

Article

Extraction of Alumina and Alumina-Based Cermets from Iron-Lean Red Muds Using Carbothermic Reduction of Silica and Iron Oxides

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Abstract

A novel strategy has been developed for extracting value-added resources from iron-lean, high-alumina- and -silica-containing red muds (RMs). With little or no recycling, such RMs are generally destined for waste dumps. Detailed results are presented on the carbothermic reduction of 100% RM (29.3 wt.% Fe₂O₃, 22.2 wt.% Al₂O₃, 20.0 wt.% SiO₂, 1.2 wt.% CaO, 12.2 wt.% Na₂O) and its 2:1 blends with Fe₂O₃ and red mill scale (MS). Synthetic graphite was used as the reductant. Carbothermic reduction of RM and blends was carried out in a Tamman resistance furnace at 1650 °C for 20 min in an Ar atmosphere. Reduction residues were characterized using scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS), elemental mapping and X-ray diffraction (XRD). Small amounts of Fe₃Si alloys, alumina, SiC and other oxide-based residuals were detected in the carbothermic residue of 100% RM. A number of large metallic droplets of Fe–Si alloys were observed for RM/Fe₂O₃ blends; no aluminium was detected in these metallic droplets. A clear segregation of alumina was observed as a separate phase. For the RM/red MS blends, a number of metallic Fe–Si droplets were seen embedded in an alumina matrix in the form of a cermet. This study has shown the regeneration of alumina and the formation of alumina-based cermets, Fe–Si alloys and SiC during carbothermic reduction of RM and its blends. This innovative recycling strategy could be used for extracting value-added resources from iron-lean RMs, thereby enhancing process productivity, cost-effectiveness of alumina regeneration, waste utilization and sustainable developments in the field.

Keywords: red mud; industrial waste; alumina regeneration; recycling; cermets; ferro-silicon alloys



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1. Introduction

Aluminium and its alloys, being lightweight with high mechanical strength and corrosion resistance, are used extensively as structural materials in the aviation, transport and construction sectors, machinery parts, packing, food wraps, beverage cans and others. The energy consumption during aluminium recycling (approximately 8.3 GJ/tonne) is about 5% of the energy required during primary production (186 GJ/tonne) [1,2]. In addition to energy savings, recycling and sustainable management of aluminium and aluminium-based products contribute significantly to reductions in associated greenhouse gas emissions.

Aluminium production involves two key processes, namely the Bayer process for refining bauxite ore to obtain alumina (aluminium oxide), and the Hall–Heroult electrolytic process for the smelting of alumina dissolved in cryolite to produce pure aluminium metal [3]. Discovered by Karl Bayer in 1887, the industrial Bayer process [4] has remained virtually unchanged over time, and nearly all the world’s alumina supply for aluminium production (over 140 million tonnes in 2024) is derived using this process [5]. Bauxite ore is ground finely in mills and mixed with hot caustic soda to dissolve alumina, and later cooled in a series of flash tanks. Leftover solid impurities settle down as a fine red mud (RM), which is also known as bauxite residue. Producing one tonne of alumina consumes around 2–3 tonnes of bauxite and generates around 0.4–2 tonnes of red mud [6]. The average global production of RM is around 1.3 tonnes per tonne of alumina [7]. The composition and volumes of RMs can vary significantly depending on the bauxite ore and processing conditions; sustainable management of red mud waste is presently a significant global challenge [8].

Based on the crystal structure and the number of water molecules of hydration, there are three main types of bauxite ores: gibbsite ($\alpha\text{-Al}_2\text{O}_3 \cdot 3\text{H}_2\text{O}$), böhmite ($\alpha\text{-Al}_2\text{O}_3 \cdot \text{H}_2\text{O}$), and diasporite ($\beta\text{-Al}_2\text{O}_3 \cdot \text{H}_2\text{O}$) [9]. Gibbsitic ores are found in several countries such as Australia, Brazil, Guinea, India, Jamaica, Surinam, Guyana, Saudi Arabia, Vietnam, Venezuela, etc. Most of the bauxite ores in China and Russia are böhmitic and diasporic ores; these typically have high contents of alumina (A) and silica (S), and low (A/S) ratios [10]. The Bayer process is generally used for bauxite ores with (A/S) ratios above 9, whereas the sinter process is used for ores with (A/S) ratios below 7 [11,12].

Major operating costs for producing aluminium involve costs associated with processing bauxite ores, fuel and the management of waste generated, including RMs [13]. Three categories of RMs include Bayer RM, sintering RM and combined RM, with the Bayer process accounting for nearly 90% of RM production [14]. Chemical compositions of RMs generated using the Bayer process and sintering techniques from a bauxite refining plant (Guizhou, China) are presented here for the sake of comparison. The Bayer RM contained: Fe_2O_3 : 26.4 wt.%; Al_2O_3 : 18.9 wt.%; CaO : 21.8 wt.%; SiO_2 : 8.52 wt.%; TiO_2 : 7.4 wt.%, whereas sintered RM contained: Fe_2O_3 : 7.9 wt.%; Al_2O_3 : 17.3 wt.%; CaO : 40.2 wt.%; SiO_2 : 17.3 wt.%; TiO_2 : 7.4 wt.% [15,16]. Broadly speaking, key constituents of RMs can range as follows: “ Fe_2O_3 (26.6–46 wt.%), Al_2O_3 (15–21.2 wt.%), SiO_2 (4.4–18.8 wt.%), CaO : (1–22.2 wt.%), Na_2O (1–10.3 wt.%), TiO_2 (4.9–21.2 wt.%)” [17,18].

The loss of alumina in the red mud has a significant impact on the productivity of the Bayer process and aluminium making. In this article, we focus our attention on the recovery of metallic constituents and alumina from red muds. Extensive research has been carried out on the extraction of aluminium and iron from RMs using physical beneficiation [19,20], pyrometallurgy, carbothermic reduction at high temperatures [21,22], hydrometallurgy, acid leaching [23,24] and others. Using various acids, e.g., hydrochloric acid, nitric acid, oxalic acid, sulfuric acid or combinations of various acids, hydrometallurgy is an effective route for de-alkalizing RMs and recovering aluminium and iron [25].

As direct acid leaching of aluminium and iron from RMs can become very challenging in the presence of complex polymetallic minerals [26], alternative strategies are becoming increasingly important. Extraction of iron particles through magnetic separation is considered very difficult as these tend to be wrapped by the gangue [27]; the formation of $\text{FeO} \cdot 2\text{Al}_2\text{O}_3$ and $\text{FeO} \cdot \text{Al}_2\text{O}_3$ complexes is likely to result in high-alumina-grade iron concentrates [28]. High-temperature carbothermic reduction of iron oxide to iron has been used to recover metallic iron, with alumina present in the RM precipitating out in the form of a slag [7,29]. Alkali sintering has been used to extract alumina from RMs; Na_2CO_3 and CaO were used at elevated temperatures to produce sodium aluminates followed by alkaline leaching [30].

The recovery of iron is one of the hottest research areas in RM utilization especially for RMs containing high concentrations of iron oxide (>50 wt.%) [31]. The extraction of iron from RMs has been investigated extensively and reported in several excellent reviews [32–34]. However, most of the studies were carried out on iron-rich RMs, and little attention was paid to iron-lean RMs, especially those rich in alumina and silica. Reductive roasting (900–1350 °C; up to 100 min) of iron-lean (27.49 wt.% Fe) RM and blast furnace dust blends were shown to produce direct-reduced iron and functional ceramsite [35]. Blends of iron-lean RMs with phosphogypsum and Na_2SO_4 have been used to extract iron values from various RMs [36]. Nearly 70% of RMs generated belong to the iron-lean categories; these tend to be dumped in RM reservoirs or storage tanks most of the time. A few representative examples of such iron-lean RMs are given in Table 1 from sources around the globe.

Table 1. Representative examples of several iron-lean RMs from across the globe. The levels (wt.%) of only four key oxides are provided in the table.

Country	Fe_2O_3	Al_2O_3	SiO_2	CaO	Reference
India	28.1	21.9	7.5	10.2	Hindalco Renukoot [37]
	27.9	19.4	7.3	11.8	BALCO Korba [37]
	24.5	24.3	6.2	-	INDAL Muri [37]
Turkey	36.9	20.4	15.7	2.2	SAP Konya [38]
	35.0	20.2	13.5	5.3	SAP Konya [39]
China	13.7	7.0	18.1	42.2	Shandong [40]
	11.8	25.5	20.6	14.0	Henan [41]
	6.8	10.1	22.2	42.3	Shanxi [41]
Korea	16.6	23.7	22.9	6.7	Korea Chemical [42]
Italy	15.2	24.7	18.6	4.2	Eurallumina [43]
France	26.6	15.0	5.0	22.2	[44]
Canada	31.6	20.6	8.8	1.6	ALCAN [45]
Australia	29.6	17.3	30.0	3.6	ALCOA [46]
	28.5	24.0	18.8	5.3	ALCOA [46]

Overall recycling rates for RMs continue to remain quite low (about 10% or less) globally. A staggering 4.6 billion tonnes of RM have accumulated worldwide [47]. China is the largest producer of aluminium in the world, as well as the largest generator of red mud waste [48]. For example, China produced more than 100 MT of RM in 2023; the average utilization rate was found to typically range between 4 to 8% (less than 10 MT) [49]. The rest were stockpiled in various storage facilities; the stockpiled RM is well known to be hazardous to the surrounding soil, water resources and the atmosphere [23]. Large volumes of accumulated stores, high generation rates and limited utilization of RM waste pose a formidable challenge to the aluminium industry [50]. Developing novel recycling routes for RMs, therefore, assumes great importance for the environmental sustainability of the industry.

Our group has been working extensively on several RM processing routes, including smelting reduction and solid-phase reduction followed by magnetic separation [51–54]. Each route has its own merits and demerits. While smelting reduction requires higher temperatures, it can provide precise control over the compositions of reaction products and produce tailored slags using fluxing additives. Although solid-phase reduction with magnetic separation is less energy-intensive, it is unable to produce high-quality iron concentrates with low phosphorus and sulphur contents. Effective magnetic separation of reduced iron necessitates additives (Na_2CO_3 , Na_2SO_4 , feldspar, etc.) that would increase processing costs while degrading product quality. Hydrometallurgical routes involving leaching, solvent extraction and precipitation would require multiple process stages and steps for the extraction of individual elements [55]. In this study, co-smelting of RM with additives rich in iron oxides was the preferred choice for achieving effective control over reaction products and extracting multiple elements.

Aims of the Investigation

In this article, we present an innovative strategy for extracting value-added materials such as alumina, alumina-based cermets and ferro-silicon alloys from iron-lean RMs rich in both alumina and silica. These RMs, currently unsuitable for subsequent alumina extraction through repeated rounds of the Bayer process, will normally be dumped in storage tanks, with significant amounts of alumina being lost and wasted. Using a novel approach, this study aims to extract valuable alumina- and silica-containing commodities from iron-lean RMs. Novel fundamentals being developed are expected to enhance resource recovery, industrial productivity and environmental sustainability together with reductions in waste volumes in this sector.

2. Materials and Methods

2.1. Materials and Blend Compositions

The red mud used in this investigation was sourced from ‘Aluminium of Kazakhstan JSC’, Pavlodar, Kazakhstan. Blends of RM with iron resources such as Fe_2O_3 and red mill scales (MS) were prepared in a 2:1 proportion to increase the net iron content of the blend for extracting aluminium-based constituents, diluting harmful impurities (e.g., phosphorus, alkalis), and for enhancing overall reaction kinetics. Treating high-purity Fe_2O_3 as a reference standard, this study will investigate the recycling behaviour of red MS and RM blends. The co-smelting of RM and MS will enable the simultaneous utilization of two industrial wastes, produce value-added products and provide economic and environmental benefits.

Red MS is composed of very fine particulates of Fe_2O_3 , typically 20–80 nm, and has significantly higher reactivity compared to the typical micron-sized particles of Fe_2O_3 due to significant differences in respective surface areas [56]. The impurity levels in both additives were kept to a bare minimum in order to keep the concentrations of other oxides under control. Compositional details of RM and the two blends are presented in Table 2. This RM has low iron values but contains large amounts of both alumina and silica (A/S of 1.1).

Table 2. Chemical compositions of the RM and its blends (wt.%) with iron oxide and red MS.

Blends	Fe_2O_3	Al_2O_3	SiO_2	CaO	Na_2O	TiO_2
RM	29.3	22.2	20.0	1.2	12.2	3.4
20 g RM + 10 g Fe_2O_3	53.0	14.9	13.4	0.8	8.2	2.3
20 g RM + 10 g red MS	64.6	11.1	10.0	0.6	6.1	1.7

2.2. Experimental

Synthetic graphite was used as the reductant in these investigations: 8 g of graphite was added to 30 g of RMs/various blends prior to heat treatments and mixed thoroughly. The amount of graphite reducing agent was determined based on preliminary experiments. About 10 g of blended mixtures were loaded into graphite crucibles. The heat treatments were carried out simultaneously on three crucibles by loading these into a container, which significantly reduces the time taken to complete various studies at high temperatures along with reducing overall energy consumption during experimental investigations. Additional information on furnace details and operation can be found in one of our earlier publications [57]. The furnace was heated with a heating rate of 10–20 °C/min to 1650 °C, continuously purged with argon (0.5 L/min). The temperature was measured with the help of a W-Re thermocouple placed within the large container. This temperature was chosen to ensure complete reduction of silica and subsequent assimilation of Si into the metallic phase. Such high temperatures have been used previously during the industrial production of pig iron and alumina cement [58]. Heat treatment was carried out for 20 min, after which the furnace was switched off. The experimental assembly was extracted from the furnace after the furnace had cooled down. Representative examples of some of the reacted assemblies after the heat treatments are shown in Figure 1. Several shiny metallic particles can be seen clearly in various images of residues and graphite crucibles.



Figure 1. Representative examples of heat-treated RM and its blends after carbothermic reduction at 1650 °C for 20 min.

Detailed characterization of the reaction products was carried out using a variety of analytical techniques such as SEM, EDS, and XRF using Tescan Vega 3 (TESCAN, Brno, Czech Republic, with an Oxford instruments EDS detector). Specimens were carbon coated prior to analysis and a number of SEM/EDS observations were made on close-lying points for determining average values. The statistical deviation in EDS measurements ranged between ± 0.1 – 0.4 wt.%. X-ray diffraction studies were carried out on a Difrey 401 diffractometer, Scientific Instruments, Russia (Cu K α radiation; 45 KV; 40 mA; angular range: 10–90°; step size: 0.1°; time step: 5 s). All experiments and analytical investigations were repeated a minimum of three times to ensure overall accuracy and reproducibility.

3. Results

3.1. Set A: Carbothermic Reduction of Iron-Lean RM

Detailed results are presented in Figures 2–6 on the iron-lean, high-alumina- and high-silica-containing RM after carbothermic reduction at 1650 °C for 20 min; 8 g of graphite was used as the reductant for 30 g of RM. Figure 2 shows a low-resolution SEM image (400×) of the reaction product after the carbothermic reduction of RM. There are four key features of this image. The black background shows residual carbon left behind after the heat treatment. An excess amount of carbon was used in this study to ensure that the reactions were not limited by the availability of carbon. There are a large number of grey refractory particulates (labelled as ‘A’) in a range of shapes and sizes. A large number of metallic particulates (‘C’) can be seen distributed throughout the matrix. Their sizes range from very small to medium, and their shapes from spherical to oddly shaped as well. A few metallic droplets contained dark grey precipitates within their boundaries (‘B’). Detailed high-resolution SEM/EDS images of complex regions are presented in Figures 3–5.

A high-resolution image (1000×) of the region ‘B’ (Figure 2), a non-spherical metallic region with grey-coloured precipitates on the outer edges and inner cross-sections, is shown in Figure 3. Grey regions are primarily slags containing oxides of Al, Ca, Si, Fe, Mg, etc. The aluminium content in these slags was quite high, ranging between 39.6 and 42 wt.%. Si contents were between 1.9 and 2.1 wt.%. Other oxides were present in minor concentrations.

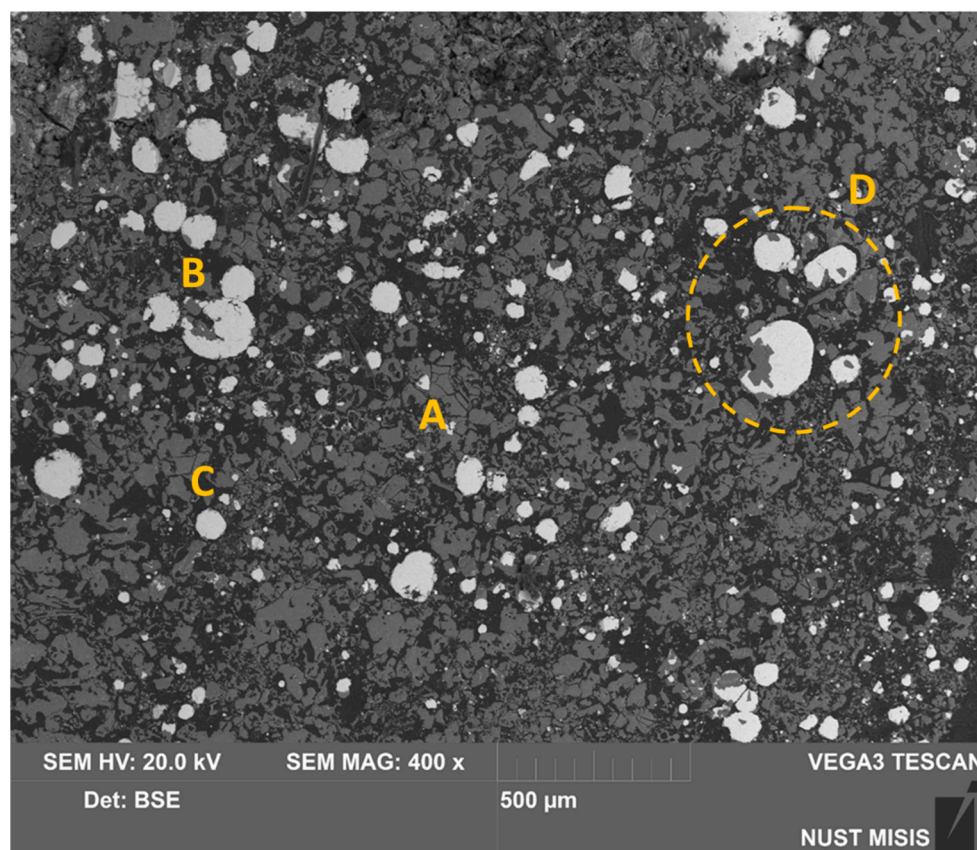


Figure 2. Low-resolution (400×) SEM image of residue after carbothermic reduction of RM at 1650 °C for 20 min.

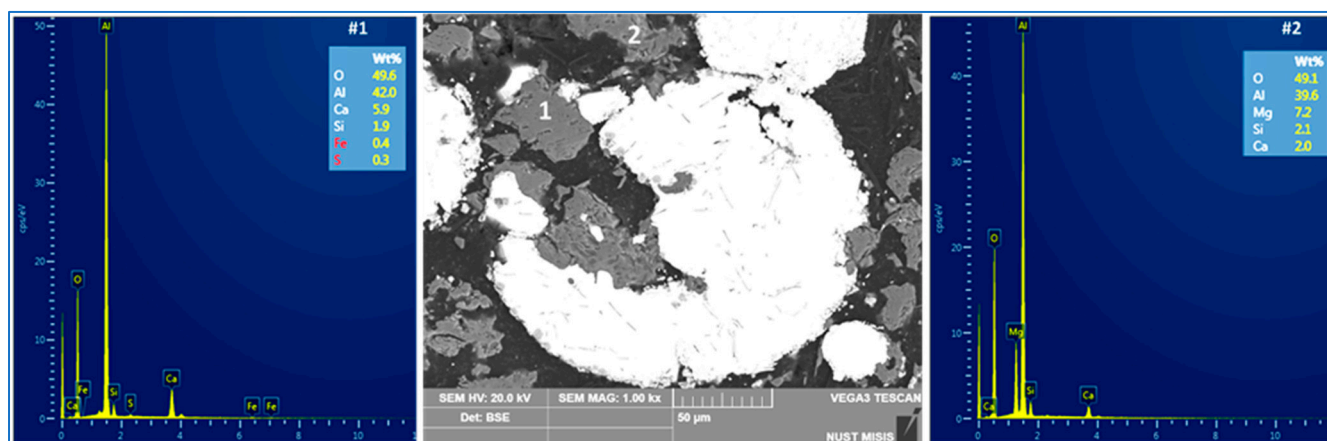


Figure 3. High-resolution (1000 $\times$) SEM/EDS images of slag-rich regions 'B' (Figure 2).

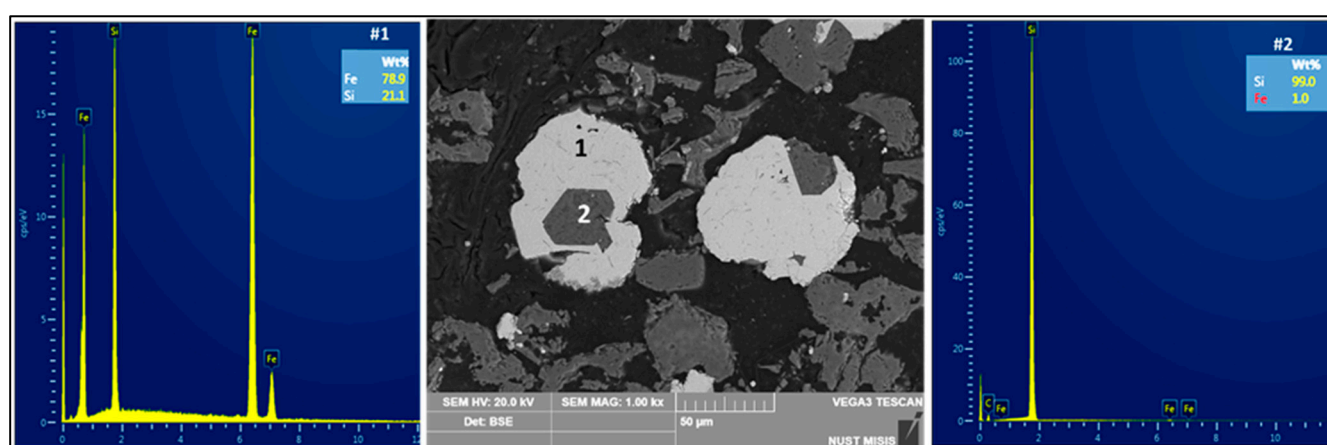


Figure 4. High-resolution (1000 $\times$) SEM/EDS images of a metallic region containing a dark grey precipitate within the metallic droplet from the dotted enclosure 'D' (Figure 2).

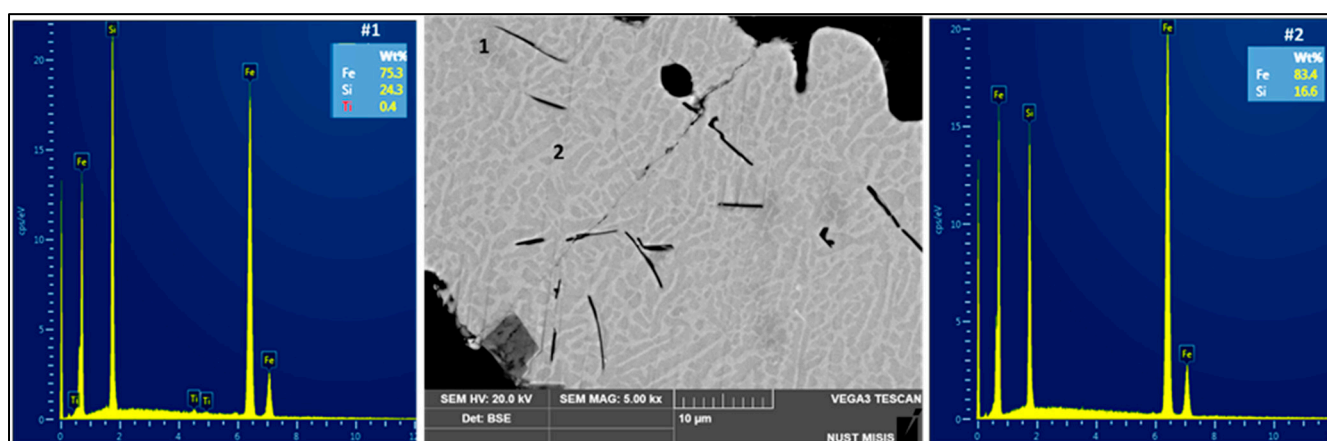


Figure 5. High-resolution (5000 $\times$) SEM/EDS images of the silica-rich metallic region (region 'T' in Figure 4).

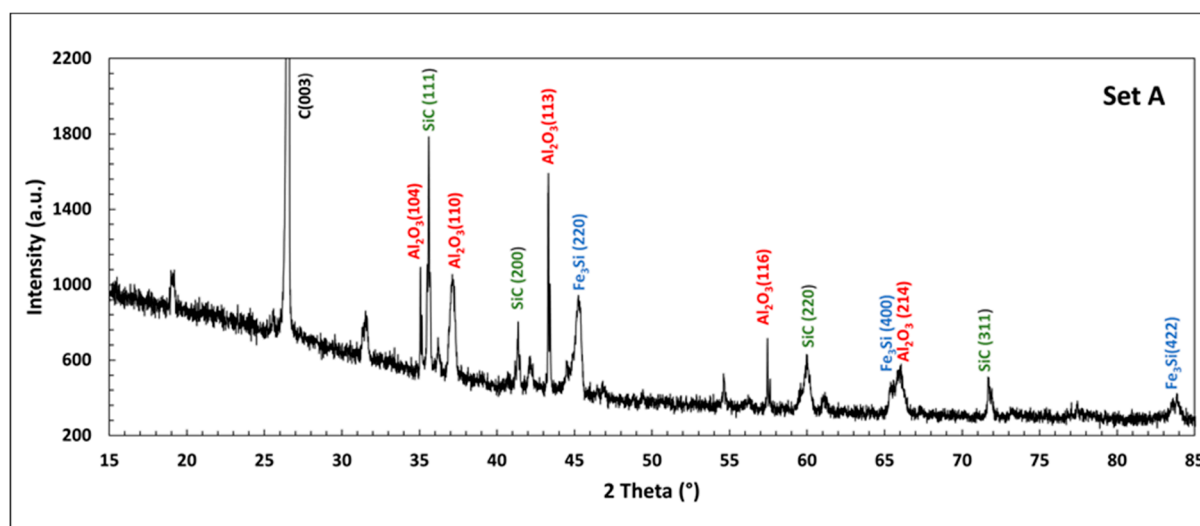


Figure 6. X-ray diffraction results on residue after the carbothermic reduction of RM at 1650 °C for 20 min.

Figure 4 shows a high-resolution image (1000 $\times$) of a metallic droplet containing dark precipitate within. Several such metallic regions can be seen in Figure 2 (e.g., within the dotted enclosure 'D'). The metallic region was identified as an Fe–Si alloy (78.9 wt.% Fe; 21.1 wt.% Si), and the dark grey precipitate was found to be primarily composed of Si (99 wt.% Si; 1 wt.% Fe). This result indicates that at high concentration levels, Si showed a tendency for precipitation within the metallic region.

Figure 5 shows an even higher magnification (5000 $\times$) SEM/EDS image of the metallic region (region '1' in Figure 4). This image shows that the distribution of Si was not uniform within the Fe matrix, with light and dark grey striations present within. Dark grey regions had a higher concentration of Si (75.3 wt.% Fe, 24.3 wt.% Si), whereas the light grey regions had lower levels of Si (83.4 wt.% Fe, 16.6 wt.% Si).

An X-ray diffraction pattern of the residue is shown in Figure 6. Distinct peaks for three phases, namely Al_2O_3 , Fe_3Si and SiC, could be clearly identified in the diffractogram. There were a few additional peaks that could not be unambiguously indexed and/or attributed to well-known phases.

3.2. Set B: Carbothermic Reduction of (20 g RM + 10 g Fe_2O_3) Blend

Detailed results are presented in Figures 7–10 on the blends of RM with Fe_2O_3 in a 2:1 proportion following carbothermic reduction at 1650 °C for 20 min. Figure 7 shows a low-resolution SEM image (400 $\times$) of the reaction product. A large number of metallic droplets were observed in the reaction product. Most of these were significantly larger in size compared to the metallic regions recorded in Figure 2 (100% RM). A number of small metallic droplets were also observed along with grey slag regions and dark areas corresponding to unconsumed carbon. Most of the metallic regions were spherical in shape, whereas the slag was composed of several oddly shaped small particulates distributed randomly.

A high-resolution (1000 $\times$) image of a large metallic droplet and surrounding areas is shown in Figure 8. The EDS results for two specific areas are also presented in the figure. The metallic droplet was composed of 82.7 wt.% Fe, 17.0 wt.% Si and 0.3 wt.% P. The slag region was completely depleted of Si and contained only alumina. No other oxides were detected. It appears as if the metallic regions and alumina have phase separated and all Si present in the system as SiO_2 has been taken up by the metallic droplets. Elemental mapping results on the system are shown in Figure 9. Most of the iron was present in

spherical shapes, indicating its molten state during reduction reactions. Si-rich regions were seen to overlap Fe regions to a great extent. The presence of iron in a given region appeared to exclude Al (present as Al_2O_3). This aspect can be attributed to the poor wettability between iron and alumina [59]. The presence of titanium was detected on the outer peripheries of metallic droplets.

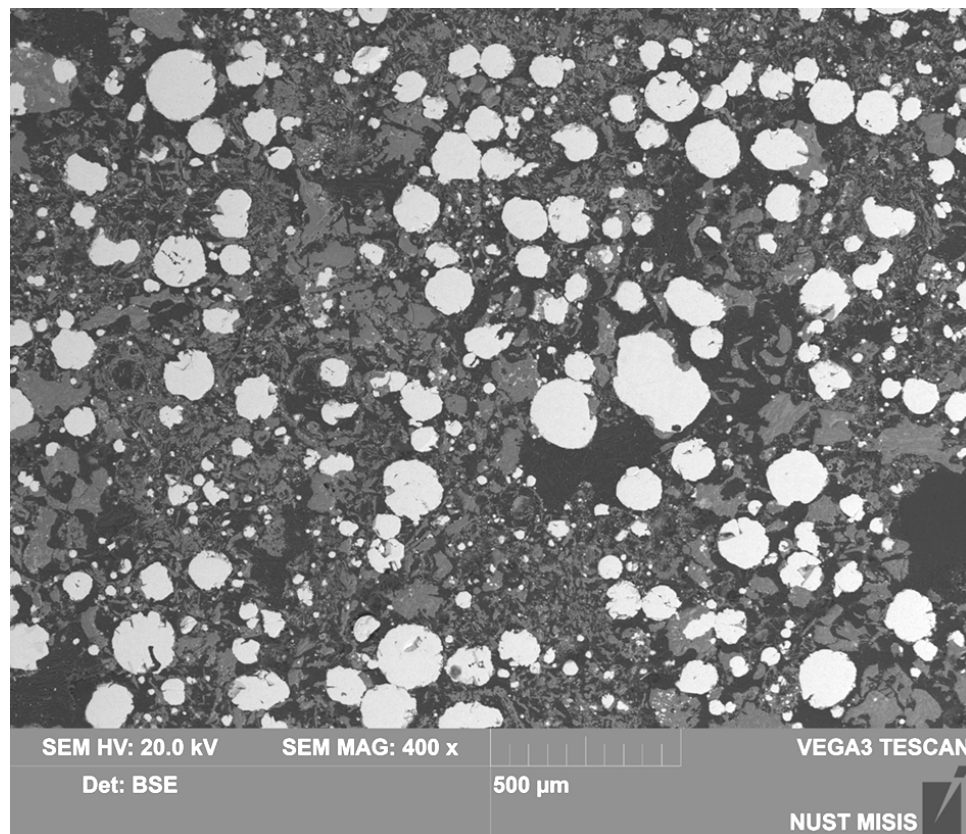


Figure 7. Low-resolution (400 $\times$) SEM image of residue after carbothermic reduction of the RM/ Fe_2O_3 blend (2:1 proportion) at 1650 $^\circ\text{C}$ for 20 min.

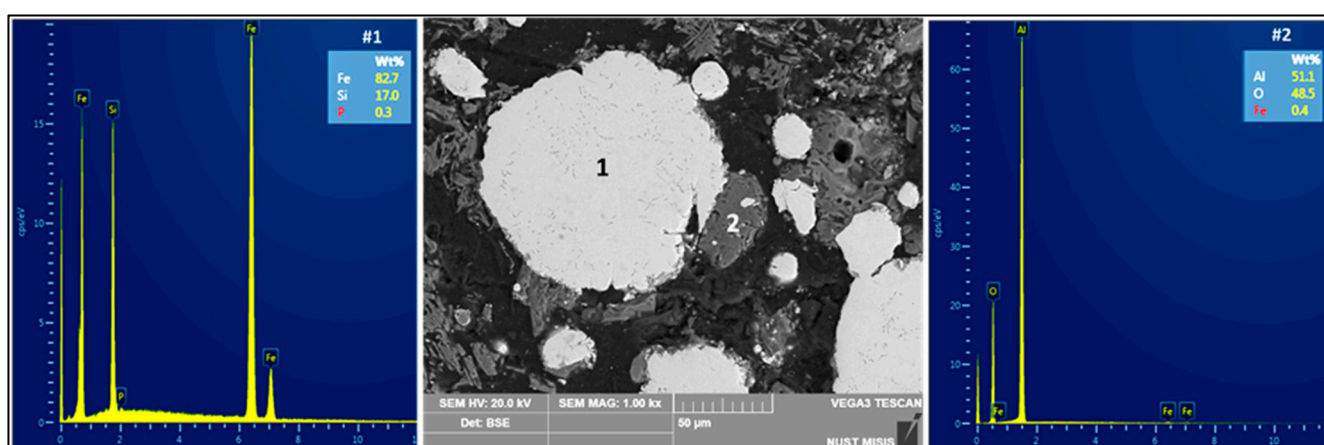


Figure 8. High-resolution (1000 $\times$) SEM/EDS images of metal/slag regions in the heat-treated RM/ Fe_2O_3 blend residue.

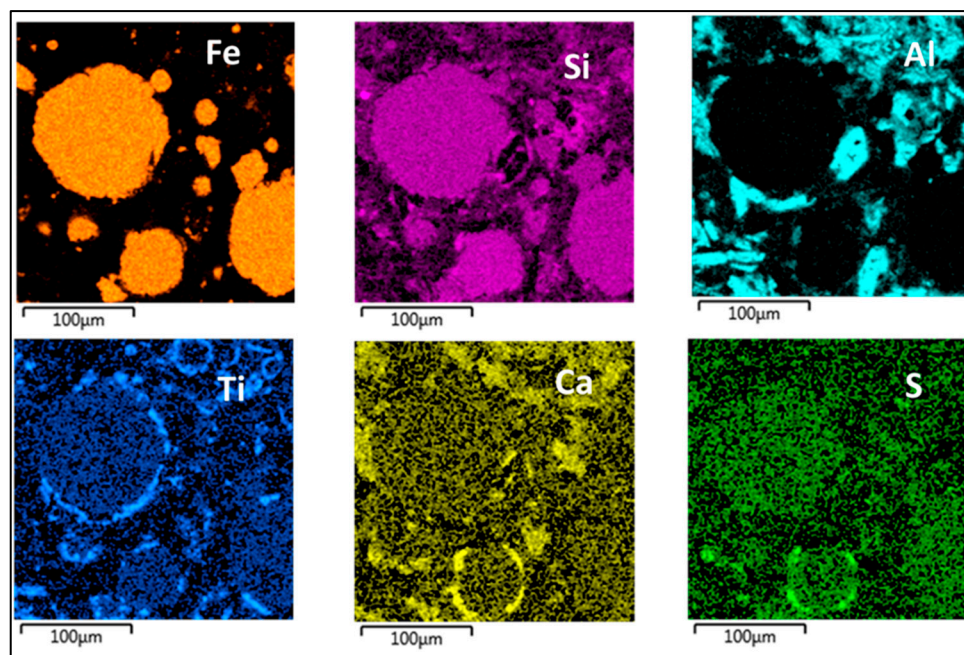


Figure 9. Elemental mapping of reduction residue in RM/ Fe_2O_3 blends after carbothermic reduction at 1650 °C for 20 min.

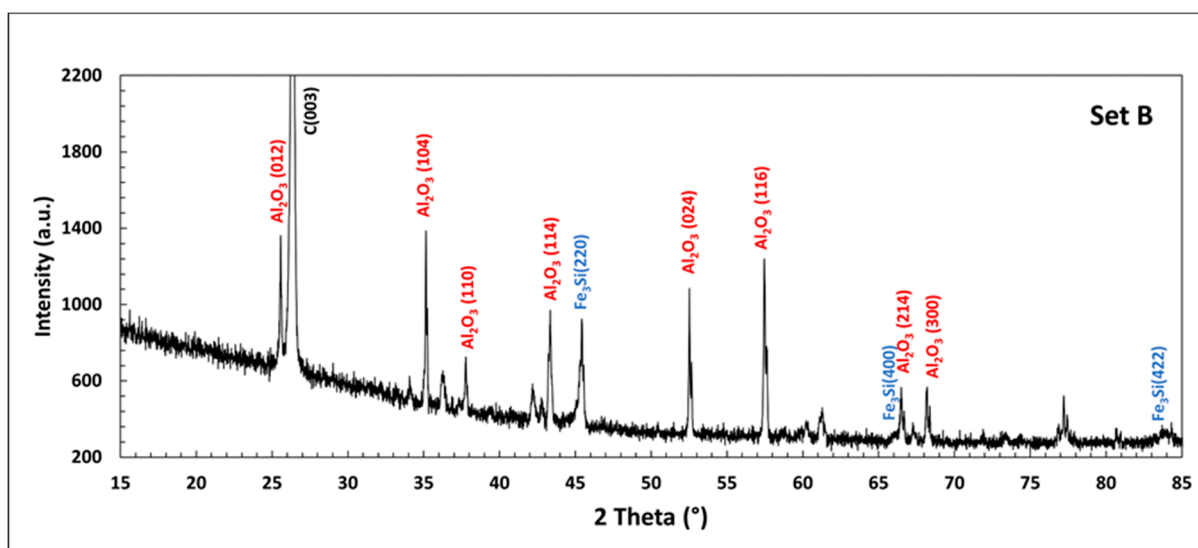


Figure 10. X-ray diffraction results on residues after carbothermic reduction of RM/ Fe_2O_3 blend at 1650 °C for 20 min.

The X-ray diffraction pattern of the reduction residue is shown in Figure 10. A large number (>8) of alumina peaks were observed in the XRD diffractogram, clearly indicating the phase separation of alumina during the carbothermic reduction of the RM/ Fe_2O_3 blend. The peaks for alumina here are much stronger than those observed during the carbothermic reduction of 100% RM (Figure 6). Alumina was the dominant phase present. The metallic phase was clearly identified as Fe_3Si . Only a few small peaks could not be fully indexed; however, none of these peaks could be attributed to SiC .

3.3. Set C: Carbothermic Reduction of (20 g RM + 10 g Red MS) Blend

Detailed results are presented in Figures 11–14 on the blends of RM with red MS in a 2:1 proportion following carbothermic reduction at 1650 °C for 20 min. The key difference between the two additives, red MS and Fe_2O_3 , is in their particle sizes and associated

reactivity. Due to micro/nano-sized particulates, red MS is far more reactive than Fe_2O_3 during the reduction processes, which could also lead to nucleation of metallic regions at a large number of sites [60].

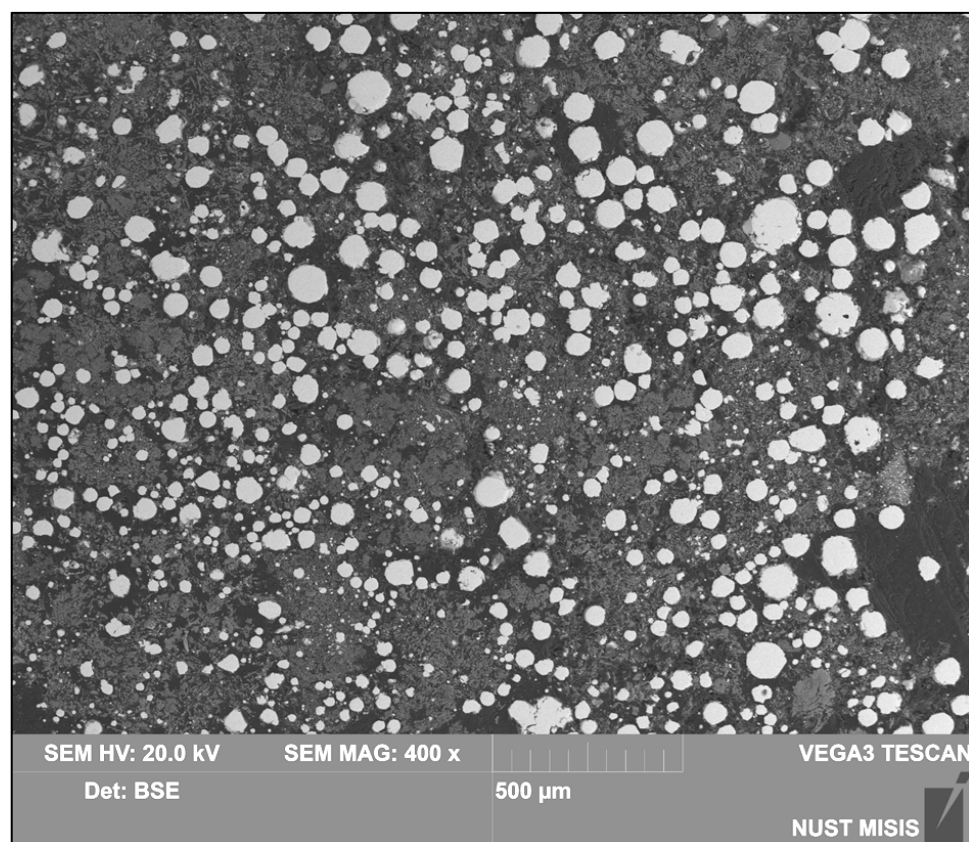


Figure 11. Low-resolution (400×) SEM image of residue after carbothermic reduction of RM/red MS blend (2:1 proportion) at 1650 °C for 20 min.

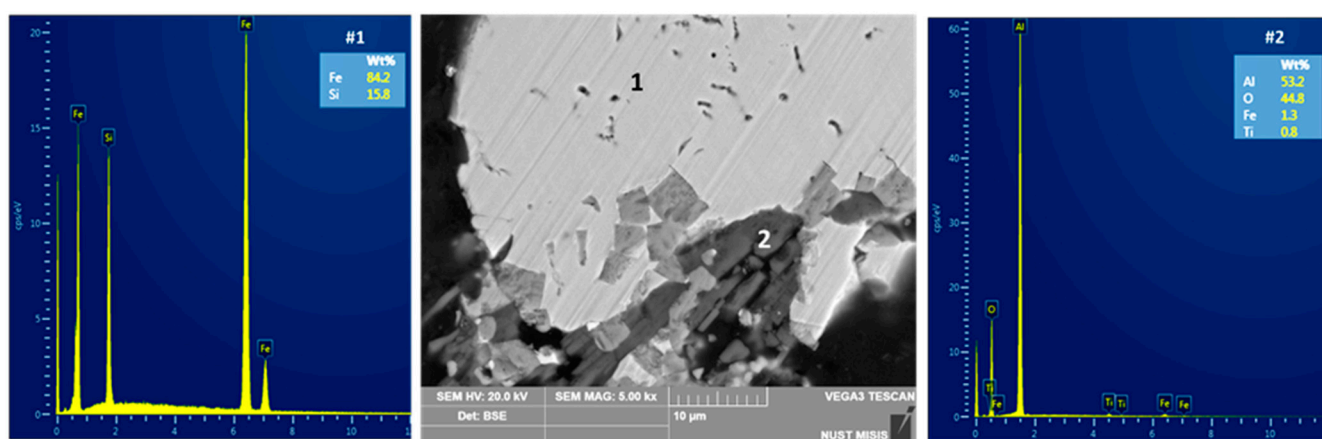


Figure 12. High-resolution (5000×) SEM/EDS images of RM/red MS blend residues after carbothermic reduction.

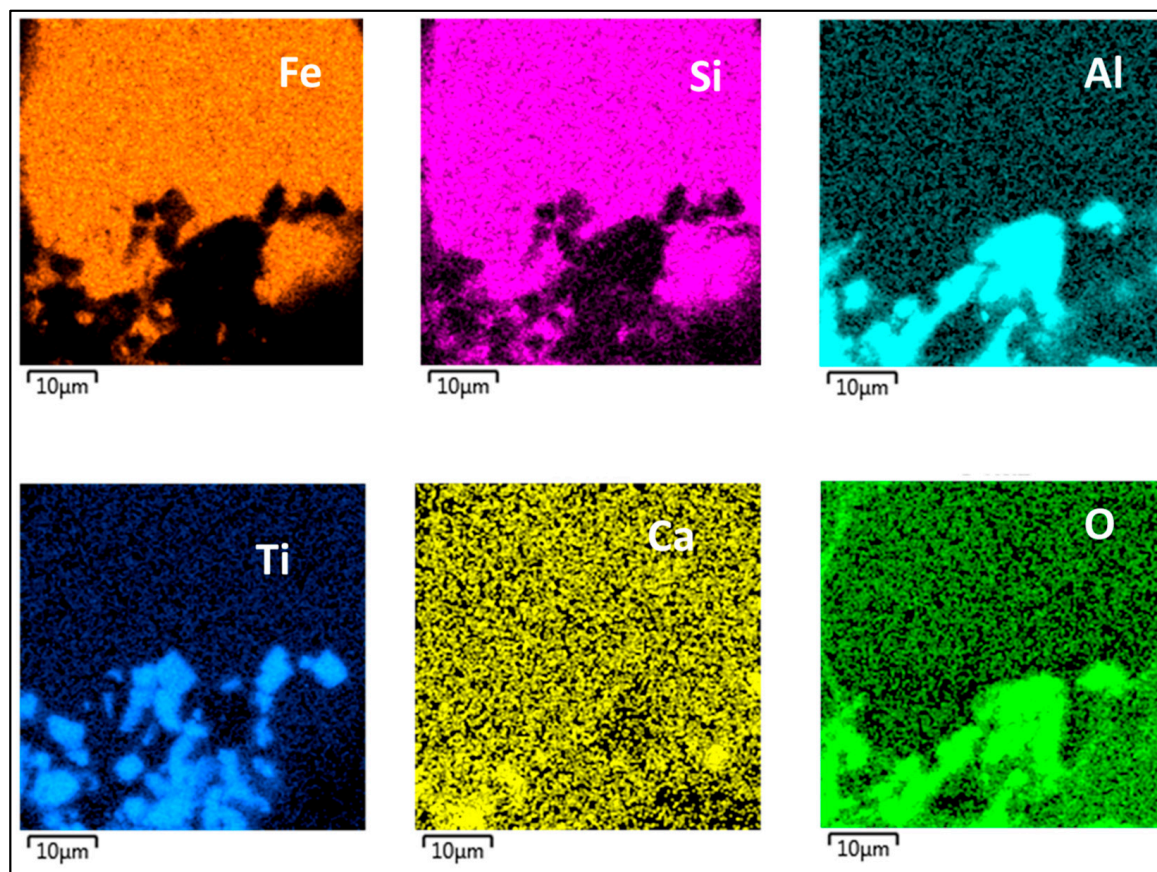


Figure 13. Elemental mapping of reduction residue in RM/red MS blend residues after carbothermic reduction.

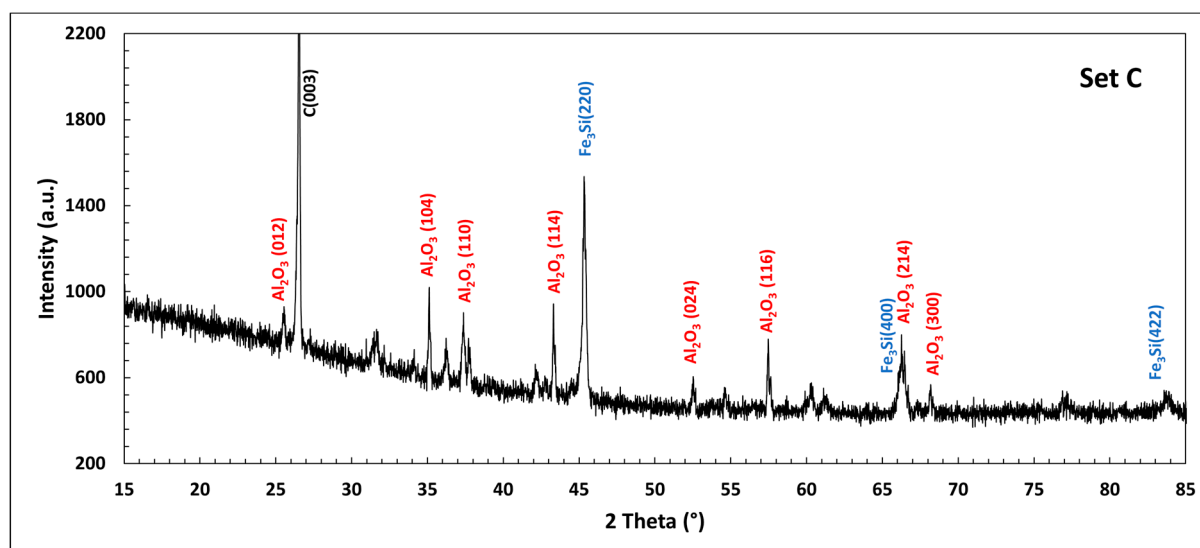


Figure 14. X-ray diffraction results for residues after carbothermic reduction of RM/red MS blends.

Figure 11 shows a low-resolution SEM image (400×) of the reaction product. A large number of small-sized metallic droplets can be seen in the micrograph. While the sizes of these droplets are smaller than those observed for RM/Fe₂O₃ blends, their number density is significantly higher (Figure 7). The sizes and number density of metallic regions for RM/red MS are also much higher than those observed for 100% RM (Figure 2). High-

resolution ($5000\times$) images and elemental mapping of RM/red MS blends are shown in Figures 12 and 13, respectively.

The metallic region in Figure 12 is composed of 84.2 wt.% Fe and 15.8 wt.% Si. No other impurities were recorded in this region. Aluminium (as alumina) was the key constituent of the slag phase. The composition of the slag phase was 53.2 wt.% Al, 44.8 wt.% O, 1.3 wt.% Fe and 0.8 wt.% Ti. The elemental mapping in Figure 13 supplements this information. Regions rich in Fe and Si were completely depleted of Al, and the regions rich in Al contained negligible levels of Fe and Si.

An X-ray diffractogram of the residue is presented in Figure 14. Peaks for Al_2O_3 and Fe_3Si were clearly observed in the diffraction spectra. However, the peak intensities for Al_2O_3 were relatively smaller than the corresponding intensities for RM/ Fe_2O_3 blends, whereas the peaks for Fe_3Si were stronger in the case of RM/red MS blends.

4. Discussion

Detailed results have been presented on the carbothermic reduction behaviour of iron-lean, high-alumina and -silica RM and its 2:1 blends with Fe_2O_3 and red MS. Significant differences were observed in all three cases. These results are summarized in Table 3 and will be discussed next in terms of resources that could be recovered after the carbothermic reduction of RM and its blends and various industrial implications.

Table 3. Characteristics of reaction products recovered after carbothermic reduction of RM and its two blends.

Blends	100% RM	RM/ Fe_2O_3	RM/Red MS
Metallic droplets			
Size range	10–100 μm	100–300 μm	25–125 μm
Composition	Si (16.6–24.3 wt.%), balance Fe	Si (17 wt.%), balance Fe	Si (15.8 wt.%), balance Fe
Number density	Low	High	High
Slag Composition	Al_2O_3 , CaO, MgO, SiO_2 etc.	Primarily Al_2O_3 , Fe_2O_3 , TiO_2	Primarily Al_2O_3 , Fe_2O_3 , TiO_2
Alumina segregation	Limited extent	Extensive	Extensive

4.1. Resource Recovery: Fe–Si Alloys

Resources recovered after carbothermic reduction of 100% RM (Case A; Figures 2–5) were small amounts of Fe_3Si alloys, alumina, SiC and other oxide-based residuals. The Si content in the Fe–Si alloys ranged between 16.6 and 24.3 wt.%. The number density of metallic droplets was somewhat low, with sizes in the range 10–100 μm . The presence of SiC and almost pure Si embedded within an Fe_3Si matrix is an interesting and novel outcome. It appears that high Si levels in the RM could not be completely assimilated within the metallic regions/slugs and tended to precipitate out. Levels of Si within slags were found to be very low (1.9 to 2.1 wt.%, Figure 3), which may be attributed to the small amount of the fluxing agent lime in the system. Generally, SiO_2 has a strong tendency to combine with CaO and/or Al_2O_3 as calcium silicates or calcium aluminosilicates [61].

However, the scenario changed considerably when small amounts of Fe_2O_3 were added to the RM (Case B; Figures 7–9), increasing the proportions of iron oxide in the system and lowering SiO_2 levels. A large number of iron-silicon (17 wt.% Si, balance Fe) metallic droplets were generated throughout the matrix; their sizes ranged from 100 to 300 μm . When red MS was added to the RM (Case C; Figures 11–13), the number of metallic droplets was even higher than those observed in Case B, but their sizes were much

smaller (25–125 μm). There was clear evidence for the formation of (15.8 wt.% Si, balance Fe) metallic droplets with zero aluminium.

4.2. Alumina Segregation

One of the key findings of this study is with regard to the segregation of alumina following the carbothermic reduction of RM and its blends. X-ray diffraction results on 100% RM (Figure 6) show a clear presence of the alumina phase in the carbothermic residue. However, these peaks had fairly low peak heights. Diffraction peaks for other phases such as Fe_3Si , SiC and residual carbon were also detected. For RM/ Fe_2O_3 blends, X-ray diffraction peaks (Figure 10) showed very strong peaks for alumina and much weaker peaks for Fe_3Si . Peaks for SiC were no longer present. While similar results were observed for RM/red MS blends (Figure 14), peaks for Fe_3Si were much stronger compared to peaks for alumina. These results indicate clear segregation of the alumina phase after carbothermic reduction of RM/ Fe_2O_3 and RM/red MS blends; such segregation was rather limited for 100% RM. Alumina segregation can be attributed to the poor wettability between the two phases [8]. The mutually displacive response of iron-rich alloys with alumina was seen clearly in Figures 12 and 13, where no inter-mixing was observed for the two, with each phase going its own way. It is also important to note the absence of aluminium in the metallic droplet and a complete segregation of alumina in the matrix. Low to small levels of the fluxing agent CaO in the RM was another factor in alumina segregation in the present case. With high levels of CaO in the system, SiO_2 shows a preferential tendency towards CaO rather than Fe_2O_3 ; the CaO– SiO_2 system then tends to form a slag with Al_2O_3 . Bound in a complex, alumina will then no longer be free to segregate out of the system [62].

During the processing of bauxite during the Bayer process, significant amounts of alumina can be lost into RM waste instead of product recovery. The present approach of blending RMs with iron-oxide resources and carbothermic reduction could be used to extract alumina from wastes, enhancing product inventory while reducing the amount of waste generated. Here, alumina was no longer present in the form of a complex, had a separate identity and could be easily recovered from the system through magnetic separation or other means.

4.3. Formation of Cermets: RM/Red MS Blends

The XRD diffraction on the residue from RM/red MS blends (Case C) showed strong peaks for Fe_3Si and relatively weaker peaks for Al_2O_3 compared to Case B (Figures 10 and 14). The phase observed in Figure 11 can be classified as an alumina-based ‘Cermet’ representing an alumina ceramic matrix and a metal reinforcement phase [63]. Cermets are novel materials composed of metal and ceramic phases, wherein the metallic properties of ceramics are enhanced after combining with metals [64]. With high hardness and toughness levels, these are finding applications as cutting tools, kilns, wear-resistant components, among others [65]. Alumina-based cermets typically consist of a metal reinforcement phase and an alumina matrix phase, together with a low-melting binder. These are usually prepared through sintering of ceramics and metallic powders, molding and calcination [66]. Lou et al. [67] have reported on the formation of alumina-based cermets by blending iron-rich (39.9 wt.%) RM with alumina dross after molding and sintering at 1400 °C for 2 h. In the present study, cermets were generated in situ during the carbothermic reduction of RM and red MS blends.

4.4. Industrial Implications

A novel and innovative route has been developed for transforming low-iron RMs into value-added resources, making a significant impact on the sustainable management of industrial waste. With typical RM recycling rates being very low (<10%), the recycling is

predominantly carried out on iron-rich (or high iron) RMs to extract iron values present within. Little effort is put towards recycling iron-lean RMs that are considered quite useless and are generally dumped in various storage facilities. A novel strategy has been developed to extract alumina and other valuables (Fe–Si alloys, SiC, cermets, etc.) from an RM containing 22.2 wt.% alumina.

The key purpose of the Bayer process is to extract alumina from the bauxite ores. In the present scenario, a proportion of alumina was being diverted to RM waste instead of being recovered as a product, severely impacting process productivity. Producing 1 tonne of alumina requires about 2.05–2.53 tonnes of bauxite ore, 0.88–0.92 tonnes of NaOH, 0.1–0.13 tonnes of lime, 0.28–0.38 tonnes of fresh water (washing) and 2.9 MWh energy for various processes, including pressure leaching, crystallization, rotary kiln and calcination [68,69]. In addition, there will be the generation of greenhouse gas emissions and waste passing on to fresh water. In the present study, alumina and other valuables will be recovered from the RM in a single process step. While it is not yet possible to quantify reductions in energy, materials and processing costs, the overall processing costs for alumina extraction are likely to be a small fraction of the Bayer process. Additional benefits will include reduction in costs associated with RM waste management and value addition through the generation of Fe–Si alloys and SiC.

5. Concluding Remarks

This study presents the first systematic approach to recovering alumina and cermets from an iron-lean red mud using carbothermic reduction. Up to 90% of such red muds (low iron, high alumina, high silica) are considered to have very little value, are too difficult to recycle and are destined for dumps. While SiC was produced during the carbothermic reduction of 100% RM, copious amounts of alumina were generated as a separate phase during the carbothermic reduction of RM/Fe₂O₃ blends. High-value cermets were also produced during the reduction of RM/red MS blends. With over billions of tonnes of RMs being dumped worldwide, there is an urgent need to develop novel strategies for recycling and resource recovery from such RMs. The recycling strategy presented could play an important role in recovering value-added resources from RM wastes, thereby enhancing process productivity, cost-effectiveness of alumina regeneration, waste utilization and making sustainable developments in RM waste management. Regeneration of alumina from waste RMs could have a strong impact on the productivity and economics of the Bayer process.

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