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Electrolytic iron production from alkaline bauxite residue slurries at low temperatures

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Abstract

The primary iron metallurgy is characterized by significant direct CO₂ emissions, due to the carbothermic reduction of the iron ore. This paper deals with the electrification of primary iron production by developing a new and innovative process for the carbon-free production of metallic iron from Bauxite Residue (BR) which is a by-product of the alumina industry. It is based on the electroreduction of iron oxides from BR suspensions in concentrated sodium hydroxide solutions, at low temperature and normal pressure. The iron oxide source, used in the present study is bauxite residue provided by Mytilineos S.A., Metallurgy Business Unit-Aluminium of Greece. The research study is a preliminary screening of BR as a potential raw material for iron production by performing experiments in a small-scale electrolysis cell. The first results presented here, show that iron can be produced by the reduction of iron oxides in the bauxite residue with high Faradaic efficiency (>70%). Although significant optimization is needed, the novel process shows great promise.

Introduction

At present, primary iron metal is commonly produced through the CO₂ intensive carbothermic reduction of iron oxides in blast furnaces at a temperature of around 1600°C. Since carbon is used as both reducing agent and fuel for the process, blast furnace pig-iron cannot eliminate its CO₂ emissions. Therefore, in recent years, carbon-free electrochemical processes have been widely investigated as potential green alternative routes for the production of iron and iron-base alloys (1) (2) (3) (4) (assuming RES powered electricity).

A large project that has been focused on alternative ways of producing iron was ULCOS (Ultra Low CO₂ in Steelmaking) in which beyond new smelting reduction concepts that were studied, the ULCOWIN electrolytic production of iron from suspensions of iron oxide particles in a highly concentrated NaOH solution at 110°C was demonstrated in Lab Scale. It has

been shown that the iron particles are reduced at solid state, which differs from the conventional electrowinning processes where the metal is deposited through the reduction of dissolved metal cations. Previous works with this process achieved high Faradaic yield (80-95%) (1) (2) (3) (4).

Based on the above-mentioned studies, the technology for alkaline pulp iron electrowinning is being studied for first time to a secondary mineral source, namely the Bauxite Residue of the alumina refining industry. This technique is referred to the SIDERWIN project (5), which aims among the others to produce iron from alternative low-grade iron sources, currently incompatible with the conventional steel making processes.

Bauxite ore is treated within the Bayer process to produce metallurgical grade alumina which is the raw material for aluminum production. Bauxite ore depending on its origin contains 40–60% alumina and the rest being a mixture of iron (20-30%), silicon and titanium oxides. 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 (BR), often termed as “red mud” due to its color. Depending on the grade of the bauxite ore used, BR, on a dry basis, is produced from 0.9 to 1.5 mass ratio to the alumina product (6). The high volume and alkalinity of this byproduct make its valorization a major challenge worldwide (7).

BR is an untapped secondary raw material source considering the presence of valuable substances such as iron (30-45% wt.), aluminum (15-25% wt.), silicon, calcium, titanium and sodium oxides as well as smaller concentrations of critical and/or industrially important elements such as rare earth elements (REEs) (mainly Ce, La, Sc, Y, Nd), V, Cr, and others (6). The recovery of the major metals from bauxite residue has never yet implemented while a lot of processes have been proposed. Despite the lab-scale success of much of the work so far, currently the industrial utilization of BR is estimated at just 2-4 million tones, accounting for less than 3% of the annual BR production (8).

The main constituent of bauxite residue is iron oxides and it can make up to 45% of the mass of the bauxite residue. In fact, the red color of bauxite residue is caused by iron (III) oxides (mostly hematite, Fe_2O_3) (9). In general, due to its high alkaline ($\text{Na}_2\text{O} \approx 10\%$) and its titanium ($\text{TiO}_2 \approx 4-15\%$) content, BR is not suitable for use as iron ore substitute in blast furnaces.

BR reductive smelting processes can be applied by several technologies (Corex, Finex, Hismelt, Romelt, AusIron and EAF) for the production of pig iron (10) (11). So far, two methods have been applied on pilot scale for the BR reductive smelting: the Romelt method

(12) and the electric arc furnace (EAF) (13) (14) (15) (16). The MISA Institute of Steel and Alloy Iron (MISA), with NALCO and RSIL (India), studied the pyro metallurgical process of BRs using the Romelt method (12). The advantage of this method is that approves materials with moisture levels of up to 10% wt. The main disadvantage is the high energy consumption and the poor quality of pig iron with a high concentration of S, P (Panov et al., 2012). In the EAF reductive smelting, mixture of BR, carbon and fluxes, put at 1500-1700°C to form pig iron with higher than 95% iron recovery (13) (6) (17). Recovery of residual iron can be further improved by later magnetic separation in slag dust (18). Post-melting slag can be used to produce rockwool or building materials (6) (19) and for the recovery of non-ferrous metals and REE (9) (18) (20) (21). However, such methods have not been industrialized as they are not competitive to established iron and steel making process.

The present paper describes a highly promising electrochemical method for sustainably extracting the iron from BR, in an alkaline environment, which is in-tune with the alkaline environment of the Bayer process.

Materials and Methods

The process is conducted in an electrolysis cell consisted of a borosilicate glass beaker (250 ml) closed with a specially configured cylindrical silicon bung (45 cm diameter). The experimental apparatus is shown in Figure 1.

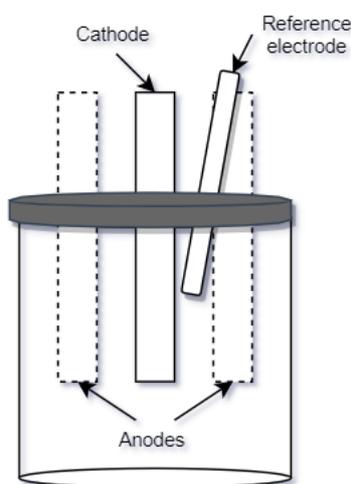


Figure 1. Electrolysis experimental apparatus

A three-electrode configuration was used; the cathode was a rectangular shaped stainless steel V2A plate (110 mm height, 10 mm length, 1 mm width), the anodes were two rectangular shaped nickel plates (100 mm height, 10 mm length, 2 mm width). All electrodes were centered according to the silicon bung's cylindrical axis in specially configured holes so that the electrodes were in certain positions and at fixed distance to each other. The working electrode's surface area that was immersed in the solution was defined to be 8 cm². The reference electrode that was used was a commercial Hg|HgO|NaOH (1M) electrode (RE-61AP, ALS) which was immersed in a distinct glass.

Bauxite residue was supplied by Mytilineos-Aluminium of Greece (AoG). Samples were solubilized via fusion method according to which a quantity of bauxite residue remained at 1000°C for 1 hour with a mixture of Li₂B₄O₇/KNO₃ followed by direct dissolution in 6.5 % HNO₃ solution. Chemical analysis was performed by Atomic Absorption Spectroscopy (AAS) with the use of Perkin Elmer 2100 Atomic Absorption Spectrophotometer. The chemical analysis of the samples used in this study is shown in Table 1. The chemical analysis showed that Fe₂O₃ is the main content of Bauxite Residue.

Mineralogical analysis of bauxite residue (Figure 2) showed that the main iron oxide phase is Hematite while a small amount of Goethite also exists. The Hematite to Goethite mass ratio in Bauxite Residue is 4,2 (22).

Prior to each experiment, the stainless-steel cathode and the nickel anodes were polished with sandpaper and were rinsed with demineralized water. Furthermore, the cathode was weighted. The electrolyte was a 50% wt. NaOH aqueous solution corresponding to molarity of 25 mol/kg, in which it was added an amount of 10% wt. bauxite residue solid particles. Typically, a mixture of 152,4 g solid NaOH with purity higher than 99% (CHEM-LAB NV) and 152,4 g demineralized water were mixed under stirring for about 30 min. After the electrolyte's homogenization, 33,9 g of bauxite residue were slowly added to the solution within 5 min.

	Fe₂O₃	Al₂O₃	SiO₂	TiO₂	CaO	Na₂O	LOI
Wt.%	44.77	18.75	6.69	6.65	9.77	2.93	9.17

Table 1. Bauxite Residue chemical analysis

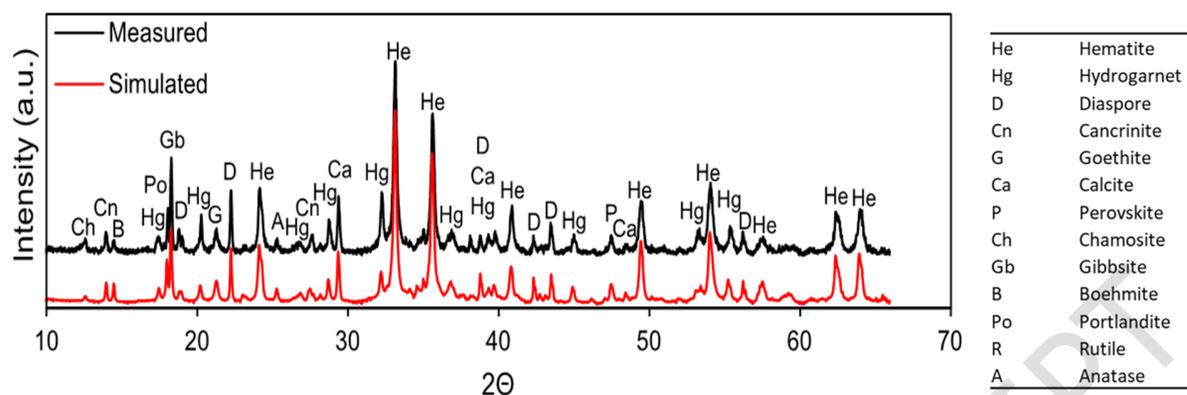


Figure 2. Bauxite residue mineralogical analysis

The slurry temperature was measured via a probe that was wrapped in a PTFE shrink tubing to avoid current leakage. The slurry was stirred using a 1 cm ringed cylindrical magnetic bar and magnetic hot plate (IKA-Werke RCT Basic) at a rotational speed of 500 rpm to keep bauxite residue particles suspended. The small size of the magnetic bar was chosen to minimize the attraction of magnetite particles to the stirrer. The cathode, the anodes and the reference electrode were connected via their respective terminal to a potentiostat (2461 Series, Keithley).

Cyclic voltammetry and electrolysis tests, under chronopotentiometry mode, were performed in a three electrodes cell connected a potentiostat (2461 Series, Keithley) and the obtained experimental data were analyzed. Regarding cyclic voltammetry, it should be mentioned that both working and counter electrode were platinum wires while the reference electrode that was used was the mentioned above Hg/HgO commercial electrode.

Electrolysis tests were performed under galvanostatic mode. The duration of its experiment was 2 h. After the end of each experiment, the cathode was thoroughly rinsed with distilled water in order to remove the remaining electrolyte, and was dried at 100°C for 24 h, before weighed. The difference between the mass of the cathode prior to and after the end of electrolysis was considered as the mass of the deposit. This value was used to deduce the current efficiency according to faradaic law.

Determination of metallic iron

According to previous studies, hematite reduction mechanism to form metallic iron on the cathode has magnetite as an intermediate product (2). For that reason, a quantitative determination of the deposit took place to identify the percentages of both metallic iron and magnetite phases.

The principle of the method is based on the selective dissolution of metallic iron from a 2% bromine solution in ethanol when it is in mixtures with its oxides. According to the procedure of the method 100 ml of bromine (ACROS ORGANICS) solution 2% in ethanol (EMSURE) was prepared and 0,2 g of sample powder was added in a 250 ml conical flask. The solution was let to stir at ambient temperature for 90 minutes and then filtered using a 47 mm diameter glass fiber filter (WHATMAN) (23). The resulting solution was titrated into a 500 ml flask with deionized water. Finally, the solution is diluted and measured in atomic absorption spectrophotometer (Perkin Elmer 2100).

Results

Cyclic Voltammetry

A voltammogram of bauxite residue (10%w/w) with 50% w/w NaOH solution in 110°C and Scan Rate 100 mV/sec is shown in Figure 3. Iron oxide from bauxite residue (mainly hematite) is reduced to metallic iron in the region of cathodic potentials from -1,2 V to -1,4 V with a peak at -1,36 V. Hydrogen evolves at more negative potentials lower than -1,4 V. The plateau observed at cathodic potentials in-between -1,0 V and -1,2 V is attributed to reduction of hematite to magnetite which is always taking place in the system under study as is seen in Raman spectra of a typical deposit in Figure 4. The peak observed at anodic scanning at about -0,7 V is attributed to the reversible oxidation of iron.

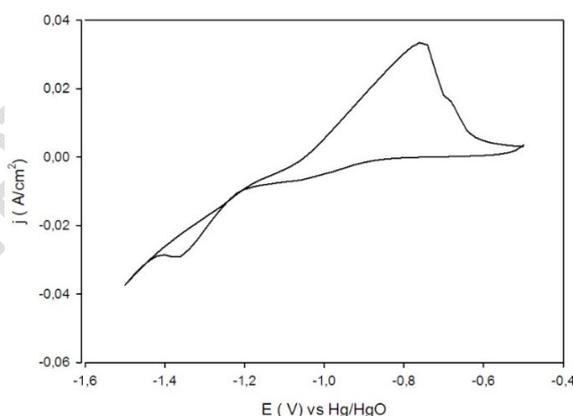


Figure 3. Cyclic Voltammetry in pulps of Bauxite residue (10% w/w) in 50% w/w NaOH solution at 110°C and 100 mV/sec

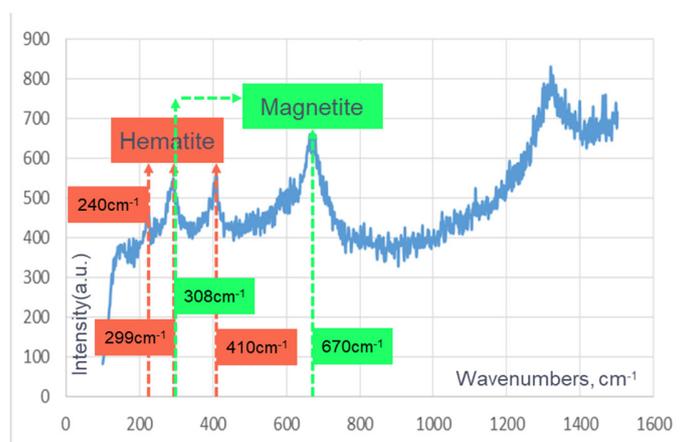


Figure 4. Raman Spectra of cathodic deposit

The electrochemical reactivity of the iron oxides of bauxite residue was evaluated by galvanostatic electrolytic experiments. The parameters that were tested were current density and slurry temperature.

Effect of Current density

Galvanostatic experiments were performed in different applied currents (62.5, 156.3, 312.5, 625, 937.5, 1250 A/m²) while all other factors remained constant (50% wt. NaOH, 10% wt. Bauxite Residue, Temperature 110°C, and stirring rate 500RPM). The open circuit potential between the working electrode and the reference electrode is -0,968V.

The cathodic potential vs Hg/HgO is shown in Figure 5. As it was expected, the increase of the applied current results in more negative values of cathodic potential indicating more intense reductive conditions at the cathode. The applied currents in-between 62.5 A/m² and 312.5 A/m² gave constant cathodic potential values for the whole duration of electrolysis tests in the range of -1,2V to -1,4V which coincide fully with the region of hematite reduction to metallic iron as is shown in the voltammogram of Figure 3. Higher than 312.5 A/m² applied current densities push the cathodic potential towards the hydrogen evolution region (<1,4 V) as is seen in Figure 3. In all experiments the cathodic potential was lower than the open circuit potential and therefore the cathodic material was stable and not corroded.

The calculated faradaic efficiency for each experiment, taking into account the purity in metallic iron of the deposition that was determined to be 89-91% in all experiments, is shown in Table 2. As impurities on the cathode have referred the particles of magnetite which are formed by the reduction of hematite on the cathode surface. In conclusion, the metallic iron produced is extremely pure and the only processing needed is a melting process to produce pure Fe ingots.

As is seen the lower the applied current the higher the faradaic efficiency is. The lowest applied currents in the region of 62.5 A/m^2 to 312.5 A/m^2 gave the highest current efficiencies which were very close to 50%. Even this current efficiency is very low indicating the strong presence of parallel unwanted cathodic reactions such as the hydrogen evolution as well as unavoidable cathodic reactions such as the reduction of hematite to magnetite that always is taking place in this system. The intense hydrogen evolution even in the cases of the lowest applied currents where the measured cathodic potentials was higher than the one of hydrogen reduction indicates a cathode with non-uniform potential. This is reasonable because the cathode is covered by electroactive hematite particles as well as non-electroactive ones coming from the Bauxite Residue. The electroactive species are the iron oxides as hematite and goethite and the non-electroactive particles are all the rest phases in bauxite residue as diaspore, cancrinite, calcite, hydrogarnet, perovskite, gibbsite, boehmite, anatase. Therefore, the current distribution on cathode is non-uniform giving rise to areas with charge accumulation (located where the non-electroactive species are concentrated) and thus more negative potential in relation to the other areas where the electroactive hematite particles are located.

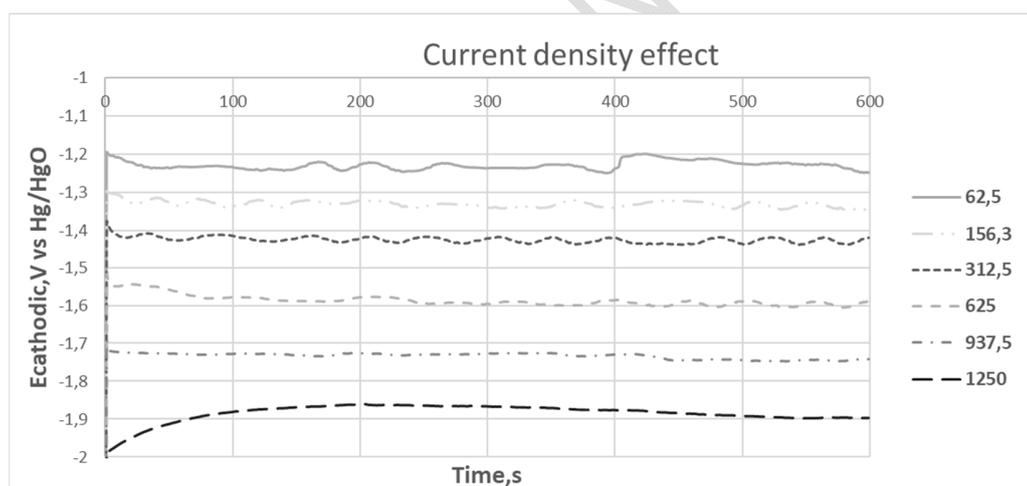


Figure 5. Cathodic potentials during galvanostatic experiment at different current densities (A/m^2)

Current density (A/m^2)	Current Efficiency (%)
62,5	48,48
156,25	48,84
312,5	41,19
625	25,28
937,5	35,14
1250	33,74

Table 2. Current efficiency of galvanostatic experiments for the investigation of the applied current effect

Effect of temperature

The temperature effect was studied in the range 70° – 135°C while the other electrolysis parameters were kept constant (50 w/w % NaOH, 10 w/w % Bauxite Residue, and stirring rate 500RPM). The applied current density was selected to be 156.3 A/m² as this value resulted in the highest faradaic yield (48,84%) in the previous experimental series. The cathodic potentials vs Hg/HgO are shown in Figure 6. As is seen the cathodic potentials within the whole duration of all electrolysis experiments were remained in the region from -1,2 V to -1,4 V which coincides with the region where the hematite is reduced to metallic iron (Figure 3). In addition, there is a tendency for less negative cathodic potentials (milder reductive conditions) as the process temperature increases. The calculated faradaic efficiencies are shown in

T(°C)	Current Efficiency (%)
70	11,23
90	24,48
110	48,84
120	54,94
130	71,58
135	59,76

. The increase of temperature from 70° to 130°C resulted a steady almost linear increase of faradaic efficiency from 11,23% to 71,58%. Step up temperature to 135°C caused a slight decrease in current efficiency to 59,76% which is still higher than the one at 120°C.

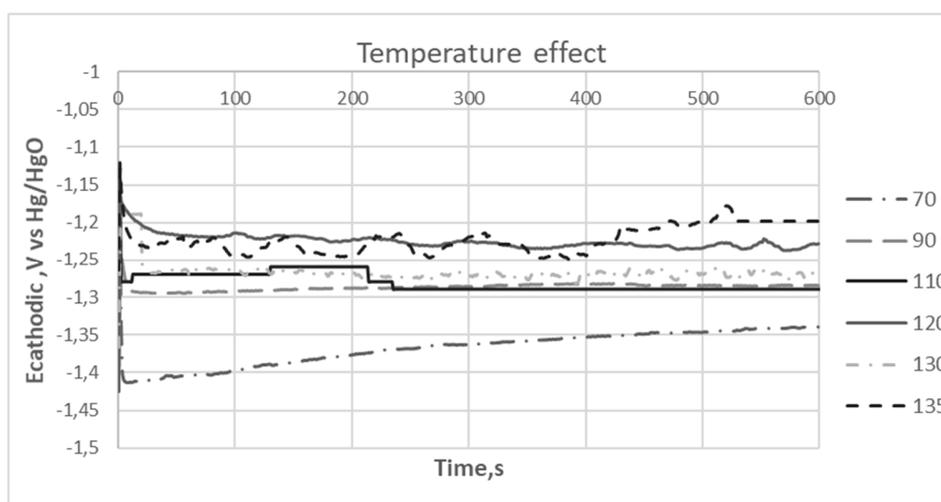


Figure 6. Cathodic potentials during galvanostatic experiment at different pulp's temperature ($^{\circ}\text{C}$)

T($^{\circ}\text{C}$)	Current Efficiency (%)
70	11,23
90	24,48
110	48,84
120	54,94
130	71,58
135	59,76

Table 3. Current efficiency of galvanostatic experiments for the investigation of pulp's temperature effect

As it is mentioned above the cathode surface is covered with electroactive a non-electroactive particle coming from Bauxite Residue. The electroactive ones are strongly attached on cathode surface due to their partial reduction to metallic iron while the non-electroactive are loosely attached on it. The temperature increase causes an increase in the rate of heterogeneous nucleation of water bubbles on particles' surface due to vapor pressure increase. Therefore, the probability of removing the loosely attached non-electroactive particles from the cathode surface increases and thus the probability of replacing the non-electroactive with electroactive ones increases. Under higher temperatures the % coverage of cathode with electroactive species increases and thus the faradaic efficiency increases. At temperatures close to the boiling point of 50 w/w % NaOH solution that is 143°C , the

water bubbling is so intense that starts to detached also the electroactive particles from the cathode surface and therefore decrease the faradaic efficiency. A compromise is achieved at an intermediate temperature which in this case is 130°C.

In any case, the faradaic efficiencies achieved during the BR pulps electrolysis are substantially lower than the ones achieved in iron ore pulps electrolysis (3) where the cathode is uniformly covered by only electroactive particles.

Conclusions

The present work has demonstrated the possibility to electrochemically reduce iron from bauxite residue in alkaline pulps. The process temperature proved to be the most crucial parameter that affects substantially the faradaic process efficiency. At the current level of process development, a faradaic efficiency of 71,58% was achieved at pulp density of 10% wt. BR in a 50% NaOH solution at 130°C. The applied current has to create a cathodic potential higher than -1,4 V vs Hg/HgO (in 1M NaOH) in order to avoid the hydrogen evolution which takes place at cathodic potentials lower than -1,4V.

The cathode in the case of Bauxite Residue pulps electrolysis is not uniformly covered by electroactive iron oxide particles (mainly hematite) and therefore there is not a uniform cathodic potential on the whole cathode surface. This affects substantially the current efficiency of the process which is always substantially lower than the one observed in pure hematite ore electrolysis.

Bauxite residue is produced as a by-product of the Bayer process, an alkaline leaching process taking place at temperatures 120-250°C. Therefore, the present process has great potential for integration in the established Bayer process as a symbiotic step to valorize the -currently wasted- iron portion of the bauxite ore.

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