



Proceeding Paper Viable Scandium Extraction from Bauxite Residue at Pilot Scale ⁺

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Abstract: Scandium extraction from the Greek bauxite residue has been demonstrated at an industrial pilot plant at MYTILINEOS. This work comes as the result of the H2020 SCALE collaborative research project. By combining sulfuric acid leaching and II-VI Selective-Ion Recovery (SIR) technology, a 22 wt% Sc concentrate has been produced from the Greek Bauxite Residue that contains 70–100 mg/kg Sc. As detailed in this work, the year-long pilot campaign achieved low acid consumption, high Sc selectivity over Fe and Ti and high Sc-loading capacity of the SIR resin, setting the basis for a viable Sc extraction technology from bauxite residue. Furthermore, the neutralized bauxite residue from the leaching process can be used in cement production, enhancing the sustainability of the operation.

Keywords: Scandium; Bauxite Residue; leaching; ion-exchange



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1. Introduction

'Bauxite Residue' (BR) refers to the insoluble solid material generated during the extraction of alumina (Al₂O₃) from bauxite ore using the Bayer process. When bauxite ore is treated with caustic soda, the aluminium hydroxides/oxides contained within are solubilized, with approximately 50% of the bauxite mass being transferred to the liquid phase, while the remaining solid fraction constitutes the -bauxite- residue. It is estimated that for each ton of alumina produced, 0.9–1.5 tons of solid residue (on a dry basis) is generated depending on the initial bauxite ore grade and alumina extraction efficiency [1]. Bauxite residue contains various major (gr/kg) oxides of Fe, Al, Ti, Ca, Si, Na, as well as minor (mg/kg) oxides of V, Ga, REE/Sc and others (depending on the initial chemical composition of the bauxite ore), along with inclusions of unwashed sodium aluminate solution.

The worldwide typical concentration of REE in BR is 800–2500 mg/kg and is related to the initial bauxite ore and the operation conditions of the Bayer process [2]. Typically, REE are hosted at alumina-bearing minerals of the bauxite ore, which are dissolved during the Bayer process; the contained REE are incorporated into secondary precipitation phases known as "desilicaton products" or "DSP", a mineral matrix that contains a mixture of Fe, Ti, Si, Al Ca and Na ions [3].

Scandium often differs from REE in its mineral behavior; especially in lateritic bauxites and their corresponding BR, it is often related to iron and titanium and zircon minerals [3–5], which for the most part are unaffected through the Bayer process. This is also confirmed by the laterite deposits in Australia and the Greek BR [6], where the main mineral with high Sc content is goethite [7]. However, there are cases of BR where scandium is related to a larger extent to soluble Al-bearing minerals [8]. Scandium is a very rarely concentrated

element, making commercially usable deposits of this critical material very rare. As a result, it is usually extracted as a by-product from the metallurgical residues from the primary extraction of TiO_2 from ilmenite and other ores, Nickel-Cobalt from laterites, U_3O_8 from various ores, Rare Earth oxides and others. It has been estimated that 70% of the world's Sc resources might be found in bauxite minerals and bauxite residue [9].

Direct leaching of BR with mineral acids [10–13] requires significant acid consumption, as BR is by nature highly alkaline and results in leach solutions with low Sc concentrations (<20 mg/L), while major BR metals such as Fe, Al, Ti, Ca, Na and Si dissolve extensively and are thus found in concentrations of several g/L. This complicates Sc extraction and refining from such solutions. Applying selective leaching on BR is the first step to generating a REE solution with significantly lower concentrations of unwanted co-dissolved metals. Mineral acid leaching performance for the Greek BR in terms of increasing selectivity of Sc against Fe, which is usually the main BR component, follows the trend of HNO₃ > H₂SO₄ > HCl [10]. In general, the recovery rate of Sc during selective leaching is limited in the range of 20–50% with Fe dissolution <3% [11,14–16]. In most cases a relatively limited selective dissolution against Fe, Ti and Si can be achieved but not against readily dissolved Ca, Al and Na.

The present work, under the H2020-SCALE project, focuses on developing an industrial operation for Sc extraction; H₂SO4 was selected as the acid to be used in BR leaching based on its Sc- selectivity, cost and ease of handling; the II-VI Selective-Ion Recovery (SIR) technology was selected as the Sc extraction technology. II-VI, headquartered in Saxonburg, Pennsylvania, USA, has research and development, manufacturing, sales, service, and distribution facilities worldwide. The company produces a wide variety of application-specific photonic and electronic materials and components and deploys them in various forms. The company has developed the II-VI Selective-Ion Recovery (SIR) technology, which includes the use of a composite extractant-enhanced ion-exchange resin to extract scandium (Sc) from acidic solution or slurries and its subsequent recovery as Sc concentrate [17,18]. The factors that contribute to its performance have been studied to a great extent using Pregnant Leach Solution (PLS) from various Sc-containing sources. The leaching conditions of BR were evaluated and optimized to improve the recovery of Sc using this technology.

Earlier exploratory leaching experiments [19–21] showed that (i) the dissolution of impurities increases with increasing acid concentration during leaching, and (ii) the solubility of silica in solution increases as acidity decreases, leading to silica gel formation over time. For the proper operation of ion exchange columns, it is important to ensure that there is no precipitation in the column to avoid pressure drop and fouling in the column.

Pilot leaching scale tests coupled with lab scale SIR testing of the optimum leaching conditions for maximizing loading of Sc while maintaining steady operation of SIR were established [22]. The leaching conditions were selected based on criteria of cost and effectiveness of the produced PLS in the subsequent ion-exchange process to extract Sc into a marketable concentrate. To achieve a low cost of leaching, sulfuric acid was used at high pulp densities, while controlling leaching of impurities such as Fe and Ti in the PLS. A year-long pilot campaign followed in which more than 10 t of bauxite residue were leached, producing 14 m³ of pregnant liquid solution (PLS), 10 m³ of which were processed with SIR to produce a 22%wt crude Sc concentrate. This paper describes this pilot campaign.

2. Experimental

The MYTILINEOS acid-leaching pilot plant consists of three polypropylene reactor tanks of up to 800 L capacity, with mechanical steering and heating/cooling through immerged coils for circulating steam and cooling water, respectively. Filter-pressed BR produced at MYTILINEOS alumina refinery is mixed with industrial water in the first reactor (100-TK-10) to produce a pulp of specific density measured through an inline Coriolis Mass Flow Meter. The pulp is pumped to the second reactor (100-TK-30), where it

is heated and contacted with concentrated sulfuric acid. The leaching takes place at 85 °C with a retention time of 30 min. The pulp exiting the 100-TK-30 is driven to the cooling tank (200-TK-40), where it is cooled to 60 °C and is subsequently passed to the filter press circuit. The filter press separates solids from liquid, generating the final PLS to be used in the SIR process. The filter press used consists of 25 frames of 470 × 470 mm with 11 chambers plates and 12 membranes plates and a total filter area of 6.6 m². Inlet slurry is drawn via a diaphragm pump with a maximum working pressure of 15 bar. The filter cake washing was conducted directly on the filter press with fresh water inserted through the inlet slurry and directed to the washate-receiving tank. Cake squeezing and cake air blowing were also applied in the filter press.

A full leaching run at the MYTILINEOS pilot, operating at a final pulp containing a 40% solids to liquids (wt/vol) unit involves a daily treatment of approximately 236 kg of BR in an 8 h daily shift that produces around 360L of PLS and 236 kg of dry cake from a 2 full cycle filter press operation. For each test, operators were responsible for sampling for chemical analysis in the lab, SCADA daily data management and resolution of issues that arose during production and equipment malfunction.

In all experiments, BR filter cake from the MYTILINEOS Alumina Refinery were used. The chemical composition of the BR were determined by alkaline fusion and wet chemical analysis by ICP-OES and AAS (Table 1). The mineralogical analysis of BR is shown in Figure 1.



Figure 1. Cont.



Figure 1. Top: XRD comparisons of bauxite residue and leached bauxite residue (filter cake). Phases identified 1: Hematite (Fe₂O₃), 2, Cancrinite (H_{0.88}Na₈Al₆(SiO₄)₆(CO₃)_{1.44}(H₂O)₂), 3, Diaspore (AlO(OH), 4, Gibbsite (Al(OH)₃), 5, Boehmite (AlO(OH)), 6, Perovskite (Ca(TiO₃)), 7, Calcite (Ca(CO₃)), 8, Katoite (Ca₃Al_{3.5}O_{4.5}(OH)_{7.5}), 9, Goethite (FeO(OH)), 10, Rutile (TiO₂), 11, Anatase (TiO₂), 12, Quartz (SiO₂), 13, Chamosite ((Mg_{1.5}Fe_{7.9}Al_{2.6})(Si_{6.2}Al_{1.8}O₂₀)(OH)₁₆), 14, Bassanite (Ca₂(SO₄)₂(H₂O)).; **Bottom:** XRF analysis of leached bauxite residue (filter cake).

Table 1. BR's Chemical composition.

wt%							ppm (mg/kg)				
Fe ₂ O ₃	Al ₂ O ₃	SiO ₂	CaO	Na ₂ O	TiO ₂	LOI	Ce	La	Y	Sc	
38.73	24.13	7.65	8.03	3.58	5.00	10.10	657	110	132	71	

Three liquid samples were taken daily directly from the leaching reactor and analyzed by ICP-OES for their Sc, Fe, and Ti concentration. Cake samples obtained from the filter press were analyzed by XRF analysis.

The II-VI SIR pilot plant consists of a series of 15 L ion-exchange columns fed from two 5 m³ PLS holding tanks at a rate of 30 L/h. The raffinate exiting the columns was sampled twice per day and analyzed by ICP-OES. During elution of the columns, the eluate was collected and neutralized in a separate reactor and a smaller filter press (up to 6 bar in operation) was used to achieve solid–liquid separation. Solids were analysed by XRF and liquid samples by ICP-OES.

3. Results

3.1. Bauxite Residue Leaching

Initial leaching tests showed that 0.28 kg acid/kg BR is the optimum reagent/feed ratio to achieve high Sc loading through SIR. Leaching conditions in the reactor were set to 65% wt water, 27% wt BR and 8% wt conc. H_2SO_4 (98%). The leaching results under conditions applied in the pilot processing of bauxite residue give an average Sc extraction to PLS of 20.5%, whereas Ti is extracted at 0.19% and Fe at 0.12%, as seen in Figure 2. The daily results of each test run are shown in Figure 3, where the daily consumption of BR, water and acid are provided, together with the PLS and dry cake production.



Figure 2. Leaching extraction (%) of Sc (top), Fe, Ti (bottom) achieved in pilot tests.



Figure 3. Leaching pilot data of inputs (BR, water, H₂SO₄) and outputs (PLS, filter cake).

The mineralogical comparison of the initial BR and the leached BR filter cake are presented in Figure 1.

A new mineral phase of bassanite due to CaSO₄ formation has been identified after BR leaching, whereas cancrinite and katoite, i.e., the desilication products produced during the

Bayer process, are not detected. The chemical analysis of inputs and outputs extrapolated to an overall mass balance is provided in Table 2.

1 t BR Input			1 t Cake Output	1.66 t PLS			% Extraction in PLS		
Al (kg)	127.71	kg	116.58	kg	11.12	kg	8.71	Al	
Fe (kg)	270.84	kg	270.51	kg	0.33	kg	0.12	Fe	
Ca (kg)	57.36	kg	54.90	kg	2.46	kg	4.28	Ca	
Si (kg)	35.72	kg	35.36	kg	0.36	kg	1.01	Si	
Ti (kg)	29.99	kg	29.94	kg	0.06	kg	0.19	Ti	
Na (kg)	26.56	kg	3.93	kg	22.63	kg	85.20	Na	
Sc (g)	83.28	g	66.13	g	17.15	g	20.59	Sc	

Table 2. Mass balance based on BR leaching campaign to pls and cake production.

3.2. Processing PLS with SIR

The PLS generated form BR leaching was transferred to the two main PLS holding tanks of the II-VI SIR unit. The holding tanks have a capacity of 5 m³ each. The chemical content of representative samples from the tanks is provided in Table 3. The PLS in both tanks had a pH between 3.5–3.7 and a turbidity <20 NTU. PLS from both tanks was fed into an SIR column at a rate of 30 L/h. The loading results are shown in Figure 4.

Table 3. Main metal concentration in the PLS prior to loading to the SIR column (two 5 m³ tanks).

(mg/L)	Fe	Ti	Al	Si	Ca	Sc	Ce	Ŷ	La
Tank 2a PLS	275	33	6530	127	590	12.8	8.9	7.5	3.4
Tank 2b PLS	278	29	6400	159	603	10.8	9.9	8.0	4.3

The loading of the SIR for Sc at C/C_o of 0.65 (where C is the measured concentration of the solution exiting the column and C_o the concentration of the solution entering the column) was about 3500 mg/L. The results clearly show that the resin did not reach the exhaustion point where C/C_o would expect to be close to 1. By projecting the curve, the full loading capacity could have been between 4500 and 5000 mg/L, which would be close to the capacity found in previous laboratory tests [22]. The Sc was eluted from the resin and the recovery was 93%. The eluate was found to have 865 ppm of Sc and 521 ppm Ti. With succeeding pH adjustments of the eluate, precipitation of Sc(OH)₃ concentrate was achieved. Upon completion of the filtration, the resulting precipitate weighed about 910 g, and after thorough washing and drying the resulting Sc conc. weighed 192 g (Figure 5). All Sc precipitated since no Sc was found in the raffinate solution. The final concentration of the concentrate is presented in Table 4.

Table 4. Crude Sc concentrate chemical analysis.

	Sc	Ti	Fe	Na	Al	Si	LOI
%wt	22.0	15.2	0.54	1.0	1.3	5.8	32.0

Through the leaching and purification pilot work, Sc was concentrated more than 2500 times from BR to crude Sc concentrate. The significant increase in Sc content substantially reduces the sizing requirements for downstream purification steps and presents a financially viable approach to the recovery of scandium from bauxite residue.



Figure 4. Loading (mg of cation/L of resin) and adsorption (concentration of solution exiting the column to initial PLS concentration) curves for Sc, Fe and Ti against number of Bed Volumes (BVs) passed through the resin column.



Figure 5. Sc crude conc. 22% produced at MYTILINEOS-II-VI pilot plant.

4. Conclusions

A new hydrometallurgical pilot unit was built in MYTILINEOS Aluminium of Greece plant. The pilot unit consists of the leaching section where bauxite residue was treated with sulfuric acid to produce PLS, which was then processed in the II-VI SIR pilot unit to recover Sc and produce crude Sc concentrate.

The pilot leaching unit has a daily operation process of 236 kg of dry BR to produce around 4 g of Sc into PLS (at 10–14 mg/L Sc). During the leaching campaign, 42 tests were conducted and almost 10 t of BR were treated, thus producing 14 m³ of PLS in the specification for the SIR purification technology. A total of 140 g of Sc was present in the resulting leachate for purification. From 1 t of BR filtercake, 17.15 g Sc is dissolved into PLS consuming 0.27 t H₂SO₄ and 2.36 m³ water.

Subsequently, 10 m³ PLS of 8–12 mg/L Sc was passed through one column of the SIR at a flowrate of 30 L/h. The loading of the SIR for Sc at C/C_0 of 0.65 was about 3500 mg/L. The results indicate that the resin did not reach the exhaustion point where C/C_0 is close to 1. By projecting the curve, the full loading capacity could have been between 4500 and 5000 mg/L, which would be close to the capacity obtained in previous laboratory tests performed at II-VI.

The first stage of elution of the loaded resin resulted in a solution containing 865 mg/L Sc, which in turn was neutralized to produce crude Sc hydroxide concentrate containing 22% Sc. Through the leaching and purification pilot work, Sc was concentrated more than 2500 times from BR to crude Sc concentrate. The significant increase in Sc content substantially reduces the sizing requirements for downstream purification and presents a financially viable approach to the recovery of scandium from bauxite residue.

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