Conditioning of Red Mud for Subsequent Titanium and Scandium Recovery – A Conceptual Design Study

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Leaching experiments were undertaken on red mud materials (red mud and red mud slag). The red mud slag was produced via the carbothermic reduction of red mud at high temperatures (T > 1500 °C) via SAF treatment. Furthermore, iron was recovered in the smelting step to the metal phase. Ti and Sc were successfully recovered from the red mud materials by hydrometallurgical treatment. For both critical metals, it was found that sulfuric acid was the best mineral acid among others. Since direct red mud leaching had some shortcomings, a route designed to overcome them is proposed. For optimal Ti and Sc recovery from red mud a promising process flowsheet combining pyrometallurgical and hydrometallurgical treatment is proposed as follows: pyrometallurgical processing (fluxed smelting to produce calcium oxide based slag phases and controlled cooling for crystalline and glassy slags), leaching for maximized Ti- and Sc extraction and followed by a multistage precipitation (for metal recovery and solution purification). Initial trial results showed that the proposed process is promising.

Keywords:

Red mud – Scandium – Titanium – Smelting

Aufbereitung von Rotschlamm für eine anschließende Rückgewinnung von Titan und Scandium – Eine Konzeptstudie

Rotschlamm, der Laugungsrückstand der Aluminiumoxidgewinnung nach dem Bayer-Verfahren, enthält neben hohen Fe- und Al-Anteilen auch sog. kritische – gemeint sind wirtschaftsstrategische – Metalle. Laugungsversuche sind mit Rotschlamm und sog. Rotschlammschlacke durchgeführt worden, um diese Metalle in den Stoffkreislauf zurückzuführen. Die Herstellung der Rotschlammschlacke erfolgte durch carbothermische Reduktion von Rotschlamm im Elektroofen bei hohen Temperaturen (>1500 °C). So wurden Eisen direkt und Titan sowie Scandium aus der Schlacke gewonnen. Für die Laugung dieser beiden Metalle ist Schwefelsäure im Vergleich zu anderen Säuren als wirksame Mineralsäure ermittelt worden. Da aber eine direkte Laugung von Bauxitrückständen schwierig ist, wurde eine alternative Route entwickelt. Für eine maximierte Wiedergewinnung von Titan und Scandium aus Rotschlamm wird der Ablauf eines vielversprechenden kombinierten Prozesses wie folgt vorgeschlagen: Pyrometallurgischer Prozess (Schmelzen unter Zugabe von Flussmitteln zur Herstellung einer calciumoxidbasierten Schlacke und kontrollierte Abkühlung für kristalline und glasartige Schlacken), Laugung und mehrstufige Fällung (Laugenreinigung und Wertmetall-Wiedergewinnung). Die Ergebnisse erster Versuche haben den angestrebten Prozess als sehr aussichtsreich bestätigt.

Schlüsselwörter:

Rotschlamm – Scandium – Titan – Schmelzen

Conditionnement de la boue rouge pour récupération ultérieure du titane et scandium. Une étude conceptual.

Tratamiento de lodo rojo para recuperación posterior de titanio y escandio – un estudio conceptual

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1 Introduction

There is growing effort from industry to promote sustainability and implementation of a zero-waste valorization; the use of by- and waste products as secondary resources is gaining more interest. One of the most readily available potential secondary resource is red mud which is the by-product of the Bayer process. In 2012, it was reported that the total global annual red mud generation from the Bayer process was approximately 120 million tonnes [1]. The estimated amount of red mud stocks is around three to four billion tonnes and is continually growing. The increasing amounts of red mud, coupled with high potential value associated with contained valuable metals and rare earth elements including Fe, Al, Ti, Sc, REEs render it an attractive secondary resource. Furthermore, storage difficulties of this dynamically growing stream and the potential impact on the environment has also accelerated the studies
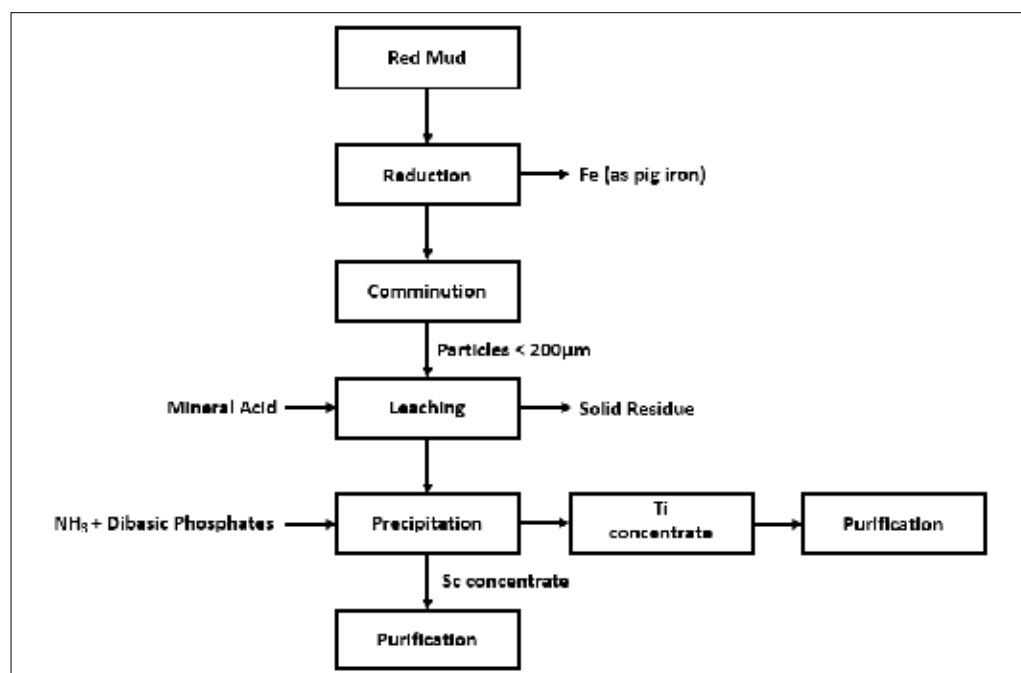
Fig. 1:
Conceptual route for Ti and Sc recovery from red mud

targeting red mud valorization in the last decade. Efforts are given to recover the critical metals contained in this largely stockpiled waste. The targeted elements which are concentrated in the red mud are in some cases nearly up to an ore level and they include Fe, Ti, Sc and REEs.

Hereinafter, a potential route for red mud processing with the target of Ti and Sc recovery by hydrometallurgical means subsequent to pyrometallurgical processing is described. The pyrometallurgical step aims at achieving iron recovery and slag conditioning for downstream processing. The proposed conceptual flowsheet is shown in Figure 1.

2 State of art and preliminary observations

2.1 Previous studies

Pyrometallurgical processing is one of the most well researched methods for iron recovery from red mud where iron is recovered via solid state reduction or in a reductive smelting process. Smelting of red mud entails selective high temperature carbothermic reduction in a furnace to produce pig iron and a slag phase enriched with oxides of the accompanying elements found in red mud. The pig iron can also collect some of the elements of compounds found in red mud such as Cr and Ni and to some extent Ti, Si, Mn and V. The ENEXAL Bauxite Residue Treatment process transforms red mud into two marketable products, namely pig iron and mineral wool fibres [2]. This is achieved via a carbothermic smelting of red mud at temperatures of up to 1580 °C in an electric arc furnace using silica and lime as fluxing agents. Borra et al. [3] undertook laboratory scale smelting of red mud for iron recovery and selective rare earths recovery. 95 % iron recovery was obtained at temperatures between 1500 and 1600 °C with 20 % wollastonite and 5 % graphite additions. Kaussen & Friedrich [4] carried out laboratory scale carbothermic reduction of iron oxide in red mud utilizing an electric arc furnace. The operating temperature for the testwork was 1600 °C

to 1700 °C. A saleable pig iron product was obtained. An increase in CaO content was found to lower the slag viscosity of the investigated slags. Na entrainment in the slag was observed mainly due to the acidic and viscous slags. High alumina in the slag was found to raise the liquidus temperature of the slag.

There have been many studies carried out to recover Ti from various red mud sources, utilizing leaching with aggressive acidic conditions, higher acid concentrations and lower solid to liquid ratios. Although higher Ti leaching efficiencies were achieved in such leaching environment with high acid consumptions, selectivity over iron and aluminium was mostly very poor which keeps that process far away from industrial application [5-7]. Agatzini-Leonardou et al. [8] reported that titanium can be leached from red mud with direct sulfuric acid leaching around 64.5 % when acid normality was 6 N with a 1:20 of solid to liquid ratio at 60 °C. In this study, selectivity of Ti recovery has not been evaluated.

In another study, lower solid to liquid ratio and higher leaching temperatures were utilized by Sayan et al. [9] for direct leaching purpose of Ti from red mud. It was mentioned that leaching temperature, acid concentration, and solid-to-liquid ratio have a dramatic effect on the leaching of TiO_2 from red mud with sulfuric acid. Lower solid-to-liquid ratio (1:25) and higher reaction temperature (90 °C) lead to higher leaching efficiency with a value of 96.3 %. Another study performed by Ghorbani et al. [10] investigated the combination of concentrated H_2SO_4-HCl with 3:1 ratio at 100 °C to reveal the leaching selectivity. In this case, the extraction efficiencies of iron, aluminium and titanium obtained as 92 %, 91.3 %, and 97.7 %; respectively. Despite the high titanium recoveries, Al and Fe are also highly extracted to leachate. Leaching selectivity is as crucial as leaching efficiency for a successful hydrometallurgical recovery. In previous studies, generally, leaching selectivity of Ti has not been well studied or very poor

selectivities achieved. This lack of knowledge induced the focus of this study, proposal of a new conceptual model.

Since Sc can be found in low concentrations in red mud leachates, main focuses to recover Sc were solvent extraction, ion exchange or the combination of both. Red mud from different parts of the world was examined in previous studies and variety of processes were proposed, and almost complete Sc recovery was achieved [11-14]. However, existence of the impurity ions such as Fe, Al, Si, etc. in the leachate, co-extraction of those impurities with Sc became a problem for complex hydrometallurgical operations. That is the reason why intensive purification steps are needed while dealing with Sc from a red mud leach solution.

2.2 Preliminary observations

Initial studies have been performed without any pre-treatment to investigate feasibility of direct leaching of red mud. Considering previous studies, for higher Ti and Sc leach efficiency, sulfuric acid solution has been used as leachate with various concentration and various red mud to acid solution ratio (S/L).

Figure 2 represents the results of three different leaching experiments with different acid concentrations with different S/L ratios with the focus on Ti. When 2.5 M sulfuric acid was used with S/L of 1/10, 32 % of Ti leaching is performed at 70 °C for two hours. When acid molarity is increased to 4 M, Ti leaching efficiency is raised to 42 % and by increasing the acid content to 1/50, extraction yield was increased up to 69 %.

Selectivity of Ti leaching with respect to Fe is evaluated with the following equations

$$m_A = \frac{\% A_{(solution)}}{\% A_{(residue)}} \tag{1}$$

$$S_{Ti/Fe} = \frac{m_{Ti}}{m_{Fe}} \tag{2}$$

where $\% A_{(solution)}$ is the content of the metal in the solution, $\% A_{(residue)}$ is the content of the metal in the residue, $S_{Ti/Fe}$ is the selectivity of Ti over Fe.
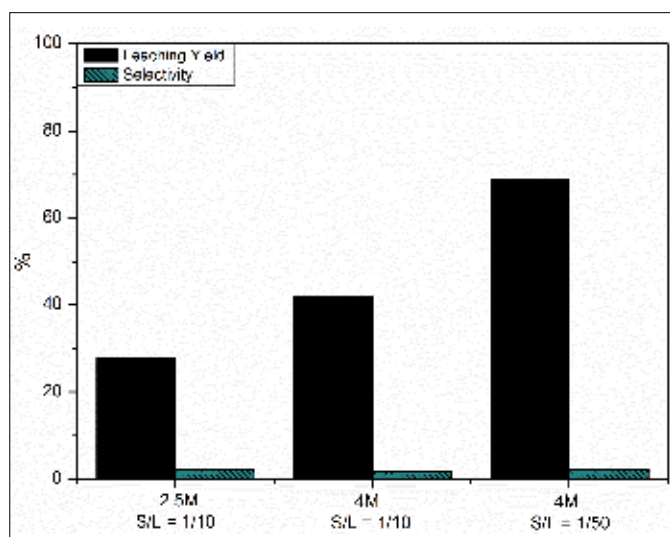

Fig. 2: Leaching efficiency and selectivity over iron values for titanium

Fig. 3: Silica gel formation in samples during sulfuric acid leaching

Relatively poor selectivity values of 2.35, 1.91 and 2.29 were observed for these three experiments; respectively. With high concentration of sulfuric acid and an excessive solid to liquid ratio of 1:50 only 69 % of Ti efficiency could be achieved with a poor selectivity. Leaching efficiency of Scandium was also found to be very low in parallel with Ti, although leaching was performed in a highly aggressive acidic environment with higher acid concentrations and lower S/L.

Another drawback of direct leaching is high silica gelation rates which is a common hydrometallurgical problem in the presence of soluble silica [3]. Due to that problem, it was not possible to separate solid and liquid after leaching. Even if they had been separated, immediate gelation of the leachate was experienced which makes them useless as shown in Figure 3.

Despite higher acid consumptions, low leaching efficiency and selectivity of Sc and Ti combined with gelation problem brought out a stepwise method for efficient valorization of red mud by combining pyro- and hydrometallurgical methods.

3 Experimental methods and set-up

The slag utilized for the leaching testwork originated from smelting of pretreated red mud from a landfill in Lünen. The details of the smelting are as outlined by [4]. In this case, red mud is smelted with the addition of lignite coke to recover the iron content as a metal phase but without the addition of fluxes for an adjustment of slag properties. The chemical analysis of this red mud and the produced slag are presented in Table 1. The leaching tests were carried out with a glass beaker, heating plate and magnetic stirrer for controlling the reaction temperature and stirring speed. Red mud slurry was dried and grounded into fine particles and poured into pre-heated (70 °C) sulfuric acid solution to start the leaching process. The experiments were carried out over a fixed leaching time of 120 minutes at a set temperature of 70 °C, 360 rpm and varying acid concentrations. 1:10 and 1:50 were examined as solid-liquid ratio (red mud to acid solution). At the end of two hours, mixture was immediately vacuum filtered with a vacuum pump and fine filter paper to separate residue and leachate. Leachate was 10-fold diluted for ICP analyses and solid residue was kept in oven at 90 °C to be dried.

The feed and product samples for the investigated work were analysed by X-ray fluorescence (XRF) and by inductively coupled plasma mass spectrometry (ICP-MS). Furthermore, the feed and slag samples were analysed mineralogically by

Table 1: Composition of the used red mud from Lünen and the produced slag and metal

	Red mud [wt.-%]	Slag [wt.-%]	Recovery (slag)	Pig iron		[wt.-%]
Fe_2O_3	35.3	1.10*	Very low	Fe		94.1
Al_2O_3	15.7	29.1	Very high	C		4.5
Cr_2O_3	0.2	0.2	Low	Cr		0.4
Na_2O	8.9	11.4	High	P		0.4
TiO_2	11.4	21.3	Very high	Ti		0.07
SiO_2	14.0	25.4	Very high	Si		40 ppm
CaO	6.7	13.4	Very high	S		0.03
Sc	86 ppm	154 ppm	Very high			
LOI**	7.3	–0.1				

*calculated as FeO; **Loss on ignition

Table 2: Composition of the initial red mud from AoG [wt.-%]

Fe_2O_3	Al_2O_3	Cr_2O_3	Na_2O	TiO_2	SiO_2	CaO	Sc
44	23	0.33	1.8	5.6	5.5	10.2	122 ppm

XRD. The metal samples were analysed by spark spectroscopy. LECO was used for C and S measurements.

Table 1 presents the chemical compositions of red mud and produced slag and metal after EAF treatment in a comparative manner. Just 1 % remaining iron oxide shows the success and the effectiveness of EAF treatment. When this major component is recovered as pig iron, other constitutents and mainly the focus of this study, Sc and Ti are twofold enriched in slag. Some desulphurisation is achieved, and phosphorus reports to the pig iron.

In order to change the mineralogy of the slag to improve its leachability and to reduce the process temperature and slag viscosity in the EAF, red mud from Aluminium of

Greece (AoG) was used for further smelting experiments with the addition of fluxes. The received samples were dried at 105 °C for 24 hours and subsequently crushed to lumps of 5 to 20 mm. The chemical composition and phase analysis via XRD of the red mud sample are shown in Table 2 and Figure 5 respectively. In addition to the dried red mud, lignite coke with a fixed carbon content of 86.5 % and lime containing 95 % CaO were fed into a 100 kVA an electric arc furnace. A batch mass of 1.5 kg of red mud was mixed with 10 % of lignite coke (i.e. 0.15 kg) and varying lime additions (10 % to 50 %) and smelted in a 1.5 litre graphite crucible at a temperature of 1500 to 1550 °C. The melt was held at temperature for one hour and the furnace was subsequently switched off. The graphite crucible was removed upon cooling from the furnace, broken to separate slag from metal. The separated slag and metal were then weighed. The samples were then sent for chemical and mineralogical analysis. The proposed smelting pre-treatment process based on a slag design via fluxing mainly with lime and controlled cooling of the slag is shown in Figure 4.

4 Results and discussion

4.1 Leaching

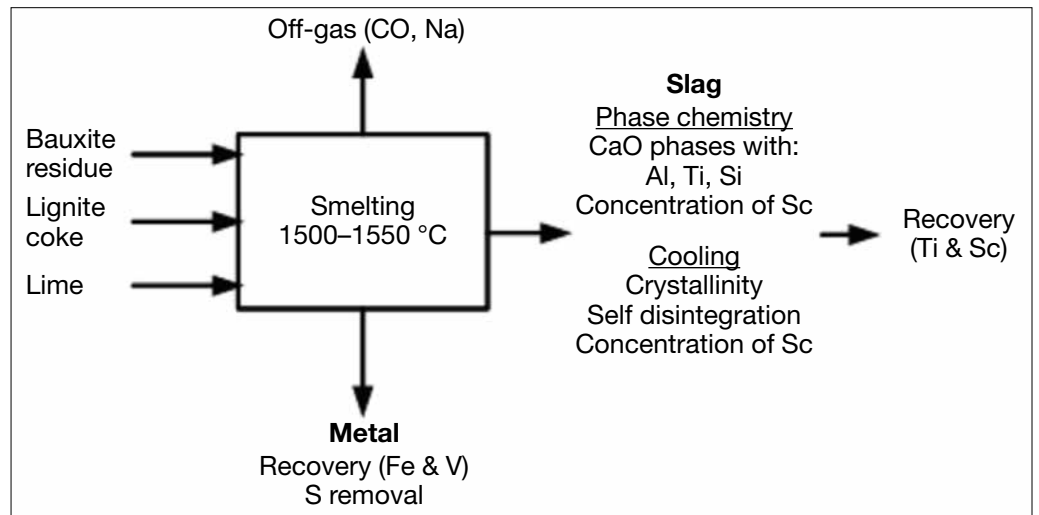EAF treated slag mentioned in Table 1 is examined with HCl, HNO_3 and H_2SO_4 to reveal the most efficient lea-

Fig. 4:
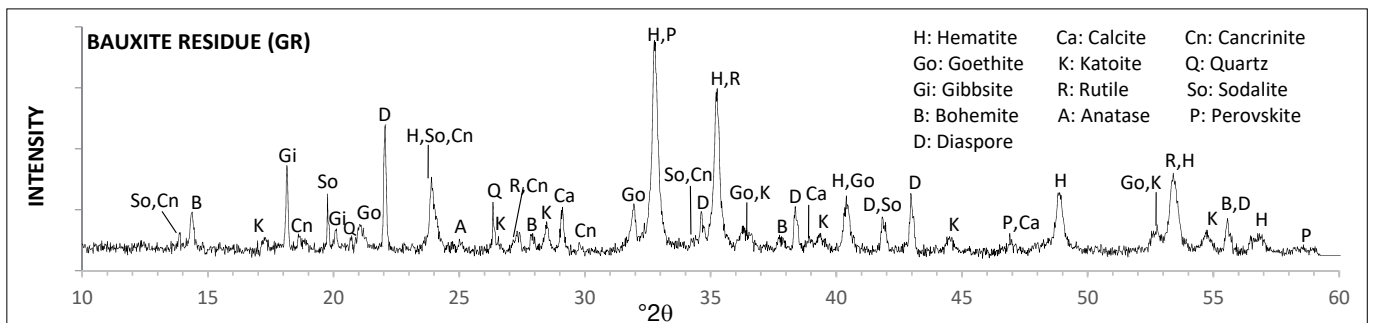Smelting pre-treatment process proposed for Fe recovery and slag conditioning for Ti and Sc recovery

Fig. 5: XRD pattern (Cu $K\alpha_1$ radiation, $\lambda = 1.54060 \cdot 10^{-10}$ m) of the initial red mud from AoG
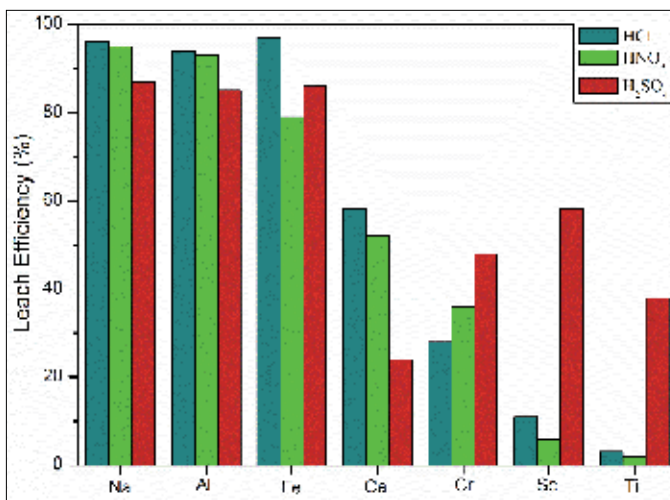
Fig. 6: Leaching efficiencies of different elements contained in slag after reductive red mud smelting with different acids
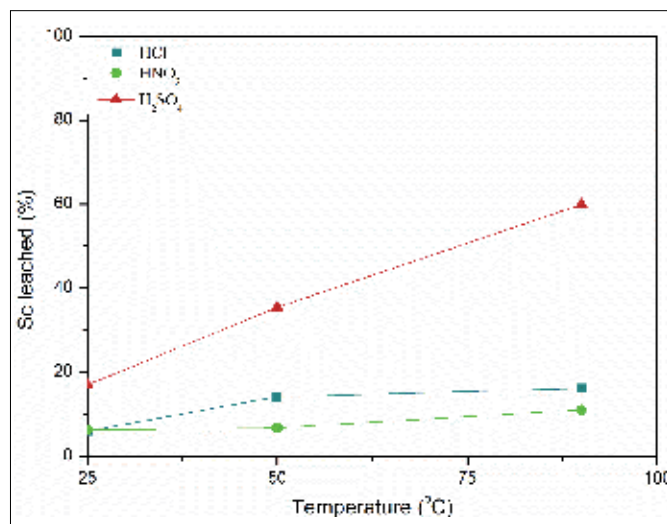

Fig.7: Sc leached out from leach residues

chate in terms of Ti and Sc recovery. Table 2 shows the ICP analyses of the constituent elements of the slag. Among three acid types, while HCl is the best alternative for Al and Fe, sulfuric acid is the most promising one for Ti and Sc. Moreover it is worth to emphasize that, lower acid molarity (3M) and higher S/L (1:10) was adjusted in these experiments. Despite lower acid consumption, leaching efficiencies were higly promising in comparison with direct leaching experiments with higher acid consumptions.

Figure 6 shows the leaching efficiencies of the constituent elements of red mud with different mineral acids at 90 °C. While Na, Al and Fe showed similar leaching efficiencies during leaching, more drastic changes in efficiencies were observed during leaching with different acids. While 50 % of Cr, 58 % of Sc and 38 % of Ti was leached out from the slag by sulfuric acid, only 28 % and 35 % of Cr, 11 % and 6 % of Sc and 3 % and 2 % of Ti was extracted from the slag by hydrochloric and nitric acid respectively.

Figure 7 shows remaining Sc content in slag after leaching which is inversely proportional to Sc leached out. Three acids and various temperatures have been investigated in terms of Sc recovery efficiency. It was found that at all tem-

peratures, the Sc amount that was not leached remained in the residue. The extraction rate is the lowest for sulfuric acid leaching while hydrochloric and nitric acid showed a similar leaching behavior. Furthermore, as reaction temperature increased in the leaching trials, extraction rates of Sc also increased in all cases. Sulfuric acid showed superior leaching ability of Sc when it compared to the other mineral acids. Considering Figures 6 and 7, it can be said that the most promising leachate for recovery of Ti and Sc is sulfuric acid.

Chemistry of slag is not adjusted in this preliminary EAF treatment to achieve more stable silicate phase to deal with gelation problem. Therefore, silica gel problem was again observed to a certain extent. However, for selected most efficient sulfuric acid leachate various temperatures have been examined to investigate Si content as shown in Figure 8.

It was observed that higher temperatures are beneficial to prevent silica gelation. Initial Si content at the very early stages of the reaction is decreasing with increasing reaction time. A sharp decrease in Si concentration in leachate is observed in sample at 90 °C. In other samples with lower reaction temperatures, that effect was not strongly highlighted. As temperature decreases, the Si content in lea-
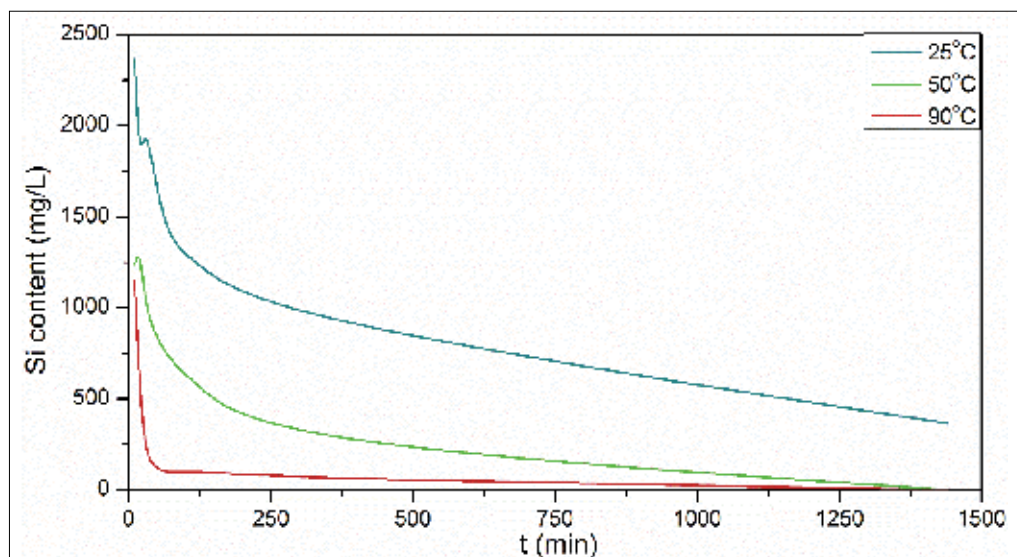

Fig. 8:
Change in Si content in time according to different leaching temperatures

chate decreases with a lower rate. When Si reaches a critical concentration, gelation or precipitation of silica takes place depending on pH, concentration and temperature. Increasing reaction kinetics owing to higher temperature may favor precipitation of silicate as soon as it reaches the solubility limit. However, for lower temperatures, Si content in leachate stays higher due to poor kinetics and super saturation of leachate in terms of Si induces gel formation as previously reported. The decrease of silica gel formation has a positive effect on the downstream processing to recover and purify the Sc and Ti. The clean aqueous solution can easily be processed. Beside hydrometallurgical treatment, an optimization in EAF treatments with various fluxes to precipitate Si in a more stable $CaO \cdot SiO_2$ phase with low solubility would be highly promising to deal with gelation problem. This strategy is followed by slag conditioning in section 4.2.

To eliminate the Si-gel formation problem completely, dry digestion method can be used as an alternative. According to this method, addition of small amount of highly concentrated acid directly to the powder leads to high Si liberation, inhibits networking and promotes sub-µm silica crystallisation. Since silicon remains in the stable residue phase while washing, remaining precious metals could be washed out without creating any Si-gel problem in the aqueous phase [15].

In order to recover Ti and Sc from the obtained impure leach liquor, precipitation process will be used. As it was proposed in the previous study, Fe can almost completely be removed from the system while losing negligible amount of Sc by ammonia solution [16]. Successive precipitation route containing dual staged Fe precipitation stage followed by Sc recovery stage by dibasic phosphate precipitation was designed to produce Sc phosphate directly from the leachate. This process offers low co-precipitation, high Sc recovery yield with extraordinary selectivity. In same manner Ti can also be precipitated with ammonia at lower pH values just before Fe removal process. Since the Fe levels will be negligible in both precipitates, the further purification processes will be much easier than the conventional routes.

4.2   Effect of slag conditioning

The proposed smelting pre-treatment process is based on a slag design via fluxing mainly with lime and controlled cooling of the slag. The slag and metal results are shown in Table 3.

Table 3: Composition of the slag and pig iron product

Pig iron	[wt.-%]	Slag	[wt.-%]
Fe	93.0	FeO	1.6
C	5.9	Al_2O_3	38.8
Cr	0.6	Cr_2O_3	0.1
Si	0.1	Na_2O	0.4
V	0.2	TiO_2	7.6
Ti	0.3	SiO_2	7.6
P	0.1	CaO	43.8
S	65 ppm	Sc	160 ppm

It can be seen from Table 3 that high iron recovery to the metal is achieved during the smelting process as slag with an FeO content of <2 % is achieved. This also signals that sufficient carbon is available for the reduction process. Chromium and vanadium are mainly recovered to the metal, there exists potential for vanadium recovery from the pig iron via downstream selective oxidation of the pig iron product. The low sulphur content in the pig iron is an indication of desulphurisation achieved during the smelting process, and this can be attributed to the added lime which renders the slag more favourable for sulphur absorption from the metal. The smelting process operates under highly reducing conditions therefore dephosphorisation is not achieved during the smelting process, phosphorus removal from the metal can be explored post smelting. The pig iron product presents a suitable feed material for secondary steel making. The slag above was produced with an addition of 20 % lime to the red mud. The slag results show that with this fluxing strategy calcium oxide becomes the major component of the slag.

The high lime slag design for the high alumina red mud is such as to lower both the slag liquidus and slag viscosity for good slag and metal separation at lower operating temperatures. The high sodium content of the slag would be expected to decrease drastically under the low viscosity slag as it would volatilize and not be entrained in the slag, the low slag sodium content confirms this. This improves the attractiveness of the slag to a wide range of applications. Furthermore, the slag phase chemistry is targeted towards phases that would render it desirable for downstream processing including but not limited to gehlenite, calcium aluminate, calcium silicate, perovskite which streamlines the downstream processing of the slag. Figure 9 shows the XRD patterns of the slags that were produced under different flux (lime) addition.

The XRD patterns clearly shows that different phase chemistry prevails for different flux additions. The high lime additions lead to formation of calcium aluminate phases in the slag and these include $12CaO \cdot 7Al_2O_3$ and $3CaO \cdot Al_2O_3$. The 30 % lime addition signals a shift from $12CaO \cdot 7Al_2O_3$ slag to a $2CaO \cdot Al_2O_3 \cdot SiO_2$ slag. Lower lime addition is dominated by the presence of gehlenite and lower stoichiometry calcium aluminate phases in the produced slags. Ti is present in the form of perovskite in all the analysed slags. Furthermore, calcium silicate phases are found to be present in all the evaluated slags. The presented XRD patterns could be useful for designing slags targeted for different applications. For example, Ti and Sc could be recovered directly from the mildly fluxed slags by acid leaching, alternatively Al could be recovered first from the highly fluxed slags via caustic leaching followed by recovery of Ti and Sc by acid leaching of the residue.

Different cooling rates present the possibility for a range of crystallinity of the slag which in turn could lead to efficient slag leaching, examples include a relatively glassy slag produced from fast cooling; a self-decrepitating slag produced via slow cooling in the presence of self-disintegrating slag phases in sufficient amounts. A self-disintegrat-
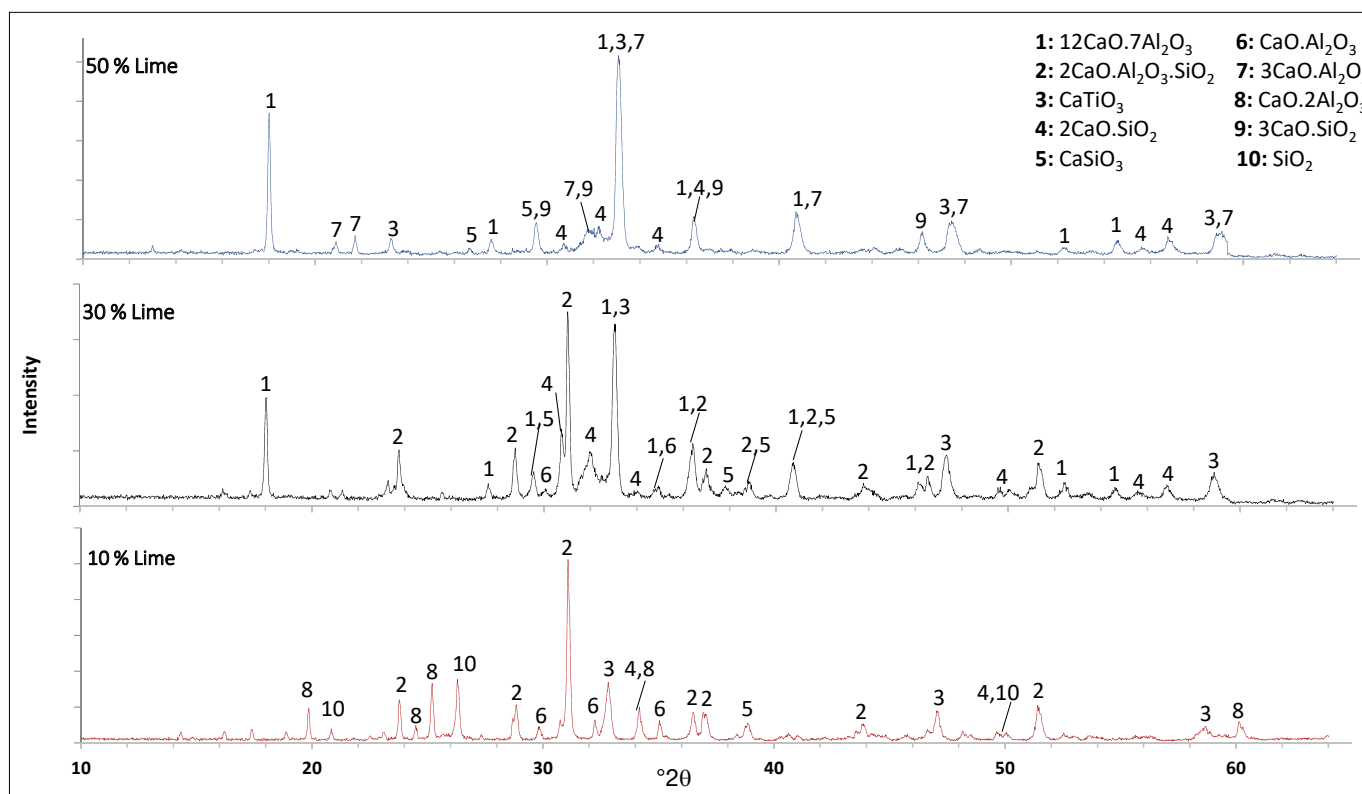
Fig. 9:   XRD patterns (Cu Kα_1 radiation, $\lambda = 1.54060 \cdot 10^{-10}$ m) of the fluxed slags under different lime additions

ing slag could be an attractive form of the slag for leaching as this could translate to a reduction or elimination in energy requirements associated with slag comminution for downstream leaching. In some cases a low crystalline slag could be preferred for downstream processing. The concentration of Sc in the slag phase is also of particular interest for the extraction of Sc, a slow cooled crystalline slag could result in the concentration of Sc in specific slag phases rather than being randomly distributed in the slag, and an understanding of this could complement the hydrometallurgical processing for Sc recovery.

One of the main reasons for the EAF treatment is to remove Fe before Ti and Sc recovery processes. This is due to its problematic behaviour during hydrometallurgical processes. The presence of high Fe content in the system is not only causing high acid consumptions during leaching but also making the processing of the aqueous solutions harder. It was reported previously that, during Ti and Sc recovery from the aqueous system by hydrometallurgical processes, Fe is the main impurity that behaves with Sc and Ti. It is co-extracted during liquid extraction routes and co-precipitate in precipitation processes. Moreover, during purification steps it is harder to remove Fe than the other impurities. Since the Fe content in the red mud was minimized before processing it hydrometallurgically, these problems should be almost avoidable in this proposed route.

5   Assessment and conclusion

Different mineral acids were compared to achieve high Sc and Ti leach efficiency. The investigation showed that

sulfuric acid was the best among the mineral acids of interest. However, acid consumption, selectivity of the targeted metals were low and silica gel formation was observed in the obtained leached samples. To attain better leaching properties and higher efficiencies with lower acid consumption pretreatment of the red mud was proposed.

Smelting of red mud offers the possibility to recover iron to the metal phase, and to concentrate other major and minor elements in the slag. The majority of the Fe in the red mud was recovered to the pig iron as expected. Minimal desulphurisation was achieved as the CaO content in the slag was low. A high liquidus slag produced, mainly due to the high alumina content. The slag reported a high sodium content and a high silica content, furthermore Ti and Sc were concentrated in this slag.

Similar leaching procedures were applied directly to this slag and the same mineral acids were compared in the same manner. It was shown that the acid needed to get the same amount of leaching efficiency was drastically decreased while the selectivities of both Sc and Ti were increased. Without chemical and physical optimization of EAF slag, very promising results were achieved. When compared with direct leaching, lower acid molarities and higher solid to liquid ratios were utilized to get similar leaching efficiencies of Ti and Sc.

The lime fluxed slag showed a lowered smelting temperature to the 1500 to 1550 °C range. High iron recoveries to the metal phase and desulphurisation of the metal was achieved. The preliminary results give an indication to achieving designed slag phase chemistry which could be tailored to recover the elements of interest. By adjusting

slag cooling rates for crystallinity and chemistry over and above flux addition adjustments would be a promising way for stepwise utilisation of red mud. Furthermore, the formation of the problematic silica gel in the aqueous samples was almost completely prevented. Based on our previous study, a precipitation route was proposed to recover Ti and Sc with relatively high purity directly from the leachate, which makes further processing much easier for achieving a high quality product.

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