

Developing a Parallel-to-Aluminium Value Chain for Scandium and Al-Sc Alloy Production. Pilot Scale Results under the SCALE Project

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Abstract

Bauxite residue produced from the Bayer process from alumina production has been identified as a potential source for scandium extraction. Scandium found in the initial bauxite ore is concentrated in the bauxite residue (BR). In the case of the Greek BR, scandium concentration varied between 80-120 mg/kg over the past seven years. With an annual production of 850 kt BR at MYTILINEOS alumina refinery, 70-80 t of Sc dissipate each year in the BR by-product stream. Under the 4-year collaborative H2020 SCALE research project, a novel process to extract Sc from BR was developed and evaluated in an industrial pilot scale. Over 10 t of BR was leached with sulfuric acid solution to produce a low impurity Sc pregnant leach solution (PLS) suitable for ion-exchange extraction, utilizing the novel proprietary “Selective Ion Recovery (SIR)” process, developed by II-VI. A crude concentrate was produced using the SIR technology to upgrade scandium by more than 2500 times into an intermediate concentrate having 22 % Sc, 15 % Ti and minor impurities. The crude concentrate at this Sc level would be a suitable feed material for pure Sc₂O₃ / ScF₃ production using existing technologies. The leached BR was neutralized and depleted in soda, making this material more attractive for BR reuse options such as cement and iron production. The end goal of Sc recovery from BR is the production of Al-Sc alloys, the use of which is expected to rapidly increase in the next decade, given their impressive properties in strength, weldability, corrosive resistance, and application in 3D printing. Lab-scale electrolysis trials at SINTEF in a cryolite melt with Al₂O₃ and Sc₂O₃ as feed material, successfully led to the production of Al metal with ~2 % Sc at current efficiencies ranging from 85 to 90 %. Thus, a new parallel-to-aluminium value chain for Sc production can be established.

Keywords: Scandium, bauxite residue, leaching, ion-exchange, Al-Sc alloys.

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 aluminum 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) are generated depending on the initial bauxite ore

grade and alumina extraction efficiency [1]. Bauxite residue contains various major (g/kg) oxides of Fe, Al, Ti, Ca, Si, Na, as well as minor (mg/kg) oxides like 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 in 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 “desilication products - DSP”, a mineral matrix that contains a mixture of Fe, Ti, Si, Al, Ca and Na ions [3]. Scandium often differs from the other REE in its mineral behavior; especially in lateritic bauxites and their corresponding BR, it is often correlated with iron, 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 concentration of Sc, is goethite [7]. However, there are cases of BR, where scandium is related to larger extent to the soluble Al-bearing minerals [8]. It has been estimated that 70% of the world's Sc resources might be found in bauxite minerals and bauxite residue [9].

The present work, under the H2020-SCALE project, focuses on developing a novel value chain from BR to Al-Sc master alloy, that runs parallel to that of the primary aluminium. The process starts with BR as the Sc source, from which Sc is selectively leached into solution with sulfuric acid [10-13]; followed with the II-VI Selective-Ion Recovery (SIR) technology [14-15] to extract Sc from the leach solution and produce a crude Sc concentrate, suitable for Sc₂O₃ oxide production based on established technologies; and concludes with Al-Sc metal production through co-electrolysis of Al₂O₃ and Sc₂O₃ in the Hall-Heroult cell [16]. Following small pilot leaching scale tests coupled with lab scale SIR testing, the optimum leaching conditions to maximize loading of Sc while maintaining steady operation of SIR were established [13]. A year-long pilot campaign followed at MYTILINEOS, 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. In parallel, lab scale and bench scale tests were conducted to achieve and validate the modified Hall-Heroult at SINTEF.

2. Experimental

The MYTILINEOS acid leaching pilot plant consist of a series of PP (Polypropylene) reactor tanks, at 800 L capacity, with mechanical steering and heating/cooling through immersed 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 minutes. 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 the solids from the liquids, generating the final PLS to be used for the SIR process. Filter press used consists of 25 frames 470x470 mm having 11 chambers plates and 12 membranes plates and a filter area of 6,6 m². Inlet slurry pump is a diaphragm pump with a maximum working pressure at 15 bar. In the filter press the cake washing is conducted directly, as fresh water is pumped through the cake and collected at a separate tank. Cake squeezing, and cake air blowing are also applied in the filter press, before the cake discharge.

Table 1. BR's Chemical composition.

| % | | | | | | | 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 |

A full leaching run at the MYTILINEOS pilot plant, involves a daily treatment of approximately 236 kg of BR in an 8-hour daily shift that produces around 360 L of PLS and 236 kg of dry cake from a 2 full cycle filter press operation. The final pulp contains 40% solids to liquids (wt/vol) ratio. At each test, operators are responsible for sampling for chemical analysis in laboratory, SCADA daily data management and resolution of issues that arise during production and equipment malfunction. In all experiments, BR filter cake from the MYTILINEOS'S Alumina Refinery were used. The chemical composition of the BR as determined by alkaline fusion and wet chemical analysis by ICP-OES and AAS and is shown in Table 1, the mineralogical analysis of the BR is shown in Figure 4. Three liquid samples were taken daily directly from the leaching reactor, 1st, and 2nd filter press cycle. PLS respectively was measured with ICP-OES for Sc, Fe and Ti content. The cake samples obtained were analyzed for elemental composition using XRF.

The II-VI SIR pilot plant consist of a series of 15 L ion-exchange columns that are 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 using 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 analyzed with XRF and liquid samples with ICP-OES.

The experimental electrolysis cell consisted of a vertical furnace that housed a graphite crucible as electrolyte container with Si₃N₄ side-lining. The bottom of the graphite crucible or steel and TiB₂ disks were used as cathodes, whereas graphite was used as anode material, inserted from above leading to a horizontal electrode arrangement. The electrolyte was a cryolite-based melt (Na₃AlF₆) with a cryolite ratio (CR=NaF/AlF₃) equal to 2.2 containing 10 % Sc₂O₃ at an operating temperature of ~ 980 °C, which was measured and monitored with a type S thermocouple. The corresponding amounts of Al₂O₃ and Sc₂O₃ were added through the course of the electrolysis to keep the melt at oxide saturation levels.

3. Results

3.1 Bauxite Residue Leaching

Initial leaching tests showed that 0.28 kg acid/kg BR is in optimum range to achieve high Sc loading through SIR. Leaching conditions in the reactor were set to 65 % water, 27 % BR and 8 % concentrated H₂SO₄ (98 %). The leaching conditions applied in pilot processing of bauxite residue indicate that Sc extraction takes place on average at 20.5% to PLS, whereas Ti is extracted at 0.19 % and Fe at 0.12 %, as seen in Figure 1. The daily results on each test run are indicated in Figure 2 where daily consumption of BR, water and acid are provided, while the PLS and dry cake production are also depicted. The overall pilot data obtained during the campaign are given in Table 2. The properties of the leached BR filter cake are presented in Figure 3 and Table 3.

Table 2. Summary of daily pilot data produced from leaching campaign.

| Data | Average | Min | Max |
|--|---------|--------|--------|
| total BR input (kg) | 236.63 | 229.18 | 243.59 |
| total water input (kg) | 558.49 | 540.92 | 574.93 |
| total PLS produced (L) | 356.92 | 300.00 | 400.00 |
| Total Cake (kg dry) | 236.86 | 214.92 | 258.49 |
| PLS m ³ / t BR | 1.51 | 1.31 | 1.64 |
| BR /cake | 1.00 | 1.07 | 0.94 |
| PLS m ³ / t cake | 1.51 | 1.40 | 1.55 |
| Specific energy consumption (kWh / t KB) | 336.05 | 308.83 | 366.45 |
| Specific energy consumption (kWh / t PLS) | 223.87 | 184.66 | 283.54 |
| Acid consumption (H ₂ SO ₄ /KB, t/t) | 0.27 | 0.27 | 0.28 |

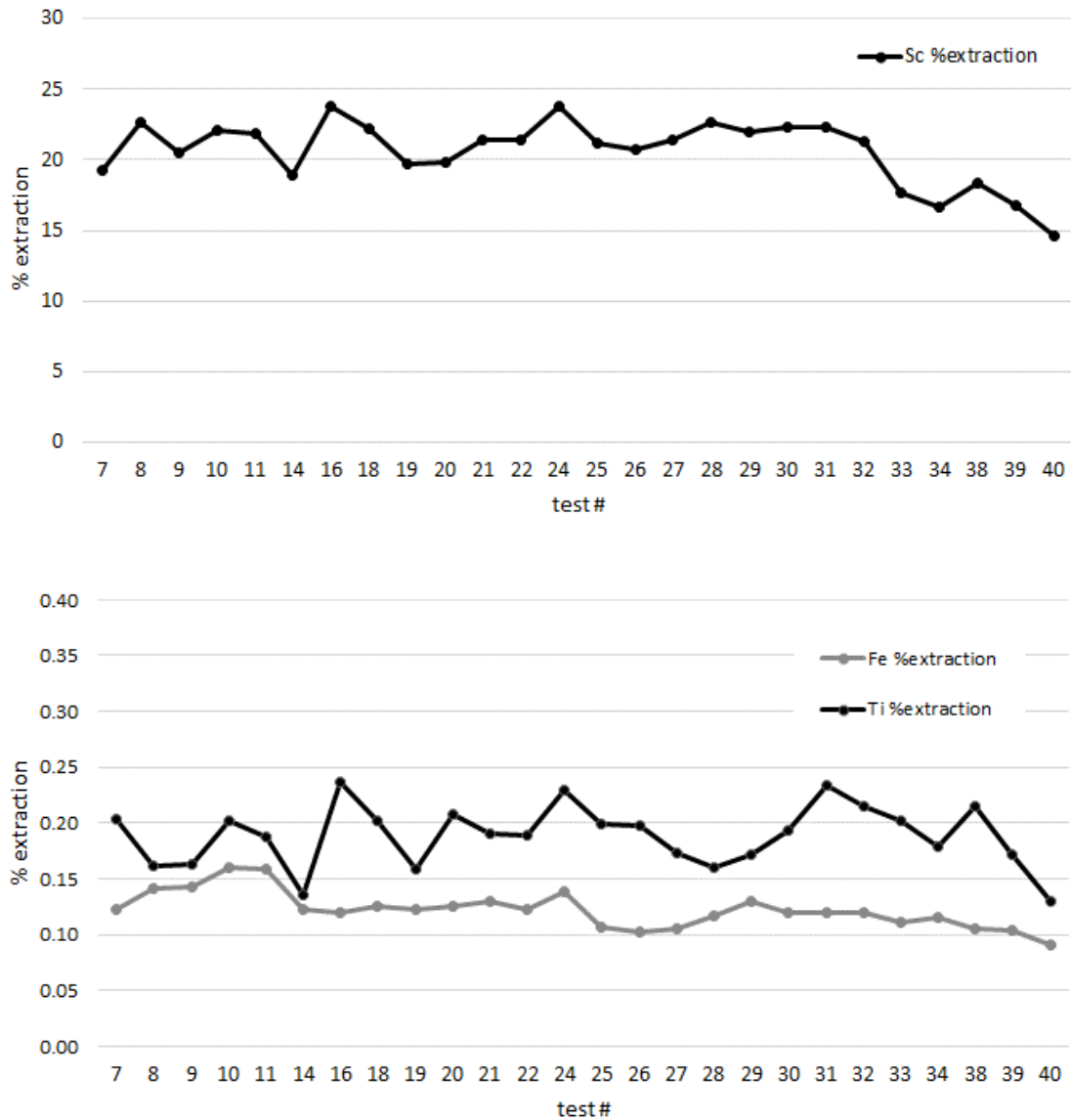


Figure 1. Leaching extraction (%) of Sc, Fe, Ti in PLS achieved in pilot tests.

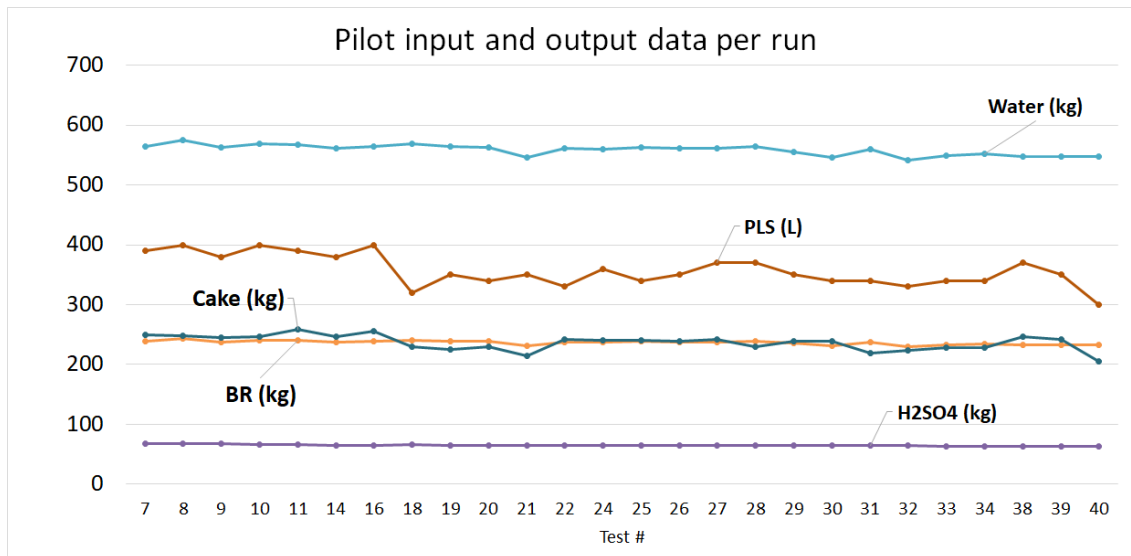


Figure 2. Leaching pilot data of inputs (BR, water, H₂SO₄) and outputs (PLS, filter cake).

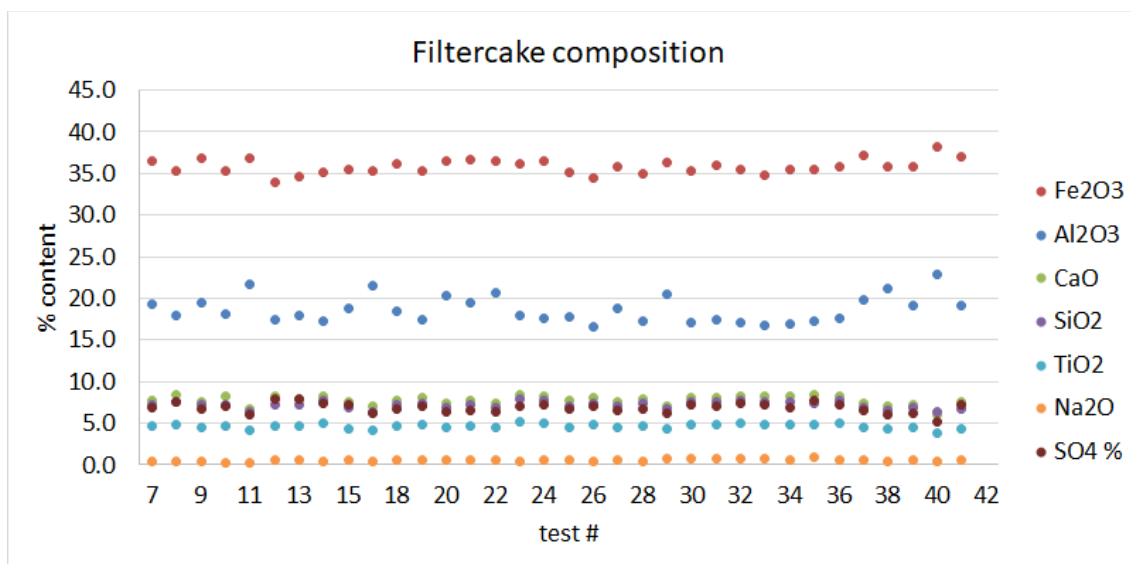


Figure 3. XRF data in cake produced from BR leaching with H₂SO₄ at 85°C, 1h, 40% wt/v pulp, pH 2.3.

Table 3. Indicative chemical analysis (XRF) of the cakes produced (%).

| | Al ₂ O ₃ | Fe ₂ O ₃ | CaO | SiO ₂ | TiO ₂ | Na ₂ O | SO ₄ |
|------------|--------------------------------|--------------------------------|-----|------------------|------------------|-------------------|-----------------|
| min | 16.6 | 34.4 | 6.0 | 6.3 | 3.8 | 0.3 | 5.1 |
| max | 22.9 | 38.1 | 8.4 | 7.8 | 5.0 | 0.9 | 7.8 |
| Average | 18.6 | 35.9 | 7.8 | 7.2 | 4.6 | 0.6 | 6.8 |
| Initial BR | 24.1 | 38.7 | 8.0 | 7.6 | 5.0 | 3.6 | 0.5 |

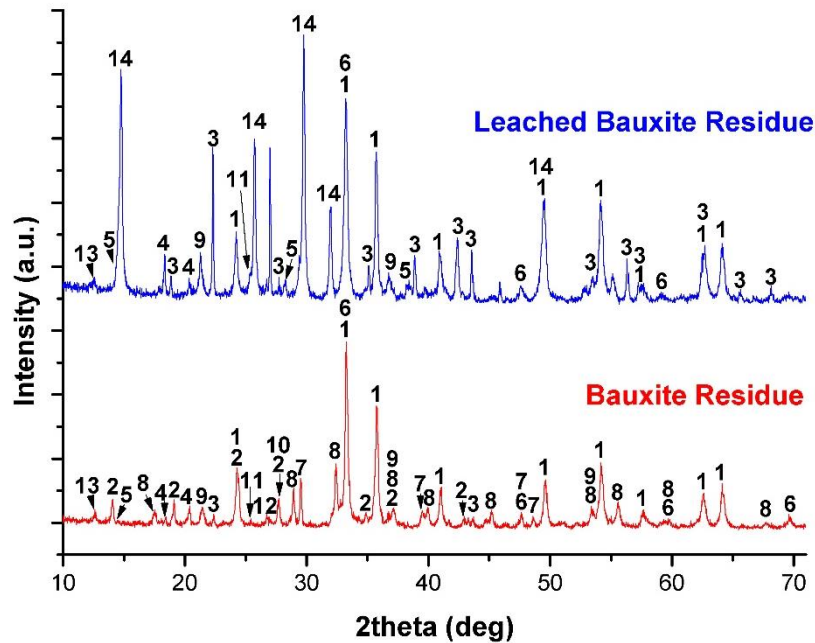


Figure 4. XRD data comparison of bauxite residue and leached bauxite residue cakes. Phases identified 1 : hematite - (Fe₂O₃), 2: cancrinite - H_{0.88}Na₈Al₆(SiO₄)₆(CO₃)_{1.44}(H₂O)₂, 3: diaspore - AlO(OH), 4: gibbsite - Al(OH)₃, 5: boehmite - AlO(OH), 6: perovskite - Ca(TiO₃), 7: calcite - Ca(CO₃), 8: 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).

A new mineral phase of bassanite due to CaSO₄ formation has been formed after BR leaching whereas cancrinite and katoite, i. e., the desilication products produced during the Bayer process, are dissolved. Chemical analysis of all inputs and outputs extrapolated to an overall mass balance provided in Table 4.

Table 4. Elemental mass balance based on BR leaching campaign to PLS and cake production.

| Element, kg | INPUT 1 t of dry BR | INPUT 0.27 t H ₂ SO ₄ +2.36 t H ₂ O | OUTPUT 1 t Cake | OUTPUT 1,66 t PLS | Extraction yield in PLS, % |
|-------------|------------------------|--|--------------------|----------------------|----------------------------------|
| Al | 127.71 | | 116.58 | 11.12 | 8.71 |
| Fe | 270.84 | | 270.51 | 0.33 | 0.12 |
| Ca | 57.36 | | 54.90 | 2.46 | 4.28 |
| Si | 35.72 | | 35.36 | 0.36 | 1.01 |
| Ti | 29.99 | | 29.94 | 0.06 | 0.19 |
| Na | 26.56 | | 3.93 | 22.63 | 85.20 |
| Sc | 0.08328 | | 66.13 | 0.01715 | 20.59 |
| S | | 93.96 | 22.40 | 71.56 | 76.16 |

3.2 Processing PLS with SIR

The PLS generated from BR leaching was transferred to the 2 main PLS holding tanks of the II-VI SIR unit. The holding tanks have capacity of 5 m³ each. The chemical content of representative samples from the tanks is provided in Table 5. The PLS in both tanks had a pH between 3.5-3.7 and turbidity <20 NTU. The PLS was acidified to 20-30 g/L with H₂SO₄ to avoid precipitation and fouling in the columns during loading. Then, the PLS from both tanks was fed into a SIR IX column at the rate of 30 L/h. The loading results are plotted in Figure 5.

Table 5. 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 | Y | 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/Co of 0.65 was about 3,500 mg/L. The results clearly show the resin did not reach the exhaustion point where C/Co would expect to be close to 1. By projecting the curve, the full loading capacity could have been between 4,500 and 5,000 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 steps of pH adjustment of the eluate the Sc(OH)₃ concentrate precipitation was achieved. Upon completion of the filtration step the resulting precipitate weighs about 910 g and after thorough washing and drying the resulting Sc concentrate weighs 192 g (Figure 6). All Sc precipitated since no Sc was found in the raffinate solution. The final concentration of the concentrate is presented in Table 6.

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

Table 6. Chemical analysis of crude Sc concentrate.

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

Based on the loading curves, the number of IX columns were calculated to maximize Sc recovery while allowing the maximum resin utilization. Maximizing resin utilization would lead to lower reagent consumption for elution, scrubbing & regeneration, and subsequent waste treatment and lower capital cost as it would reduce the size of all tanks and columns. Considering all data to date, 6 columns are needed in lead/lag configuration to allow the exhaustion of resin in the first column, while in the fifth column, the resin is reaching the breakthrough point. Once the first column is exhausted, it will proceed to washing, elution and regeneration, while the 2nd column will be placed in the lead position and the column 6 becomes the last column in loading. Through operation, five columns will be in the loading position, while one column is being eluted and regenerated to be ready for the next cycle.

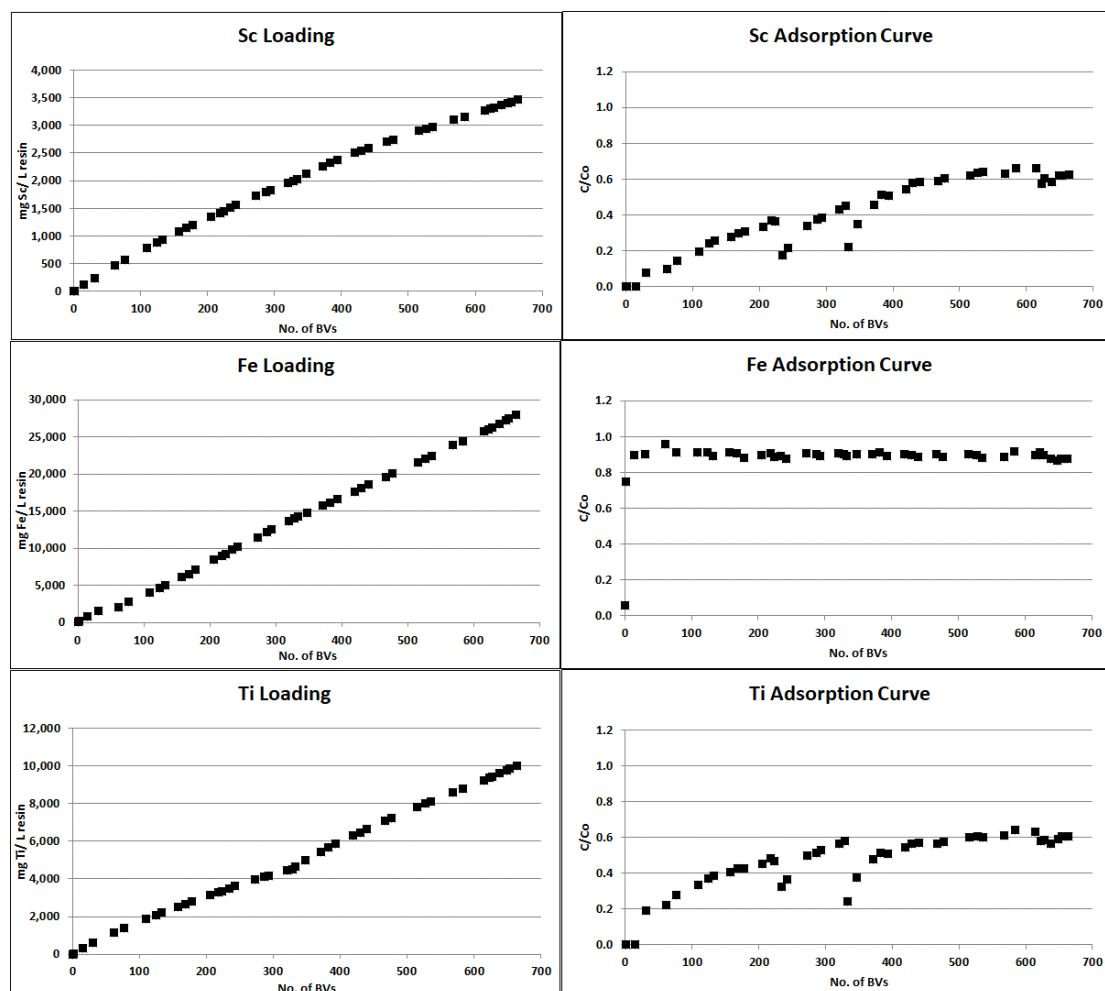


Figure 5. Loading (mg of cation / l of resin) and Adsorption (concentration of solution exiting the column to initial PLS concentration) Curves for Sc, Fe, and Ti against number of Bed Volumes (BV) passed through the resin column.



Figure 6. Crude Sc concentrate (22%) produced at MYTILINEOS II-VI pilot plant.

The concentrate produced is suitable for use in the established Sc refining process, like the one practiced in II-VI plant, which can produce Sc_2O_3 of 99.99% purity. The ratio of Sc to impurities has increased significantly in the crude concentrate relative to bauxite residue. This would make the final purification through solvent extraction more effective. Additionally, to be economical, the Sc concentration in the PLS has to be above certain level (i.e., > 600 mg/l) before it is fed to the solvent extraction. If the purification is planned to be on-site, the Sc concentrate solution after elution can be directly fed to the solvent extraction, without the need to intermediately precipitate

the crude Sc concentrate. Alternatively, if Sc is not being purified on-site, Sc can be precipitated to produce the crude concentrate as it would be more practical to transport solid in lower quantity than large volume of Sc containing solution. The concentrate can then be transferred to the purification plant where it is dissolved and processed.

3.3 Producing Al-Sc in a Hall-Heroult cell

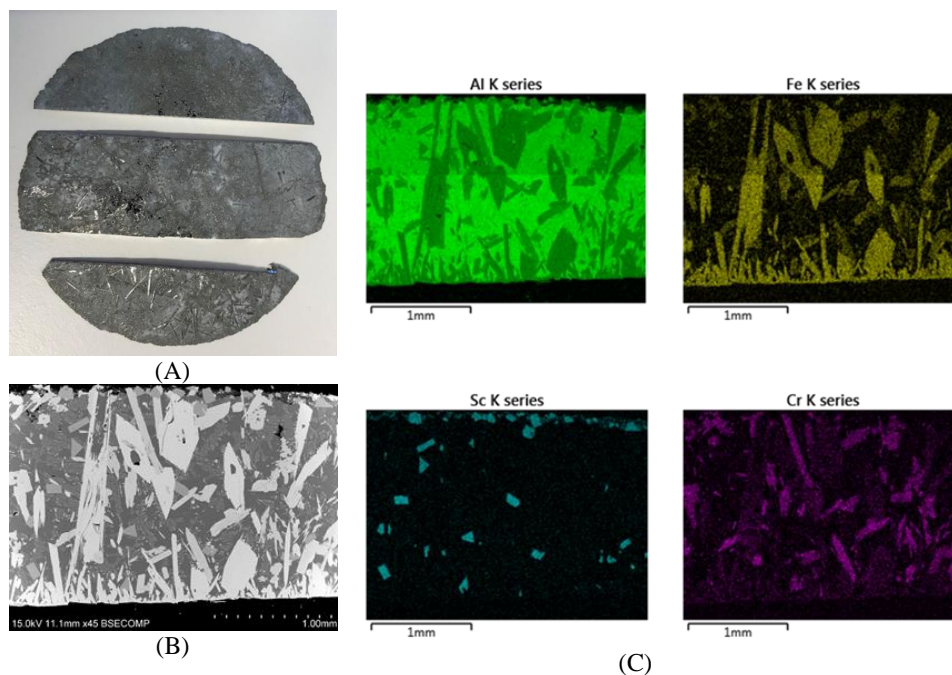


Figure 7. SEM micrograph and EDS elemental analysis of the metal products obtained when using steel as cathode material. A) cathode product once detached from the steel cathode; B) SEM micrograph of the cross-section of (A); C) EDS elemental analysis.

Electrolysis experiments were carried out at galvanostatic mode while monitoring the cell voltage and the operating temperature. In some cases, the electrolysis was run without consecutive Al_2O_3 and Sc_2O_3 feeding until anode effect was achieved. That means current blockage at the anode due to the depletion of oxide-containing species. In long term (bench scale) electrolysis, Al_2O_3 was fed using an automatic feeder from the top of the electrolysis cell, while Sc_2O_3 was added manually every 2 h. When finishing the electrolysis, the cell was cooled down for 24 h before inspection. SEM-EDS analysis of the metal products (after eliminating the electrolyte remains with an AlCl_3 solution) confirmed the presence of intermetallic Al_3Sc phases in the aluminium matrix, which were probably formed upon slow cooling of the electrolysis cell. In the case of steel cathodes, Fe and Cr dendrites could also be observed in the cathode product (cf. Figure 7), owed to its high solubility in aluminium at the operating temperature. ICP-MS analysis of the metal product showed a Sc content of $\sim 2\%$ in the Al matrix. The current efficiencies of the electrolysis process were determined by comparing the charge passed and the amount of metal harvested and were in the order of 85-90 % and 77 % in the lab-scale and bench-scale, respectively.

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 is treated with sulfuric acid to produce a PLS, which is processed in 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 ppm of Sc). During leaching campaign 42 tests were conducted where almost 10 t of BR were treated and 14 m³ of PLS was produced in the specification of SIR purification technology. A total of 140 g of Sc was present in the resulting leachate for purification. From 1 t of BR filter cake, 17.15 g Sc are dissolved into PLS consuming 0.27 t of H₂SO₄ and 2.36 t of water.

Subsequently, 10 m³ of PLS of 8-12 ppm Sc were passed through 1 column of the SIR at 30 L/h flow. The loading of the SIR for Sc at C/Co of 0.65 was about 3,500 mg/L. The results indicate the resin did not reach the exhaustion point where C/Co would be expected to be close to 1. By projecting the curve, the full loading capacity could have been between 4,500 and 5,000 mg/L, which would be close to the capacity found in the laboratory from PLS generated by employing the II-VI leaching conditions. The first stage of elution of the loaded resin resulted in a solution containing 865 ppm Sc, which in turn once precipitated produced a crude Sc hydroxide concentrate containing 22 % Sc. Through the leaching and purification pilot work, Sc was concentrated more than 2500 times from BR to a crude Sc concentrate. The significant increase in Sc content substantially reduces the sizing requirements for downstream purification steps and presents a pragmatic approach to the recovery of scandium from bauxite residue.

Electrolysis from a cryolite melt at CR=2.2 and 980 °C has demonstrated to be a suitable method to prepare Al-Sc alloy, while using the same electrolysis cell arrangement as in the current industrial aluminium production. The results showed that it is possible to electrodeposit Sc in the Al matrix due to underpotential deposition of Sc, i.e., at activities lower than 1. Intermetallic Al₃Sc phases in the aluminium matrix were found by SEM-EDS analysis of the cathode product, probably formed upon slow cooling of the cell after the electrolysis trials. Uniform distribution of Sc in the aluminium matrix is expected, owed to the electromagnetic forces created during electrolysis.

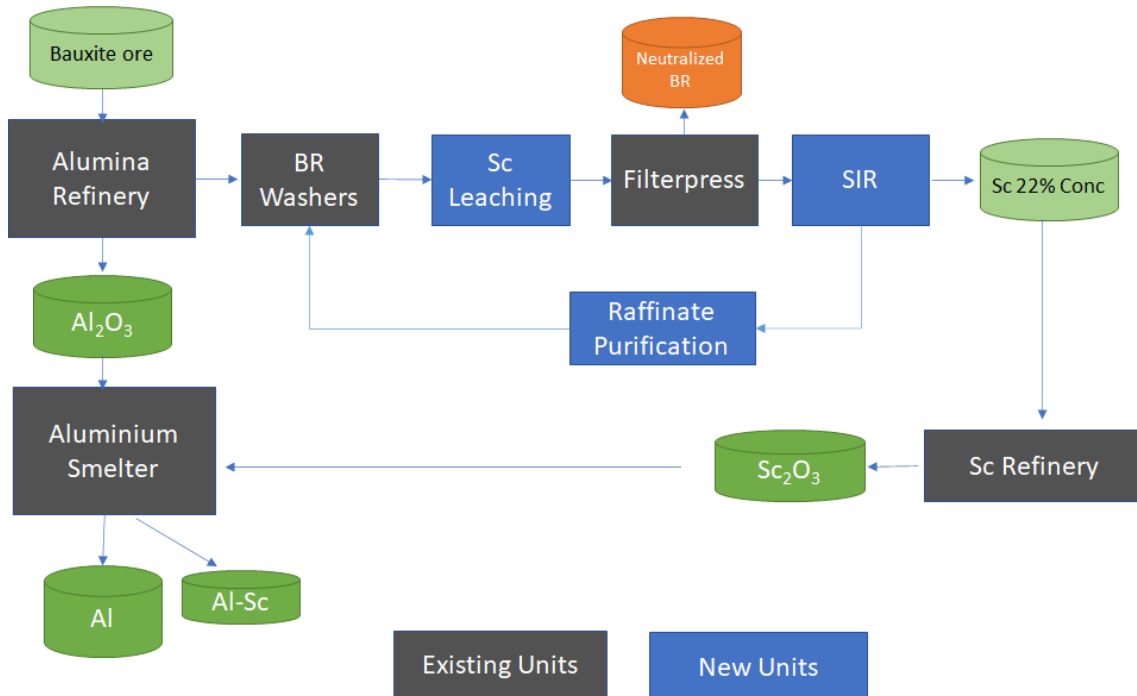


Figure 8. A novel flowsheet for Al-Sc production from bauxite ore.

The synthesis of the above-described results can lead to a novel flowsheet, where Sc extraction is integrated with alumina production and refined Sc₂O₃ is used to produce Al-Sc master alloys in

aluminum smelters (Figure 8). The BR leaching process can be integrated in the alumina refinery at the final stage of BR washing, just before filter pressing (or pulp disposal). Based on the achieved results, in the case of MYTILINEOS SA, which consumes annually 1.8 million tons of bauxite ore to produce 835,000 t of alumina and 850,000 t of BR, a total of 62 t of 22 % Sc concentrate can be produced, which in turn are refined to 22 t of Sc₂O₃. The latter can be returned to the aluminium smelter to produce the 733 t of Al-Sc 2% master alloy.

The new flowsheet can achieve sustainable co-extraction of Sc from bauxite ore, adding value to the final product of the aluminium smelter. Furthermore, the neutralized BR cake, produced in the process, is depleted of alkalinity, and has an increased amount of calcium sulfates, which makes it more attractive for reuse in Ordinary Portland Cement (OPC) production.

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