

## Valorization of Canadian Bauxite Residue for the Recovery of Strategic Materials

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### Abstract

With the aim of moving toward a sustainable future and building the circular economy, waste valorization is highly motivated. Bauxite residue is the by-product of the Bayer process for alumina production. It contains considerable amounts of rare earth elements (REEs) and refractory metals as well as aluminum and iron, some of which are considered critical materials and initiatives have begun to mine them from secondary sources, such as landfilled industrial process residues. In this study, a two-step process consisting of carbothermic smelting followed by acid baking-water leaching was developed. During carbothermic smelting, iron is separated in the metallic phase and other valuable metals are concentrated in the slag phase and they are leached in the acid baking-water leaching step. Design of experiments methodology was utilized to investigate the effect of the various operating parameters and optimize both processing steps. Furthermore, fundamental investigations were carried out to elucidate the physiochemical mechanisms governing both processing steps. The developed process proves to be a promising technique as the first step of a potential near-zero-waste integrated process for the sustainable valorization of bauxite residue to help build the circular economy.

**Keywords:** Bauxite residue, carbothermic smelting, acid baking-water leaching, rare earth elements, near-zero-waste.

### 1. Introduction

Bauxite residue (BR), the solid by-product of the Bayer process for alumina production, is increasingly viewed less as a problematic waste product, and more as a potential valuable resource for the sustainable recovery of critical materials such as scandium (Sc), rare earth elements (REEs), titanium (Ti), hafnium (Hf), aluminum (Al), iron (Fe), and others. Bauxite residue is an abundant, economical, and sustainable resource because it is produced globally (on all continents except Antarctica) at a rate of 150 million tonnes per year, with 3 billion tonnes stockpiled worldwide. [1–3] Several efforts have been made to develop a method to recover the valuable materials that become enriched in the residue during the Bayer process,[4–6] in particular Sc, which is extremely valuable (US \$4600/kg<sub>Sc2O3</sub>)[7] due to its low rates of worldwide production, and important applications for producing lightweight Al-Sc alloys and solid-oxide fuel cells. However, no valorization strategy has been adopted because the complex mineralogy of the residue makes efficient and economical extraction and purification of the target materials difficult. Furthermore, bauxite residues from different regions are different, depending on the characteristics of the starting bauxite ore, and the specific Bayer process parameters, meaning that no single solution is universally applicable.

A common challenge for the separation and purification of valuable materials from bauxite residue leachates is the separation of scandium and iron, because of the close association between Sc and the Fe-bearing mineral phases. Generally, any direct leaching process that recovers Sc results in co-extraction of Fe.[8] As a result, Sc recovery processes generally require complex solvent extraction, selective precipitation, or ion-exchange approaches. One proposed alternative

route for the removal of iron from the system is a pyrometallurgical carbothermic smelting to reduce the Fe content (+3 oxidation number) in the BR to molten Fe (0 oxidation number), which is immiscible with the liquid slag phase formed by the remaining aluminum, calcium, silicon, and sodium oxides, and it can be recovered as a crude iron product. Most of the target valuable materials (scandium, REEs, titanium, hafnium) partition into the slag phase.

A summary of previous studies examining the application of smelting to the valorization of bauxite residue is given in Table . In most cases, bauxite residue was mixed with carbon and a fluxing agent (calcium/silicon oxides, or other industrial waste products), similar to the blast furnace process for producing metallic iron from iron ore. The carbon is used to reduce the iron, and the flux is used to reduce the temperature and viscosity of the melt, allowing for efficient phase separation. Because the reduced iron and the slag are immiscible both in the molten state and after solidification, the two phases can be readily separated, allowing the production of an iron-depleted slag that can be used for the recovery of the valuable materials via leaching processes.[9]

**Table 1. Previous studies on carbothermic reduction smelting for iron recovery from bauxite residue.**

Reference	BR Source	Smelting temperature (°C)	Smelting duration	Flux composition	Flux amount (wt%)	Carbon amount (wt%)	Additional Notes
Alkan (2017) [10]	Greece	1500-1550	1 h	CaO	10-50	1	Controlled cooling used
Alkan (2018) [11]	Greece	1500-1550	1 h	CaO	20	10	Dry acid digestion of slag (75 °C conc. H <sub>2</sub> SO <sub>4</sub> ), then water leach
Balomenos (2014) [12]	Greece	1580		0.9-1.1 (CaO + MgO): 1 SiO <sub>2</sub> (wt ratio)	33-35	13-20	
Borra (2016) [13]	Greece	1500-1600	1 h	CaSiO <sub>3</sub>	5-40	5	Slag leached with HCl, HNO <sub>3</sub> or H <sub>2</sub> SO <sub>4</sub> at 90 °C
He (2017) [14]	China	1600	35 min	N/A	3-9	25	BR mixed with laterite nickel ore as feedstock
Kaußen (2015) [9]	Germany	1600-1700	100 min	N/A	0	6.25	
Lucas (2018)	Greece	1500	1 h	CaO, MSWI bottom ash*	20	10	MSWI bottom ash

Reference	BR Source	Smelting temperature (°C)	Smelting duration	Flux composition	Flux amount (wt%)	Carbon amount (wt%)	Additional Notes
[15]							used as flux
Rivera (2019) [16]	Greece	1500	1 h	CaO, 1 CaO: 1.5 SiO <sub>2</sub> , 0.8 CaO: 1 SiO <sub>2</sub>	18-25	10	Slag subjected to High pressure acid leaching (H <sub>2</sub> SO <sub>4</sub> or HCl)
Xakalash (2018) [17]	Greece	1500	1 h	CaO, BOF Slag**	30	10	BOF slag used as flux
Yagmurlu (2017) [18]	Greece	1500-1550	1 h	CaO	20	10	Slag direct acid leached
Zeng (2018) [19]	China	<i>Not reported</i>	~ 1 h	N/A	0	25-50	BR mixed with laterite nickel ore as feedstock
Zhang (2018) [20]	China	Pre-reduction: 1250 °C, Smelting: 1500 °C	Pre-reduction: 30 min, Smelting: 20 min	CaO	31	6.6	Magnetic separation of iron

\*MSWI bottom ash: municipal solid waste incineration bottom ash

\*\*BOF slag: basic oxygen furnace (steelmaking) slag

Generally, processes which aim to recover valuable materials from bauxite residue have employed traditional direct acid leaching, [13,18] although some alternative techniques, such as dry digestion,[11] and high-pressure acid leaching [16] have been investigated. It has been shown that acid baking–water leaching (ABWL), a combined pyro-hydrometallurgical process, can be used to enhance the extraction of scandium and other valuable materials from bauxite residue.[21]

In acid baking–water leaching, bauxite residue is mixed with concentrated sulfuric acid, then baked at moderately high temperatures (200–400 °C) to convert the minerals to more-soluble sulfate phases, which can subsequently be readily leached at ambient conditions (1 atm, 25 °C) in water. The baking step is a crucial part of this process, allowing for enhanced extraction, in particular for titanium and hafnium extraction, when compared with the leaching of bauxite residue which has been mixed with acid at room temperature without additional treatment.[21] The primary disadvantage of direct acid baking–water leaching of bauxite residue is that the iron content of the feedstock is strongly extracted, the effect which allows for high (up to 80%) extraction of scandium.

In this work, bauxite residue was first smelted to separate the iron content as the metallic phase and the slag that is enriched with valuable materials was processed with acid baking–water leaching to extract scandium and other valuable materials.

## 2. Materials and Methods

### 2.1. Materials and Analysis

Bauxite Residue samples were sourced from Rio Tinto Vaudreuil alumina plant located in Jonquière, Quebec, Canada. The crystal structure of bauxite residue was characterized by XRD and it was found that it contains hematite (Fe<sub>2</sub>O<sub>3</sub>), goethite (FeO(OH)), boehmite (AlO(OH)), aluminum hydroxide (Al(OH)<sub>3</sub>), synthetic titanite CaTiOSiO<sub>4</sub>, anatase (TiO<sub>2</sub>), quartz (SiO<sub>2</sub>), and sodalite (Na<sub>6</sub>(Al<sub>6</sub>(Si<sub>5.5</sub>(H<sub>4</sub>)<sub>0.5</sub>)O<sub>24</sub>)(NaCl)<sub>0.4</sub>(NaOH)<sub>0.7</sub>). To determine the elemental composition of the smelting slags, they were digested by alkali fusion in 49.75 wt% Li<sub>2</sub>B<sub>4</sub>O<sub>7</sub>: 49.75 wt% LiBO<sub>2</sub>: 0.50 wt% LiBr flux at 1050 °C, followed by dissolution in 19 wt% HCl. The digest solution compositions were assessed inductively coupled plasma optical emission spectroscopy (ICP-OES), the results of which are presented in Table 2.

**Table 2. Compositional and morphological characteristics of dry bauxite residue.**

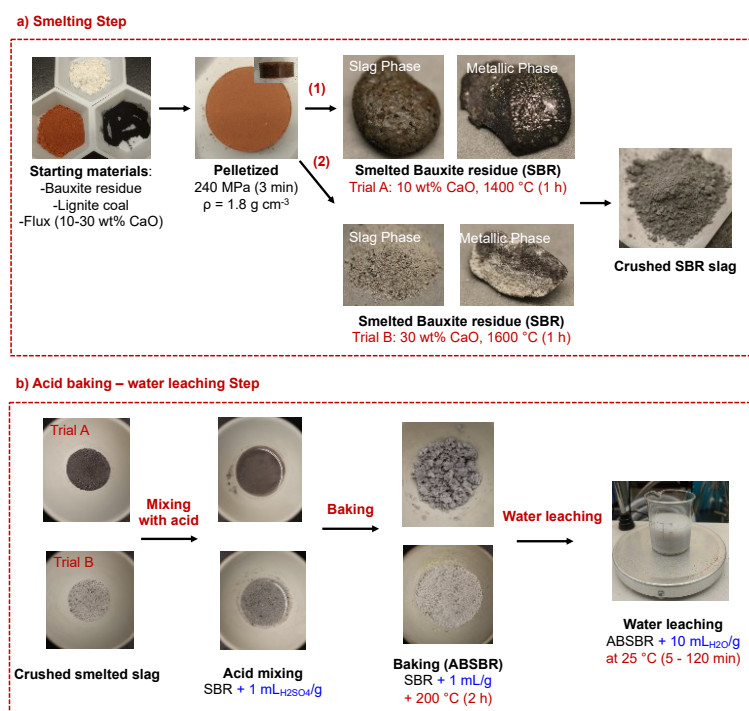
Rare earth elements			Refractory metals			Bulk materials		
Element	mg/kg <sub>DBR</sub>	std	Element	mg/kg <sub>DBR</sub>	std	Element	wt%	std
Sc	31.1	±0.9	Ti	38,000	±0.1	Fe	24.3	±1.0
Y	59.1	±2.5	Nb	106.0	±8.7	Al	11.8	±0.4
Ce	101.0	±8.3	Zr	2171	±5.5	Ca	2.3	±0.1
La	52.3	±0.4	Hf	60.7	±4.5	Na	5.2	±0.2
Nd	39.9	±1.3				Si	6.1	
Sm	10.0	±0.3						

The smelting fluxes were prepared as CaO powder and crushed lignite coal was used as the carbon source for carbothermic reduction. Concentrated (95-98%) H<sub>2</sub>SO<sub>4</sub> was used for acid baking, and deionized water (0.055 µS) was used for water leaching.

### 2.2. Smelting- Acid Baking-Water Leaching (ABWL) Process

The smelting-ABWL process is illustrated in Figure . The dried and crushed bauxite residue was first mixed with carbon (10 wt% relative to BR), and with the flux (10 – 30 wt% relative to BR). This mixture was pelletized at 240 MPa for 3 min to achieve a pellet with an apparent density of 1.8 g/cm<sup>3</sup>. The pellets were smelted at 1400 to 1600 °C for 1 h in graphite crucibles in an Argon atmosphere. The resulting slag and metal phases were then manually separated readily, and the slag phase was crushed for further processing.

The crushed slag phase was then mixed with concentrated sulfuric acid in porcelain crucibles and baked at 200 °C for 2h in air. The resulting acid-baked slag was then leached in water at ambient conditions (1 atm, 25 °C) for 120 min, with sampling at 5, 30, and 120 min for analysis.



**Figure 1. Illustration of the a) carbothermic reduction smelting and b) acid baking - water leaching process. The primary steps of each process are illustrated with accompanying photographs and associated processing conditions.**

Note: part a) and part b) depict two different samples that were smelted at different conditions.

### 3. Results and Discussion

#### 3.1. Bauxite Residue Smelting

To assess the ability of carbothermic reduction smelting to concentrate target elements in the slag phase and to maximize the separation of iron from the other elements, bauxite residue smelting trials were performed at two sets of conditions, as described in Table 3. These operating conditions were selected on the basis of previous literature (Table ), thermodynamic simulations, and preliminary trials. It should be noted that a higher temperature was used for trial B because it was found that phase separation could not be achieved at 1400 °C with the pellet composition selected for trial B (30 wt% CaO flux, 10 wt% C).

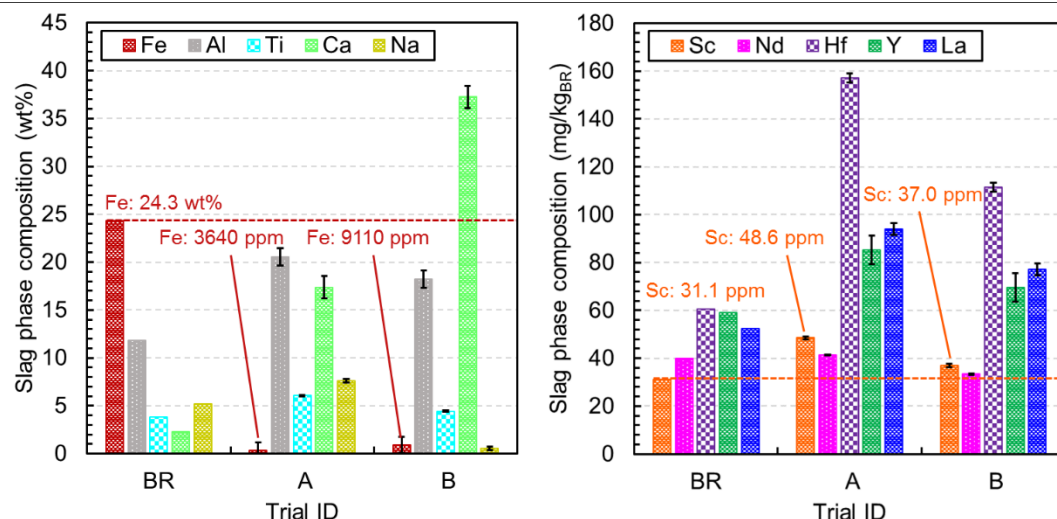
**Table 3. Operating parameters for the bauxite residue smelting trials.**

Condition set	Smelting temperature (°C)	Smelting duration	Flux composition	Flux amount (wt%)	Carbon amount (wt%)	Smelting atmosphere (L/min)
A	1400	1 h	100% CaO	10	10	Ar: 1.42
B	1600	1 h	100% CaO	30	10	Ar: 1.42

As shown in Figure a, the slag and metallic phases fully separated into distinct phases, which upon cooling, were readily separated from each other. For smelting conditions in trial A, the slag phase comprised a single porous and non-magnetic bead. In contrast, for smelting conditions in trial B, the cooled slag formed a loose non-magnetic powder (as shown in Figure 1).

The composition of the slag phases obtained from both smelting conditions were assessed by digesting the samples by alkali fusion, and elemental quantification by ICP-OES (Figure ). This

quantification demonstrated that during the smelting process, almost all Fe content of the starting BR is separated, yielding a nearly Fe-free slag phase composed of Al, Ca, Ti, and Si (not quantified by ICP-OES), which is slightly enriched in Sc and other valuable materials, in particular Hf.

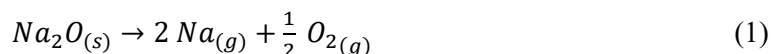


**Figure 2. Elemental composition of the two slag phases of the smelted bauxite residue.**

For comparison, the elemental compositions of un-smelted bauxite residue, as reported in Anawati & Azimi (2019)[21] are shown. The compositions of Fe and Sc are highlighted. The error bars represent the estimated standard deviation ( $n = 3$ ) based on a repeatability study.

Although the Sc enrichment factor is relatively small due to the dilution effect of adding flux to the system in addition to the other slag-forming components of the residue, the primary advantage of the smelting step is that the difficult-to-separate iron, which represents 24 wt% of the feedstock, was nearly completely removed from the other elements. In addition, the iron is recovered as a crude metallic product, which can be directly sold for use in steelmaking.

A notable difference between trials A and B is that the concentration of most of the valuable materials is reduced in trial B due to the dilution effect of using more flux; however, the sodium (Na) content of trial B is considerably lower than that of trial A. This decrease in Na content can be attributed to the relatively high volatility of Na salts (NaOH) and oxides (Na<sub>2</sub>O) at high temperatures. It is known that solid Na<sub>2</sub>O sublimates to atomic sodium vapour (Na) and oxygen gas (O<sub>2</sub>), according to reaction 1.[22]



As such, these results potentially indicate that sodium volatilization can be controlled by manipulating the smelting temperature – a consideration that could potentially have safety, corrosion, and environmental considerations at the industrial scale.

### 3.2. Acid Baking–Water Leaching of Smelted Bauxite Residue

Based on our previous study which demonstrated considerable extraction efficiency and advantages of acid baking–water leaching over conventional direct acid leaching for the recovery of scandium and other elements from bauxite residue,[21] ABWL of the BR smelting slag phases was investigated to achieve high extraction efficiencies with rapid leaching kinetics.

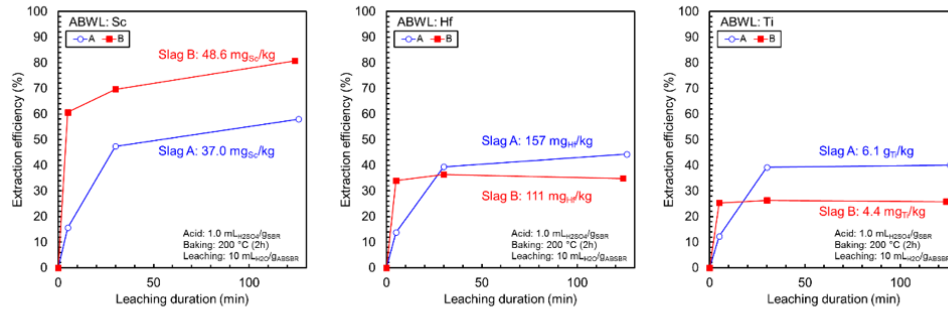
In these trials, the slag phases were crushed and fed into the ABWL process. Photographs of the slag before and after acid baking are presented in Figure b. In the contrary to what was observed when un-smelted BR was mixed with H<sub>2</sub>SO<sub>4</sub>, upon mixing with the sulfuric acid, the slag did not experience a strong exothermic reaction nor turned into a thick paste. After baking at 200 °C, which is below the boiling temperature of H<sub>2</sub>SO<sub>4</sub>, the slag-acid mixtures were converted to dry solids, and a considerable volume expansion was observed.

The acid baked slag was then water leached to assess the feasibility of this process for valuable material extraction (Figure ). Similar to our previous study on un-smelted BR,[21] the leaching kinetics of the slag baked at 200 °C was fast, with the majority of Sc, Hf, and Ti extraction occurring within the first 30 min of leaching. In particular, these elements were primarily extracted within the first 5 min for slag of trial B. In comparison, co-extraction of residual Fe, the primary problematic contaminant, followed slower leaching kinetics, suggesting that the valuable materials can be selectively leached by limiting the leaching duration.

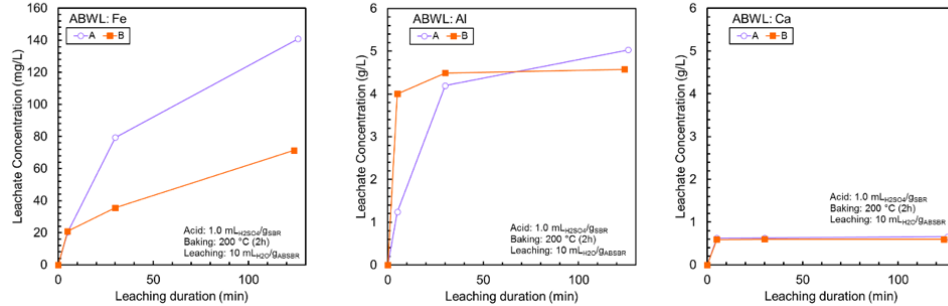
Scandium was extracted more efficiently from slag B than slag A, achieving 60.7 % extraction (0.87 mg<sub>Sc</sub>/L) after 5 min, and 80.8 % extraction (1.2 mg<sub>Sc</sub>/L) after 2 h. In addition, co-extraction of Fe was reduced for slag B relative to slag A. The Fe contamination of the leachate was only 20.9 mg<sub>Fe</sub>/L (6.0 % extraction) after 5 min, and 71.4 mg<sub>Fe</sub>/L (20.3 % extraction) after 2 h. For comparison, ABWL of un-smelted BR under similar conditions resulted in 53% Sc extraction (0.71 mg<sub>Sc</sub>/L), with a residual Fe concentration of 5,331 mg<sub>Fe</sub>/L.

The other major soluble slag components, i.e., Al and Ca, generally leached rapidly (within 5 to 30 min), reaching their solubility limit. Al reached concentration of 4.6-5.0 g<sub>Al</sub>/L and Ca reached considerably lower concentration at 599-660 mg<sub>Ca</sub>/L. In comparison with the direct acid leaching of BR, these relatively low concentrations of contaminating bulk materials would facilitate the separation and recovery of Sc from the leachate, which can be achieved by selective precipitation,[23] solvent extraction,[24] ion-exchange,[25] or other methods.

**a) Valuable material extraction efficiencies**



**a) Bulk material leachate concentrations**



**Figure 3. Water leaching extraction of valuable and matrix elements from acid baked smelted BR.**

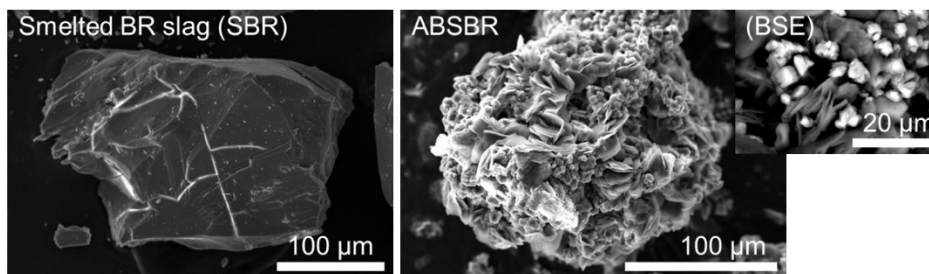
The following ABWL parameters were used: acid to BR ratio = 1.0 mL<sub>H<sub>2</sub>SO<sub>4</sub></sub>/g<sub>BR</sub>, solid to liquids ratio = 10 mL<sub>H<sub>2</sub>O</sub>/g<sub>ABSBR</sub>, leaching temperature = 25 °C, agitation rate = 400 rpm.

### 3.3. Mechanistic Investigation of the Acid Baking Mechanism

To gain additional insight into the mechanism of acid baking for the smelted bauxite residue slag, secondary electron microscopy energy dispersive spectroscopy (SEM-EDS) was utilized to observe the changes in the microstructure and phase composition that occur during the acid baking step.

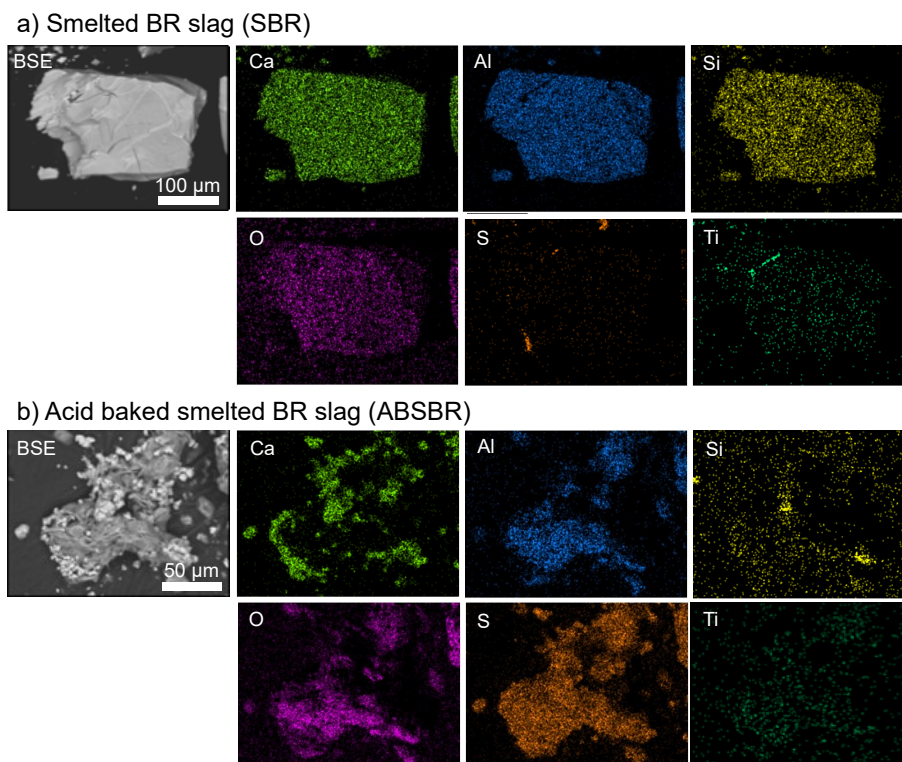
The distinct change in macroscale physical appearance during acid baking was accompanied with a distinct change in microscale morphology, as shown in Figure . Prior to acid baking, the BR slag (smelting condition: B) consisted of large flat particles with near-zero porosity; however, after acid baking, the morphology shifted to smaller particles with considerable surface roughness, high porosity, and surface granules of distinct composition from the bulk particle (as shown in the backscattered electron image (BSE)). These morphological changes suggest that acid baking increases the available surface area for the subsequent water leaching step.

Elemental mapping of the particles before and after the acid baking process (Figure ) revealed that prior to acid baking, the smelted BR particles were relatively homogenous, corresponding to a Ca-Al-Silicate phase; however, after acid baking, the uniform particles appeared to have been separated into individual Ca, Al, and Si-bearing phases that do not fully co-localize. The elemental mapping results suggest that the acid baking process breaks down the homogenous slag particles into separate phases, which would potentially release any valuable materials trapped within the slag crystal lattice, making them available at or near the particle surface to be leached-away during water leaching.



**Figure 4. Morphology of smelted BR slag (SBR) before and after acid baking (ABSBR: SBR + 1 mL<sub>H<sub>2</sub>SO<sub>4</sub></sub>/g<sub>SBR</sub> + 200 °C (2 h)).**

The morphology was observed by SEM in the secondary electron mode. The inset panel on the ABSBR sample shows a magnified back scattered electron image (BSE) of the acid baked SBR sample.



**Figure 5. EDS elemental mapping of the smelted BR slag (SBR) a) before and b) after acid baking (ABSBR: SBR + 1 mLH<sub>2</sub>SO<sub>4</sub>/gSBR + 200 °C (2 h)).**

The morphology and composition were characterized using SEM-EDS.

#### 4. Conclusions

In this study, it was demonstrated that carbothermic reduction smelting process can be utilized to separate Fe from the bulk bauxite residue and to concentrate Sc and other valuable elements in the slag phase. Acid baking-water leaching can then be utilized to extract the valuable materials from the slag phase. This study consisted of a preliminary investigation of the “smelting-ABWL process”, which demonstrated its feasibility for valorizing bauxite residue. Work is currently underway to study the specific effects of different smelting and ABWL process parameters, such as smelting temperature, flux and carbon composition, acid-SBR ratio, baking temperature, and water-ABSBR ratio, to gain a fundamental understanding of the underlying physicochemical mechanisms, and to achieve an economically and operationally optimal set of process parameters. The ultimate goal of this research is to develop a near-zero-waste bauxite residue valorization process to meet modern society’s demand for several critical materials, while reducing dependence on environmentally damaging primary extraction processes, exploiting an abundant and readily available resource, and reducing the accumulation of bauxite residue in landfills.

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