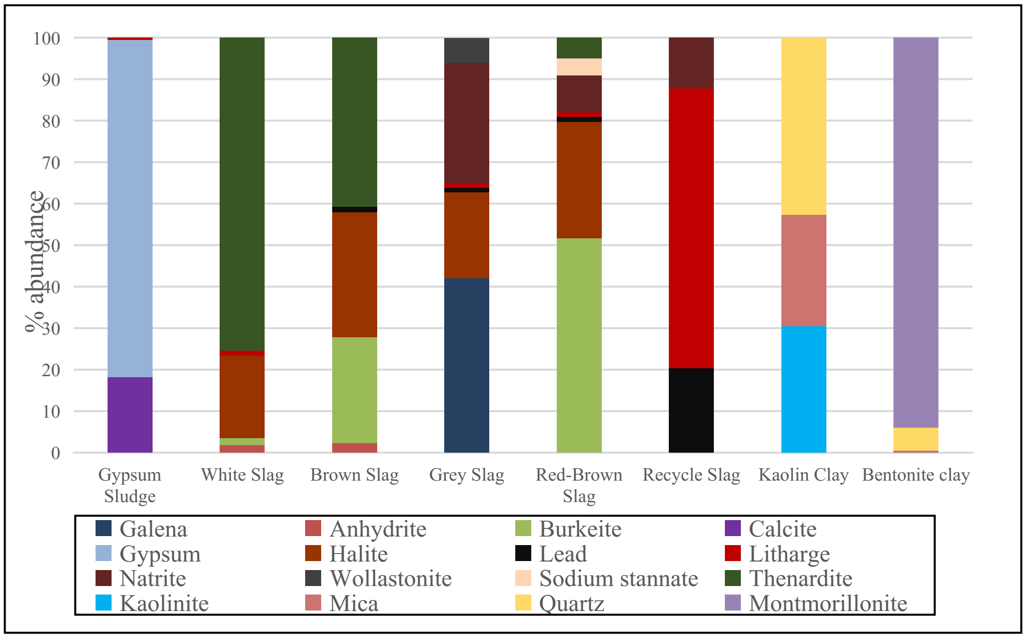

Figure 1: Bulk modal mineralogy of the different slag and gypsum sludge samples, as well as the two clay samples

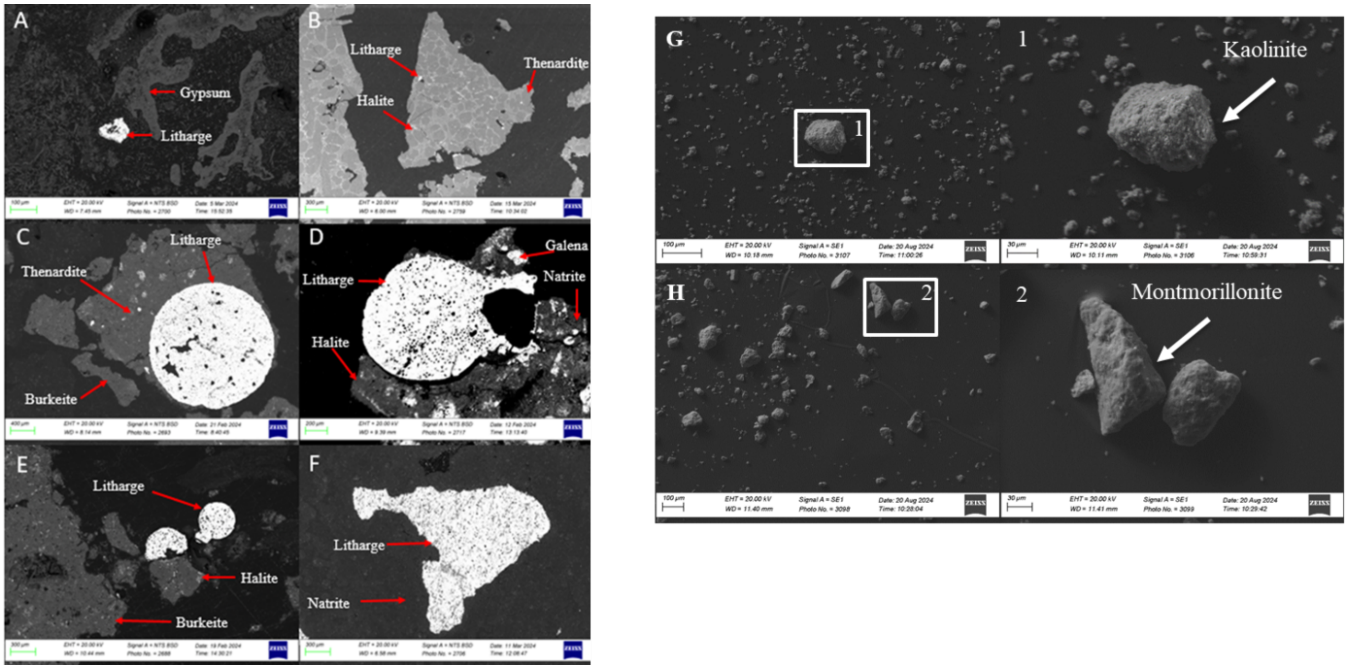

Figure 2: Backscattered electron images showing the various phases present in (A) effluent treatment sludge (B) white slag, (C) brown slag, (D) grey slag, (E) red-brown slag and (F) Recycle slag. Secondary electron images showing the (G1) Kaolin clay sample and (E2) Bentonite clay (eccabond)

Table 2 Summary of Mineralogical Variations Across Samples

Phase	White Slag	Brown Slag	Grey Slag	Red-Brown Slag	Recycle Slag	Gypsum Sludge
Burkeite	Na_2O ~46%, SO_3 ~41%, PbO ~5%	Na_2O ~51%, SO_3 ~44%, PbO ~0.38%	Minor	Na_2O ~49%, SO_3 ~35%, PbO ~0.24%	Na_2O ~40–44%, SO_3 ~42–48%, PbO ~7%	Absent
Thenardite	Minor	Na_2O ~42%, SO_3 ~50%	Minor	Not detected	Minor (Na_2O ~44%, SO_3 ~48%)	Absent
Natrite	Absent	Absent	Absent	Na_2O ~64%, variable Cl	Absent	Absent
Anglesite	PbO >60%, abundant	Minor	PbO ~65.6%, abundant	Minor	PbO >60%, abundant	Absent
Litharge	Absent	Absent	Absent	Absent	PbO >90%, distinct oxide phase	Trace
Portlandite	Absent	CaO ~69%, unique to Brown Slag	Absent	Absent	Absent	Absent
Magnetite	Minor	Minor	FeO-rich (77–80%), abundant	Absent	Minor	Absent
Gypsum	Absent	Absent	Absent	Absent	Absent	CaO ~32.5%, SO_3 ~46.7%, hydrated

This integrated mineralogical characterization combining XRD, SEM-EDS, and EPMA highlights the chemical complexity and microstructural diversity of slags and sludge derived from lead-acid battery recycling. The coexistence of lead sulfate (anglesite) and lead oxide (litharge) phases, variable sodium-calcium sulfates, iron oxides, and hydrated gypsum underscores the diverse thermal and chemical processes involved in battery waste treatment and the resulting environmental implications.

3.3 Leaching Behaviour

The Synthetic Precipitation Leaching Procedure (SPLP) analysis revealed significant metal mobilization under acidic conditions (pH 4.2). Among the samples tested, Recycle slag released the highest concentration of lead (Pb), reaching 609.5 ppm, while Brown slag exhibited the highest arsenic (As) concentration at 154 ppm. Grey slag demonstrated an exceptionally high S release of ~6250 ppm, exceeding the Environmental Protection Agency (EPA) regulatory limits. All slag leachates surpassed the U.S. EPA drinking water standards for Pb and As, which are 0.015 ppm and 0.01 ppm, respectively.

Under neutral pH conditions, water leaching confirmed the continued mobilization of metals. Recycle slag again released significant Pb (981.0 ppm), and Brown slag released high levels of As (153.0 ppm). Grey slag continued to exhibit abnormal sulfur release (6,560 ppm), and elevated concentrations of sodium (Na^+) and potassium (K^+) were recorded in Brown and Red-brown slags, measuring 3.5 g/L and 59 ppm, respectively.

Figure 3 compares the concentrations of Pb, As, and S released during SPLP and water leaching, clearly illustrating the differences in element mobility under acidic versus neutral conditions. The graph highlights that water leaching generally resulted in higher Pb release, while SPLP promoted greater sulfur leaching due to the presence of sulfates, with arsenic showing substantial solubility under both conditions. This visual comparison reinforces the need to assess leaching behaviour under varied environmental scenarios.

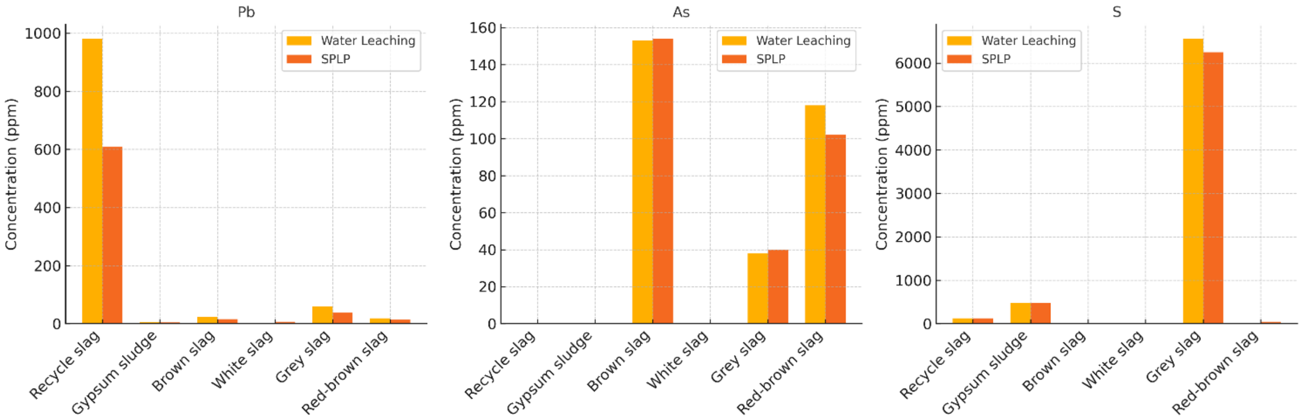

Figure 3: The comparison between SPLP and water leaching for Pb, As and S.

Post-leaching mineralogical transformations, as identified through qualitative XRD, indicated complete dissolution of soluble salts such as halite and thenardite. Galena (PbS) was absent in most leached slags, confirming substantial Pb mobilization. Partial conversion of gypsum to anhydrite was observed in sludge samples. Additionally, secondary mineral formation occurred, including burkeite in Red-brown slag and baryte in Grey slag. Litharge (PbO), a less soluble lead phase, remained present after water leaching.

The stability of clay minerals during SPLP was also assessed. Kaolinite experienced minor Al leaching, with 1.1 wt% reduction (Figure 4), while bentonite showed negligible changes in composition, with <0.34 wt% variation for all measured elements (Figure 5). Leachate analysis further highlighted bentonite's superior chemical stability, as it released significantly lower amounts of Al and Ca, measuring less than 2 ppm compared to kaolinite's 9–20 ppm (Figure 6).

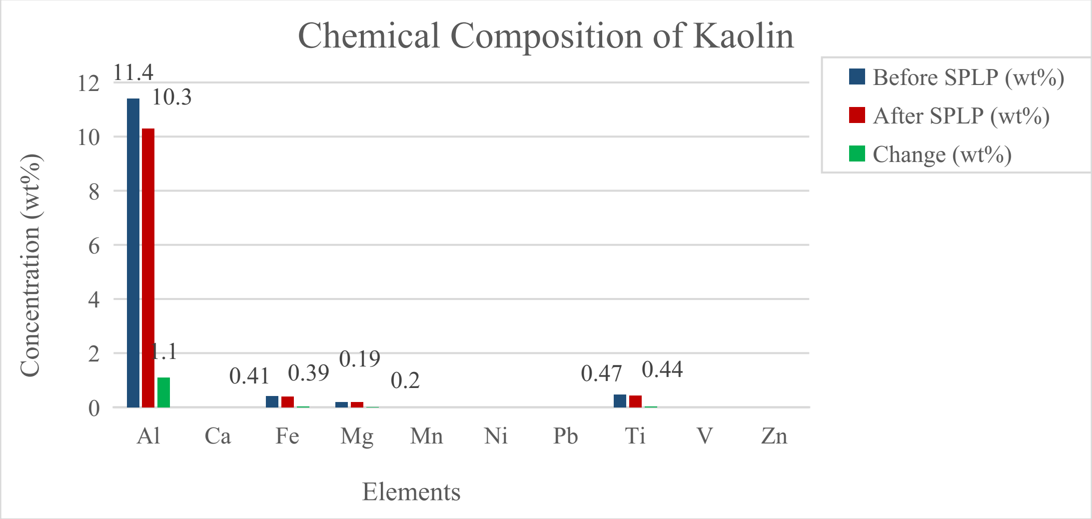

Figure 4: Leaching behaviour of kaolinite clay under acid conditions

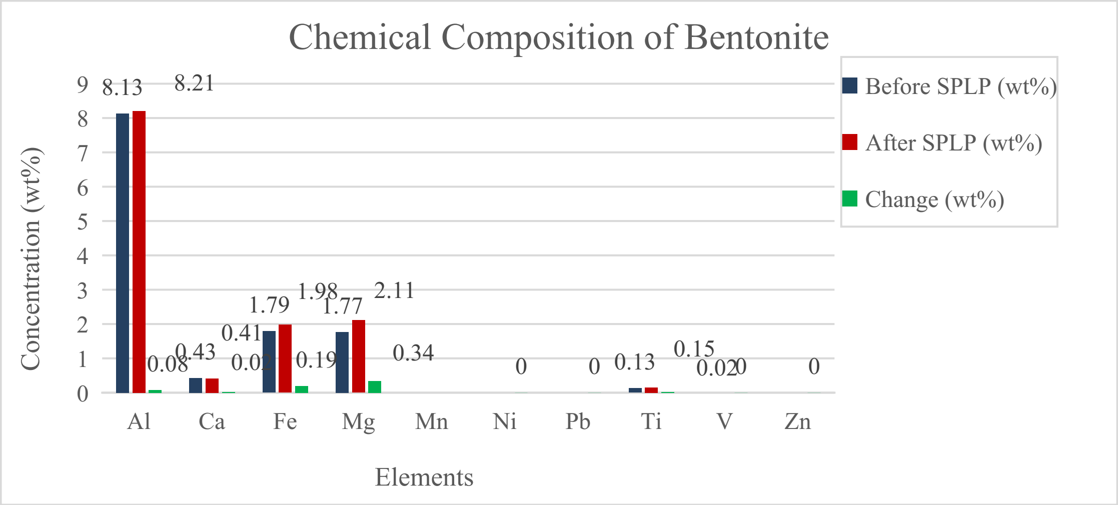

Figure 5: Leaching behaviour of bentonite clay under acid conditions.

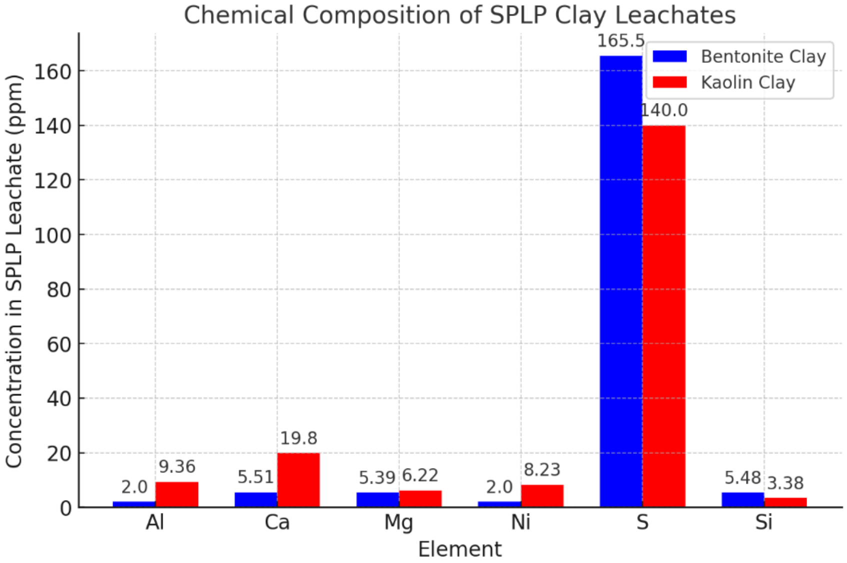

Figure 6: Leachate analysis from clays.

3.4 Performance of Kaolinite and Bentonite in Metal Adsorption

The effectiveness of kaolinite and bentonite clays in adsorbing metals from leachates derived from Recycled slag and Brown slag was assessed. Tables 3 and 4 present the elemental concentrations in the leachates of the Recycled and Brown slags before and after treatment with the respective clays. In both leachates, treatment with kaolinite and bentonite led to a marked reduction in lead (Pb) concentrations.

Table 3: Elemental Concentrations in Recycled Slag Leachate (ppm unless stated)

Element	Initial	+ Kaolinite	+ Bentonite
K	3.06	4.02	6.75
Na (g/L)	0.43	—	8.28
Al	<2	89.95	6.66
As	<2	1.99	4.48
Ca	<2	<2	12.9
Fe	<2	<2	5.16
Mg	<2	<2	11.6
Si	<2	186.5	147
Pb	981	43.75	5.91
Cl^-	<2	82.85	336

The recycled slag leachate showed the most substantial decrease with bentonite treatment, where Pb was reduced from 981 ppm to 5.91 ppm. Kaolinite reduced Pb to 43.75 ppm in the same leachate. For the Brown slag leachate, Pb concentrations were reduced from 22.6 ppm to 1.46 ppm with kaolinite and 2.56 ppm with bentonite.

Arsenic (As) concentrations behaved inconsistently. In the Brown slag leachate, kaolinite treatment resulted in a slight increase in As (153 ppm to 182 ppm), whereas bentonite reduced As to 107 ppm. In the Recycled slag, initial As levels were below detection, but small amounts were measured after treatment.

Several elements, including Al, Si, Na, and Cl^-, showed significant increases after clay treatment—particularly in samples treated with bentonite. These increases suggest possible clay dissolution or ion exchange with the leachate matrix during the adsorption process.

Table 4: Elemental Concentrations in Brown Slag Leachate (ppm unless stated)

Element	Initial	+ Kaolinite	+ Bentonite
K	55.6	48.4	174
Na (g/L)	3.53	49.1	105
Al	<2	18.3	<2
As	153	182	107
Ca	2.84	<2	8.75
Fe	<2	<2	<2
Mg	<2	<2	74.6
S (g/L)	12	14.5	16.3
Si	19.5	9.18	4.71
Pb	22.6	1.46	2.56
Cl^-	<2	14.3	15.2

4 DISCUSSION

The leaching behaviour of LAB recycling wastes revealed critical insights into contaminant mobility under varying environmental conditions. The elevated Pb release (981 ppm) under neutral pH (water leaching) versus acidic SPLP conditions (609.5 ppm) underscores the solubility of lead oxide phases (e.g., litharge) in non-acidic environments, consistent with prior studies on pyrometallurgical slags [8]. Arsenic mobilization (153 ppm) across both leaching regimes highlights its pH-independent solubility, likely due to the presence of arsenate anions (AsO_4^{3-}) that resist protonation [9]. Sulfate dominance in SPLP leachates (up to 17.4 g/L) aligns with the dissolution of sodium sulfates (thenardite, burkeite), exacerbating risks of groundwater salinization.

Bentonite's superior Pb adsorption (99.4% reduction) is attributed to its high cation exchange capacity (CEC) and swelling properties, facilitating interlayer trapping of Pb^{2+} ions [10]. In contrast, kaolinite's lower efficiency (95.5%) reflects its rigid 1:1 lattice and reduced CEC. The paradoxical increase in As concentrations after kaolinite treatment (153 → 182 ppm) suggests competitive desorption: silicate/aluminate leaching (Figure 6) may release pre-adsorbed arsenate or create binding sites for cations (e.g., Na^+), displacing AsO_4^{3-} via anion exclusion [11]. Bentonite's partial As reduction (153 → 107 ppm) confirms its limited affinity for anions, a well-documented constraint of unmodified smectites [12].

Significant post-adsorption releases of Al, Si, and Na^+ (Table 3–4) indicate clay dissolution, particularly problematic for bentonite (Na^+: 0.43 → 8.28 g/L). This compromises long-term structural integrity and poses secondary contamination risks, emphasizing the need for pretreatment (e.g., acid activation) to enhance stability [13]. While bentonite offers a low-cost solution for Pb immobilization in developing regions, its inefficacy against As necessitates complementary strategies—such as iron-oxide modification or co-application with zero-valent iron—to target anionic species [14].

5 CONCLUSION

This study demonstrates that South African bentonite effectively immobilizes Pb (99.4% reduction) in LAB recycling wastes, outperforming kaolinite. However, both clays show limited As adsorption, with kaolinite increasing As mobility due to competitive ion effects. Mineralogical analyses confirm that Pb release is amplified under neutral conditions due to litharge solubility, while As remains highly mobile across pH ranges. Clay dissolution post-treatment (evidenced by Al/Si/Na^+ release) presents practical challenges for long-term deployment. Bentonite otherwise represents a viable, locally sourced adsorbent for Pb containment in battery recycling facilities, advancing circular economy goals in resource-limited settings.

6 ACKNOWLEDGEMENTS

The author gratefully acknowledges MINTEK for financial support provided for this research. Special thanks are extended to the MINTEK team members involved in this work for their invaluable assistance and collaboration throughout the study.

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