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THE IMPORTANCE OF USING HYDROMETALLURGICAL TREATMENT OF DIFFERENT RAW MATERIALS AT HIGH PRESSURES FOR ENVIRONMENTAL PROTECTION AND SUSTAINABLE DEVELOPMENT

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Abstract: *High pressure hydrometallurgy offers many promising approaches for industrial application in order to improve the environmental impact of conventional metals productions replacing pyrometallurgical processes whose gas emissions and a high content of formed metals (As, Cr, Pb) are becoming increasingly unacceptable. The main advantages of pressure hydrometallurgy are fast kinetics, enhanced selectivity over iron and other dissolved species. Gradually, industrial application took place firstly in aluminum and later in nickel production. Today, in addition to nickel and aluminum, the pressure hydrometallurgy is well established in a wide spectrum of industrial applications for production of different metals (gold, zinc, molybdenum, titanium, germanium) from ore deposits and secondary materials. It operates in a sealed reactor (autoclave) at high temperatures and pressures, speeding up the dissolution kinetics. It is particularly helpful for processing refractory ores that need more aggressive conditions to extract metal effectively. These diverse leaching methods offer versatility in handling various ore kinds and maximize metal recovery while taking environmental and financial factors into account. The chosen high pressure leaching process will be explained for aluminum, titanium, and iron from bauxite residues and lead concentrate.*
Keywords: autoclave, high pressure, lead concentrate, bauxite

1. INTRODUCTION

Dissolution at high pressures in autoclave is one of the unit operations in non-ferrous metallurgy, which represents traditional metallurgy used for the treatment of ores, concentrates, and waste raw materials. There are few methods that have been so enduring a closed device under pressure a maximum temperature of 250-270°C. The first information regarding a reaction under high pressure is reported by Vladimir Nikolayevich Ipatieff (1900). The precipitation processes, however, were performed by Nikolai Beketoff in 1859 and metal dissolution by Karl Bayer in 1892. Although initially autoclaves were used for leaching bauxites for more than 100 years, pressure hydrometallurgy has progressed extensively since the middle of 1980s, when it was applied for zinc sulphide concentrates and refractory sulphide gold bearing ores. The application of pressure and high

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temperature leaching in autoclaves is definitely one way of overcoming the slow kinetics of hydrometallurgical processes. In the early seventies Habashi [2] reported that pressure hydrometallurgy is the best option for better and non-polluting leaching processes. In this paper he explained how pressure hydrometallurgy is successfully applied for the leaching of nickel oxide, sulphide and arsenide, highlighting the importance of the pressure reactors, i.e., autoclaves. Nowadays, laboratory autoclaves for hydrometallurgical investigations are available in different sizes, models and materials.

The maximum pressure and temperature at which the autoclaves can operate depend upon the design of the vessel and the materials used in its construction (rotary autoclave, horizontal and column autoclave system). Titanium is an excellent material for use with oxidizing agents, such as nitric acid, aqua medium and other mixed salts. Prospective users must remember that titanium will burn vigorously in the presence of oxygen at elevated temperatures and pressures. While there have been many successful applications where oxygen and sulphuric acid are handled in titanium equipment, the danger of ignition is always present and must be prevented [3].

The high pressure acid leaching HPAL is the current widely accepted process for greenfields nickel laterite projects. The extension of the process from tropical limonites to Western Australian laterites is a new approach, and as such the behaviour of the different minerals in tropical and arid laterites during leaching is widely examined. Tindall and Muir [4] conducted fundamental investigation of the HPAL technique using synthetic goethite as a model ore. It was found that goethite transforms to hematite by dissolution and the re-precipitation mechanism. The leaching rate depended on the acid concentration, solid liquid ratio and mixing rate [5].

Aluminium oxide production is performed using the Bayer process for dissolution of bauxite ores with sodium hydroxide under high pressure in an autoclave, as one most important operation. Due to impurities in the bauxite, the ore needs to be treated in order to produce pure alumina (Al_2O_3). That is achieved by blending and grounding it to ensure consistent infeed, then placing it in a autoclave at 150°C along with caustic soda (NaOH). During this operation bauxite residues (red mud) as a by-product is formed as new source for the recovery of critical metals.

In this work we will explain the high pressure leaching of bauxite residues from ALUMINA, Zvornik, Bosnia and Herzegovina and lead concentrate from RUDNIK mine, Gornji Milanovac, Serbia. The high pressure leaching process is combined with pyrometallurgical treatment in order to remove iron from the initial material. The main aim is to study metal recovery from different materials using sulphuric acid in an autoclave.

2. MATERIAL AND METHOD

2.1. Material

Due to its properties such as high alkalinity, bauxite residue can be used as an input material in various neutralization processes. This table shows that the bauxite residue from Zvornik, Bosnia and Herzegovina contain mostly iron oxide:

Table 1. Chemical composition of BR, Bosnia and Herzegovina
Source: *original author's*

Compounds	Fe_2O_3	Al_2O_3	CaO	SiO_2	TiO_2	Na_2O	Cr_2O_3
Percent (%)	49.3	12.0	8.2	10.5	4.6	2.5	0.13

The factory Alumina has been in continuous production mode since October 6, 1978 and continuously processes bauxite and produces alumina, aluminium hydroxide, zeolites and other related aluminosilicate products. The company Alumina currently has about 1500 employees, which is about

25 % of all employees in Zvornik. Alumina owns a red mud disposal site located about 5 km away from the factory. Transportation of the red mud suspension from the factory to the landfill is carried out by suitable pumps. The area of the red mud landfill is about 1 km^2 with disposed 20 million tons of BR.

The Pb concentrate (PbS) was collected from a polymetallic ore deposit "Rudnik" froth flotation plant (Rudnik, Serbia). Prior to characterization and leaching tests, the concentrate was ground and used in all further experiments.

Table 2. Chemical composition of Pb-concentrate, Rudnik, Serbia
Source: *original author's*

Elements	Pb	Fe	S	Zn	Cu	Ag	Bi	Sn	Ca	Si
Percent (%)	56.49	3.58	9.02	0.76	0.60	0.10	0.36	0.01	0.45	0.98

2.2. Method

High pressure leaching method was used for the treatment of bauxite residues and lead concentrate. A 5M sulfuric acid solution was prepared in a 1-L flask and introduced into a Buchi autoclave (as shown at Figure 1), containing slag derived from the hydrogen plasma reduction process.

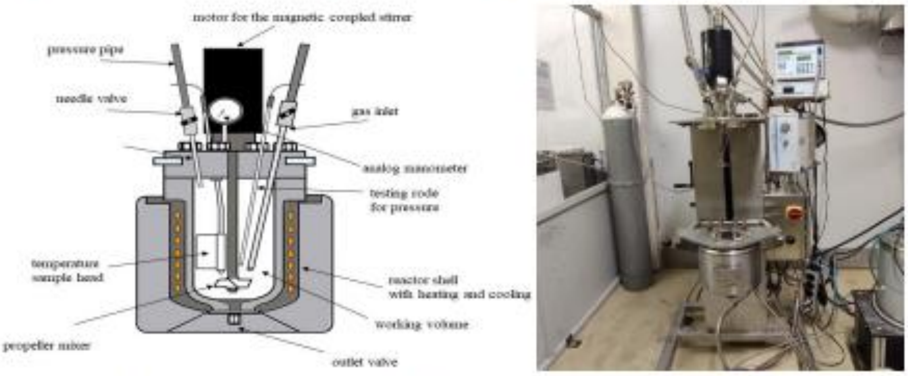

Figure 1. Sketch of one autoclave and experimental setup at the RWTH Aachen University
Source: original author's [3]

The system was pressurized to 9 bars with oxygen, the temperature was set to the desired level, and the magnetic stirrer was operated at a speed of 500 rpm. The leaching process was conducted for up to 120 min. The leaching process was performed in a Buchi autoclave, a specialized system designed for acid leaching with a capacity of 1.53 L, a maximum pressure of 200 bars, and a maximum temperature of 270 °C, and maximum agitation rate of 2000 rpm.

The autoclave is equipped with a heat exchanger controlled by a thermostat, a mixer, pressure adjustment probes, and the capability for high-pressure sample extraction during operation. The system is fully integrated with computer software, enabling precise control and real-time monitoring of operational parameters, with all data recorded for detailed post-experiment analysis.

The pressure within the system was monitored using both a manometer and digital sensors, with the total pressure comprising oxygen (9 bars) and water vapor (12–15 bars). Cooling was achieved using a dedicated cooling system, and the heating rate was controlled at 10 °C/min. Prior to each run, the autoclave was manually sealed with screws and subjected to a pressure integrity test to ensure safe and reliable operation. Due to very small amount of sample only one experiment has been conducted. Parameters were chosen considering experience from previous paper [6]. After the leaching process was completed, the autoclave was cooled to room temperature, and the system pressure was carefully released. The leachate solution was then subjected to filtration and neutralization with distilled water. Filtration was conducted using a vacuum-assisted filtration system integrated into the setup, ensuring effective separation of solid and liquid phases.

3. RESULTS AND DISCUSSION

3.1. High pressure Leaching of bauxite residues after pyrometallurgical treatment

After hydrogen reduction of bauxite residues in rotary kiln at 920°C and in electric arc furnace with plasma hydrogen, magnetic separation was used for separation of iron. The obtained solid residue was leached with 5M sulphuric acid at 150°C in oxygen atmosphere. The leaching efficiency of titanium, aluminium and iron are shown at Fig. 2.

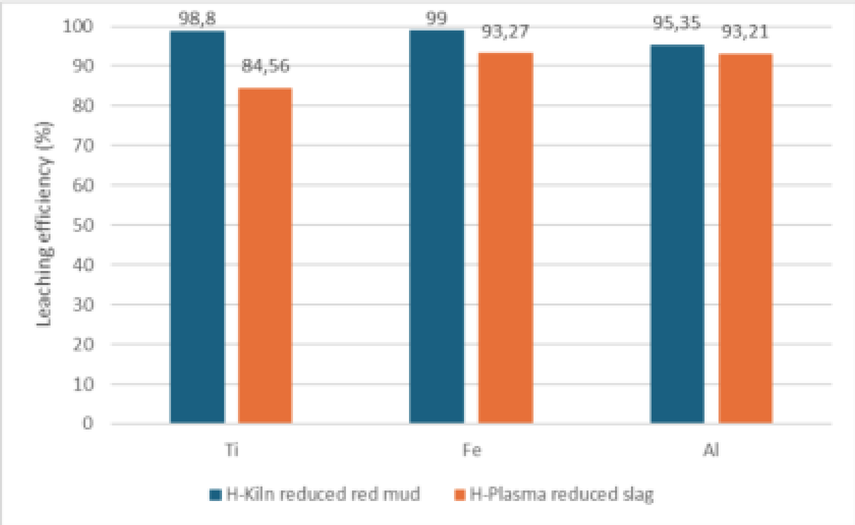

Figure 2. Leaching efficiency of Fe, Al and Ti after treatment of solid residue in an autoclave
Source: original author's

The obtained results have confirmed that reduction in rotary kiln eneables sufficient leaching efficiency from solid residues. The maximal leaching efficiency after reduction was 99 % for Ti and Fe. The leaching efficiency of Ti from slag obtained from hydrogen plasma reduction amounted 86 %. The reason of decreased leaching efficiency of titanium is in a mineralogical structure of slag in comparison to solid residue obtained from rotary kiln. The mechanism of dissolution of Fe, Al and Ti is shown with Equations (1)-(4).

$$Fe_2O_{3(s)}+3\ H_2SO_{4(aq)}\rightarrow Fe_2(SO_4)_{3(aq)}+3\ H_2O \quad (1)$$

$$TiO_2+H_2SO_4\rightarrow[TiO]SO_4+H_2O \quad (2)$$

$$TiO[SO_4]+\frac{1}{2}\ O_2\rightarrow[TiO\text{-}O]SO_4 \quad (3)$$

$$Al_2O_3+3\ H_2SO_4\rightarrow Al_2(SO_4)_3+H_2O \quad (4)$$

3.2. High pressure leaching of lead concentrate

The lead concentrate was leached using different concentration of sulphuric acid in autoclave between 120°C and 180°C, as shown in Table 3 and Figure 3.

Table 3. Parameters for dissolution of lead concentrate (S/L: 70g/700 ml; stirring speed: 600 rpm)
Source: *original author's*

Experiment	Acid Molarity (M)	Temp (°C)	O_2 Pressure (bar)
1	1.0	120	6
2	1.0	150	10
3	1.0	180	10
4	1.0	150	12
5	2.5	150	6
6	1.0	150	8
7	2.5	150	8
8	2.5	150	10
9	2.5	150	12
10	5.0	150	6

This diagram shows that time and reaction temperature positively influence iron dissolution. Additionally, an increase of partial pressure of oxygen from 8 bar (Exp. 6) to 12 bar (Exp.4) leads to an increased leaching efficiency of iron using 1 mol/L sulphuric acid at 150 °C. An increase of concentration of sulphuric acid from 1 mol/L (Exp. 6) to 2.5 mol/l (Exp. 7) leads to an increase of leaching efficiency of iron at 150°C. The maximum leaching efficiency of iron reaches 85% after 4 hours between 120°C and 180°C.

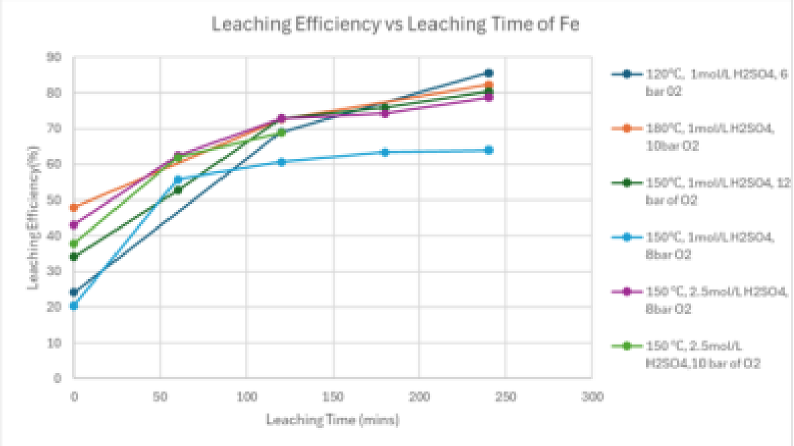

Figure 3. Leaching efficiency of iron after leaching of lead concentrate in an autoclave
Source: original author's

The leaching of iron and lead from lead sulfide concentrate with sulphuric acide in oxygen atmosphere is shown using Eq. (5) and Eq (6).

$$2\,FeS + H_2SO_4 + 4.5\,O_2 = Fe_2(SO_4)_3 + H_2O \qquad (5)$$

$$PbS + H_2SO_4 + 0.5\,O_2 = PbSO_4 + S + H_2O \qquad (6)$$

The leaching of Pb with sulfuric acid and oxygen produces $PbSO_4$ and elemental sulphur (S^0), which may be chemically resistant to leaching. Insoluble lead compounds may have formed during leaching, which may have suppressed solubilization.

4. CONCLUSION

The obtained results confirm that it is possible to dissolve iron, titanium and aluminum from bauxite residues after pyrometallurgical treatment using sulfuric acid in a closed device at high pressure with the introduction of oxygen at 150°C. The dissolution of iron from lead sulphide concentrates is possible between 120 °C and 180 °C with sulfuric acid with the introduction of oxygen in the autoclave. It is generally confirmed that leaching at high pressures in an autoclave can be used as one of the important operation for obtaining valuable metals from various raw materials.

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