**International Journal of Science and Research Methodology (IJSRM)**
Volume 28, Issue 1, January 2025 **ijsrm.humanjournals.com** ISSN: 2454-2008

# Study on Methods for the Extraction of $Fe_2o_3$ and $Tio_2$ from Bauxite Resists (Red Sludge)

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Received: 2025-1-07 Revised: 2025-1-18 Accepted: 2025-1-25

## ABSTRACT

Bauxite processing waste, more commonly known as red mud, is an industrial residue produced during alumina production. The storage of this waste is considered a real environmental concern because of its caustic nature. Nowadays, most of these applications are found in civil engineering and construction, chemistry, metallurgy (metal recovery), as well as in the environment for the treatment of polluted water and soil. The main aim of this study is to develop a methodology for identifying and understanding the best possible way of recovering these bauxite residues, in order to improve the management of these sludges. The development of this method would be of great importance to alumina companies. The method developed could reduce the problem of sludge at the disposal site. The main objective of this study is to review the literature on methods for extracting iron and titanium from red mud. The approach consisted in highlighting the various tests on red mud in order to arrive at a high-performance and more ecological technique.

**Keywords**: Method, Extraction, Red mud, Treatment

## 1. INTRODUCTION

Bauxite residues (more commonly known as red mud), due in particular to the use of soda ash and lime during the Bayer process, are an alkaline, salty and sodic material that prevents any form of life from taking root **(Gräfe et al., 2011)**. Red mud is the bauxite processing waste obtained during extraction by the Bayer process after decantation of the suspension containing the sodium aluminate dissolved during etching.

The storage of red mud is a major problem for alumina production plants, as they generate large quantities of residue that must be stored (Power et al., 2011). Another major concern is the presence of essentially toxic trace metals and metalloids such as aluminium, vanadium, chromium and lead. A great deal of research is therefore being carried out to examine not only the environmental and health risks associated with these residues, but also remediation measures to limit their dispersion **(Gräfe et al., 2011), (Klauber et al., 2011).**

However, this process, which is still the most widely used, involves several successive operations, for which a number of improvements and optimisations have since been made. From the raw bauxite received at the plant, alumina powders are produced, which are used among other things to produce aluminium ingots that are sold to industries that transform them into parts of all kinds, mainly for transport and construction, but also for containers, electrical components, machinery and equipment. Today, the volume of aluminium used exceeds that of all other non-ferrous metals **(Verreault, 2009).**

The problem lies in the extraction process, which generates a fair amount of red mud, which is a real problem for our ecosystem. The composition of red mud depends on the nature of the bauxite, which depends on its deposit. The Bayer process is universal, but it is less environmentally friendly because the quantity of red mud produced is enormous.

In recent years, the industrial structures needed to manufacture alumina have had to be adapted to meet growing market demand. In addition, as the quality of available bauxite deposits has declined, larger quantities have to be processed using optimal methods to meet demand **(Verreault, 2009).**

The aim of this work is to contribute to the recovery of red mud with a view to extracting certain metals, using a rational and competitive technique. At present, several approaches to recovery are being made using a number of different methods, spread out over time and space.

Bauxite processing waste, more commonly known as red mud, is an industrial residue produced during the manufacture of alumina. The storage of this waste is considered to be a real environmental concern, given the caustic nature of the material (PH >10) and the quantities produced (around 1.5 per tonne of alumina produced) **(Krishna et al., 2005).**

Bauxite waste, known as red mud, is a by-product of the manufacture of alumina using the Bayer cycle. Today, the global quantity of red mud produced amounts to more than 4.6 billion tones, almost 600 million tones in Russia **(Zinoveev et al., 2021).** These bauxite residues occupy vast areas, causing environmental damage and risks. In addition, they contain a sufficient quantity of sodium, which is easily solubilised in subsoil water, so a sustainable technique for recycling the tailings is needed.

The goal in the alumina extraction industry should be zero waste, and to achieve this goal, techniques will need to be popularised that allow this red mud to be used as a raw material for several purposes **(Garg et al., 2015).**

Red mud can be considered a polymetallic material, containing a large quantity of precious metals. It consists of iron, titanium, aluminium and a few minor elements or traces of rare earth elements **(Gräfe et al., 2011).**

## 2. METHODS FOR EXTRACTING Fe2O3

Several techniques have been reported in the literature for using bauxite residue for environmentally-friendly practices. This began as early as the 1940s, with most of them claiming that the separation of iron oxides is the flagship technology.

As iron is one of the most important components in red mud, its extraction has received particular attention **(Lyu et al., 2021).** In red mud, iron is generally found in the form of hematite (Fe2O3), and a certain quantity is present in the form of goethite **(Klauber et al., 2011).**

According to **(X. Li et al., 2009),** the huge quantity of residues generated by the manufacture of alumina by the Bayer cycle not only degrades the environment but also results in a loss of secondary resources. Bauxite residues with a high iron content from the Bayer process have been used to extract alumina and ferric oxide using reduction-sintering, leaching and magnetic separation techniques. Haematite and goethite in bauxite residues with a high iron content can be reduced to magnetite by roasting, magnetic separation in suspension with CO (**J. Yu et al., 2022). Z. Yu et al. (2012),** extracted iron by 1 mol/L oxalic acid liquor at 75 °C for 2 h, followed by UV light.

### 2.1. HYDROMETALLURGY METHOD

This method allows Fe2O3 to be extracted through certain dissolution reagents, mainly acids. As a result, some have reported it in their literature;

**Z. Yu et al (2012),** extracted iron using 1 mol/L oxalic acid liquor at 75°C for 2 h, followed by UV light.

Leaching with hydrochloric acid to separate iron and alumina, followed by digestion with sulphuric acid **(Rai et al., 2019)**.

### 2.2. PYROMETALLURGICAL METHOD

It involves developing a technique for extracting iron oxides in magnetic form from bauxite residue by reduction and certain oxides if possible. The development of this extraction method involves mastering the factors controlling the magnetic separation process applied to calcined bauxite residue.

This will be achieved by:

- Characterising the material by observing its physical structure and behavior in solution;

- Determining, understanding and, if possible, quantifying the parameters that can affect magnetic separation;

- Carry out a parametric study using one type of separator;

- Numerically test the separation process;

- Adopt a model by means of experimental tests

Some publications mention magnetic separation used directly on haematite. In 1981, using a high-gradient magnetic separator producing a high magnetic field intensity of up to 22,000 gauss on ferruginous bauxite from Venezuela, the extracted bauxite residue contained 39% $Fe_2O_3$, with an enrichment rate of 15% obtained in the magnetic part and 60% of the extraction rate approximately **Bolsaitis et al (1981).** The literature reports the use of high-gradient superconducting magnetic separator systems to extract haematite particles **(Y. Li et al., 2011)**. Due to the low magnetic susceptibility of haematite, magnetic separation was achieved with a high magnetic intensity of over 40,000 gauss.

Using two high gradient wet magnetic extractors in series providing a high magnetic intensity of 8,000 gauss each, will increase the $Fe_2O3$ content from an original value of 51% up to 60% in the magnetic part, with a recovery of 40% **(Rai et al., 2019).** By raising the magnetic intensity to 15,000 to 18,000 gauss, the recovery rate will be able to reach 60% without considerable effects in the $Fe_2O_3$ content in the magnetic fraction. By combining a granulometric separation operation with a hydro-cyclone before the serial magnetic separation sequence, the $Fe_2O_3$ content will reach 70% with an extraction rate of 75%.

Another very successful iron recovery technique is direct reduction of iron by elimination using flotation **(López et al., 1998).**

In the fusion reduction technique, bauxite residues with a high iron content, the reductant and additives are mixed and fused at a high temperature. The high reduction temperature produces molten iron containing carbon and molten slag Molten iron containing carbon can be used to make steel, alumina and rare earths can be extracted from the slag, and the slag can also be used to make building materials. In this experiment, a coal-based reductant was used to reduce the haematite in the bauxite residue, and CO combustion provided the necessary heat **(K. Wang et al., 2022**). The recovery yield is mainly determined by the mineralogical composition of the iron in the bauxite; coarse iron particles are easy to recover, while fine iron particles are difficult to recover **(Zhou et al., 2020).**

**X. Li et al (2009)** recovered iron and aluminium from bauxite residues. They did this by reducing the haematite with carbon powder, then basic dissolution (NaOH) of the material to dissolve the aluminium and magnetic enrichment of the dissolution slurry to provide a magnetite-rich product. Using this technique, they were able to extract 90% of the aluminium and 62% of the iron from bauxite residues. In the work by **(W. Liu et al., 2009),** they recovered 81% of the metallic iron using the magnetic method. The protocols were as follows: 18 g of carbon powder with 6 g of additions (3 g of $CaCO3$ + 3 g of $MgCO3$) and 100 g of bauxite residue at a temperature of 1300°C for 110 minutes**.**

### 2.2.1. RÉDUCTION PAR GRILLAGE

Among these methods, the conversion of hematite to magnetic element can be done by a roasting technique, which can happen by roasting pyrite ($FeS_2$) followed by magnetic separation **(Y. Liu et al., 2014)**. From the primary product containing an Fe content of 9%, the magnetic element was enriched to 37% with an extraction rate of 60%. They also observed agglomeration phenomena between magnetic and non-magnetic substances; these accumulations have an influence on the separation results. In addition, given the good conversion of iron oxides, this method gives off sulphur dioxide, which needs to be treated by another process. Roasting techniques include carbon (coal) roasting, which is carried out at a temperature of 1050°C using lignite, a sedimentary rock that essentially contains 60-70% carbon (Rao et al., 2016). They also use sulphite and sodium sulphate as additives to improve the degree of metallisation of the iron. After a separation performed on a Davis tube and a magnetic power of 1,000 gauss, the magnetic particle marked an iron content of 90% with an extraction rate of 95% when the initial product had an iron content of 48%. The same principle of coal roasting at a temperature of 800°C and low-intensity magnetic extraction (800 gauss) enriched the magnetic substance with 61.3% iron and an extraction rate of 88.2% **(C. Li et al., 2010).**

During heat treatment (reduction roasting) of refractory iron materials, such as nickel laterites **(Samouhos et al., 2013)**, oolitic hematite ores **(G. Li et al., 2012)**, ludwigite ore **(G. Li et al., 2013)** and alumina-rich limonite ore **(G. Li et al., 2014)**, experiments have shown that sodium salts ($Na2SO4$ and $Na2CO3$) positively facilitate iron reduction. Some mixed 50% of the carbon powder, with 4% of the additive elements to the bauxite residues with 19.6% iron, then roasted at 700°c for 20 min. The roasted substances were ground and separated to obtain 60% total iron in the magnetic concentrate, with an iron recovery rate of 91% **(Y. Liu et al., 2016).**

### 2.2.2. REDUCTION IN GASEOUS ATMOSPHERE

In order to reduce haematite to magnetite, studies have used gases as reducing agents. Carbon monoxide (CO) has been used by some as a reducing agent **(X. Li et al., 2009)**. It is reported that the magnetic part contained 89% iron for an extraction rate of 81%.

However, this reduction takes place at a high temperature (1300°C) and involves considerable energy costs. Dihydrogen $H_2$ has been experimented with by others as a reducing element during calcination of the bauxite residue at 800°C **(Sadler & Venkataraman, 1991).** Magnetic separation was carried out on a Davis tube with a magnetic intensity of 4,000 gauss. The magnetic proportion had an iron content of 13% and a recovery rate of 42%.

The use of dihydrogen at a temperature of 480°C, is carried out via wet magnetic separation at an intensity of 1160 gauss (**Samouhos et al., 2017**). The magnetic portion yielded an iron content of 40%. The use of dihydrogen facilitates low-temperature reduction, and limits sintering and particle agglomeration processes in the residue.

### 2.2.3. MICROWAVE REDUCTION

Microwave reduction has motivated some to investigate this process (**Agrawal et al., 2018b**). The red mud is mixed with activated carbon and then the reduction is carried out in a microwave furnace. The reduction takes place at a temperature of 800 to 1000°C (800 W, 2.45 GHz). The reduction is preceded by magnetic separation at low intensity (2000 gauss). With an initial iron content of 27%, the magnetic part is enriched to 48% for an extraction rate of 95%. Comparing microwave reduction with conventional reduction methods, this technique enables faster reduction and is more economical in terms of energy and reducer consumption **(Agrawal et al., 2019).**

## 3. METHOD OF EXTRACTION OF TiO2

Titanium dioxide is used in a wide variety of industries, including paint, paper, leather, textiles and pharmaceuticals. Ilmenite and rutile are the most important mineral sources for the production of titanium dioxide. Because of the scarcity of rutile and the quality of ilmenite, other alternative sources of titanium dioxide must be found. This material could be red mud.

Red mud is obtained during the extraction of alumina from bauxite and is accumulating at a rate of 30 millions tonnes a year worldwide. The production of alumina from bauxite generates a fair amount of red mud. Red mud consists of $Fe_2O_3$, $TiO_2$, $Al_2O_3$, CaO, $SiO_2$ and $Na_2O$.

It would be important to consider a technique to enrich the titanium dioxide content so that it could replace ilmenite. The technique would be more beneficial if the essential constituents were recovered selectively.

Much work is being done on the use of red mud as red oxide paint, soil improver, building material, synthetic fertiliser, paints and pigments, etc., but little is known about the extraction of titanium dioxide from sludge. The information available is limited to proposals for extracting titanium dioxide, hence the motivation for this research to establish a more economical and environmentally friendly technique.

At present we have two basic operations for extracting titanium from bauxite residues: hydrometallurgical and pyrometallurgical methods.

### 3.1. HYDROMETALLURGY METHOD

Using this method, trials often focus on reagents for dissolving TiO2 in red mud.

Some have set up a technique in a pilot plant to extract titanium dioxide from red mud (Bhatnagar et al., 1945). The technique consisted of attacking the red mud with sulphuric acid, followed by hydrolysis and calcination. The extraction rate of titanium dioxide in this process was 70.2%. Others envisaged a process that involved dissolving some of the iron and alumina with hydrochloric acid, followed by attack of the precipitate with sulphuric acid **(Damodaran & Gupta, 1955).** Still looking for a recovery process, the red mud was reduced with coke to extract the iron and the residue was dissolved in a chlorinated solution to obtain enriched titanium oxide **(Miodrage and Bratamira, 1963)**. Selective chlorination of the iron in the red mud was proposed in this way **(Samal, 2021).** The red mud was subjected to heat treatment in a reducing atmosphere at 800-900°C and a chlorine solution at 500°C to obtain alumina chloride.

More often hydrolysis and calcination follow the acid dissolution step for $TiO_2$ extraction in leachate **(Deng et al., 2017).** Therefore, as the extraction of a single metal is not beneficial, efficient utilisation techniques should be popularised, to reduce red mud while obtaining titanium and other valuable elements, thus promoting the broadening of the value chain in terms of the economy **(Gräfe et al., 2011)**.

Some literature envisages that titanium dioxide can be extracted by dissolution with sulphuric acid followed by hydrolysis or a chlorination process, converting it into titanium tetrachloride and then into titanium dioxide of appreciable quality. Alumina is recovered by pressure etching and leaching in the form of sodium aluminate or by making alum by dissolving it in sulphuric acid.

Titanium is of great commercial importance and is used in a number of fields The recent technique for extracting titanium dioxide from red mud has been developed.

**(Maitra et al., 1993).** In this process, the sludge is first rinsed with water and then attacked with dilute hydrochloric acid at 95°C and pH 4. After separation of the liquid phase concentrated in calcium oxide, sodium oxide and a small quantity of aluminium chloride, the solid fraction is leached by concentrated hydrochloric acid at 95°C, which facilitates solubilisation of the iron and the rest of the aluminium. After separation, the solid part is dissolved in concentrated sulphuric acid at a temperature of between 150 and 280°C. The titanium is easily solubilised in the form of sulphate. Sequences of hydrolysis, precipitation and calcination help to obtain titanium dioxide with a purity of at least 97.5%. This process is supposed to be efficient, but it is rather more expensive than extracting titanium dioxide from its ore. According to the author, this difference in financial margin could be made up by considering the cost of destroying the land, the environmental effects of depositing the red mud, the gains from recovering the elements lost in the red mud and the market value of the treatment by-products, namely ferric chloride and aluminium chloride. In the same vein, others have thought of a method for enriching bauxite residue in titanium dioxide and have studied the reaction mechanism **(Kasliwal & Sai, 1999).**

The rapid recovery kinetics of titanium depend on the strong dissolution of concentrated sulphuric acid **(Y. Wang et al., 2019).** In terms of recovery rate, titanium exceeds those of aluminium and iron.

Leaching of Ti, Al, and Iron should follow this logic: reaction time > extraction temperature > stirring speed > S/L (**Agrawal et al., 2018a**). A high temperature is not beneficial for titanium recovery.

Some researchers have considered methods for extracting several metals from bauxite residues, using the process of successive dissolution and precipitation by varying the pH using sulphur dioxide (**Astukawa et al., 1967).** Sodium, iron oxides, titanium, silica and alumina are finally separated from the red mud. Others have also experimented with the technique of selective dissolution and precipitation by adjusting the pH (**Barnet & Mezner, 2001**).

Other researchers have succeeded in extracting iron, aluminium, silicon and titanium from red mud. They have studied the extraction of iron (III), aluminium (III), titanium (IV) and sodium(I) ions by Donnan dialysis using a charged poly-sulphonated membrane **(Çengeloğlu et al., 2001), (Cengeloglu et al., 2003**). According to the literature, this process is faster and consumes less energy than current techniques (chemical precipitation, carbon adsorption, ion exchange, encapsulation/chelation, etc.).

Citric acid has been used by some to extract titanium from red mud **(Zhu et al., 2015)**. Sulphuric acid is the most suitable for wet extraction of titanium dioxide from bauxite residues, because TiO2 can react with sulphuric acid to give soluble TiOSO4 **(Zou et al., 2021).**

**Lei et al (2021)** have developed a technique for extracting and recovering titanium and scandium from bauxite residues, with this process the recovery of scandium and titanium is significant and the loss is minimal. This method was carried out using a neutralisation approach by precipitation-acid leaching, with precipitation performances of 93.74% scandium and 99.47% titanium. **Bonomi et al (2018)** also tested the extraction of scandium and titanium from Bayer red mud by direct dissolution using a Bronsted acidic ionic liquid. This technique recovered 80% of Sc and 90% of Ti, with complete leaching of Fe, Al and Na. Almost all the Ti produced in the mines (around 94%) is used to extract TiO2, the most widely used white dye. The remaining 6% is used to extract metallic Ti **(Malabad, 2022).**

**Y. Ghorbani et al. (2008),** in their literature, discussed the extraction of titanium from red mud by a bioleaching technique, using certain fungi. This technique made it possible to extract titanium in a remarkable way. Piga et al. (1993) dissolved bauxite residues in HCl solution, but titanium dissolves in H2SO4 but not in HCl. This mechanism increased the extraction of TiO2 in the reject from 31% to 58%. The solid fractions were leached with a sulfuric acid solution at 270 °C, followed by hydrolysis and calcination, finally the TiO2 obtained was 96%.

### 3.2. PYROMETALLURGY METHOD

For the pyroxene method, iron, aluminum, silicon and other substances present in the bauxite residue are removed at a high temperature by roasting or reduction by melting, therefore, in the slag the TiO2 is enriched. The slag will finally be leached by sulfuric acid for TiO2. Regarding the wet process, diluted hydrochloric acid is used to leach alumina, ferric oxides and other elements contained in the bauxite residue, thus the leaching sludge is leached by sulfuric acid to extract titanium oxide**.**

After this step a solvent extraction and a reverse extraction are carried out, titanium would subsequently be obtained (**Şayan & Bayramoğlu, 2004**). A two-stage dissolution process was carried out to separate SiO2 and Al2O3 to enrich Sc2O3 and TiO2 from non-magnetic substances from reduction, roasting from mining, enrichment of Sc2O3 and TiO2 from of non-magnetic materials from reduction, calcination and magnetic separation, and the findings below were drawn from the study **(Deng et al., 2017):**

1) Through a process of dissolving phosphoric acid and alkali, a product containing 0.044 Sc2O3 and 25.5% TiO2 with recovery rates greater than 85% was obtained from 'a non-magnetic substance grading 0.044% Sc2O3 and 25.5% TiO2 with recoveries greater than 85%, from a non-magnetic material which contains 0.0134% by weight of Sc2O3 and 7.64% by weight of TiO2 from the reductive roasting and magnetic separation process of bauxite.

2) The Sc2O3 was enriched during the dissolution phase by phosphoric acid depending on the pH of the solution compared to the leaching pH 0.5, the ScO3, and the TiO2 were enriched at the same time because that perovskite (CaTiO3) cannot be dissolved in the acidic solution at ordinary temperature and atmosphere.

According to the literature of **(Agatzini-Leonardou et al., 2008),** that the dissolution process is focused on the recovery of titanium with diluted sulfuric acid from bauxite residues in atmospheric situations and without making any prior treatment. Did a study on red mud, where they heat treated the red mud mixed with dolomite and coke. During the process, the titanium extraction efficiency based on slag weight was 84-7%.

## 4.CONCLUSION

This study is part of a dynamic that makes it possible to valorize red mud, by extracting other metals contained in these residues. The Bayer process emits enough red mud, if a recovery measure is not considered, the storage site is overflowed, there is a very high risk of pollution, through infiltration of alkaline substances into the water tables. In this vein, it would be a question of opting for iron and titanium extraction techniques, if possible other metals which will be linked to our protocol.

Our bauxite-holding countries are concerned about the valorization of bauxite residues, because of the protection of our environment, for this reason it would be imperative to carry out research with a view to finding the best solutions that can answer the questions of management and valorization. of this red mud.

This study allowed us to know the different methods of extracting iron and titanium from red mud and to know other alternatives for valorizing these bauxite residues. It would be important for aluminum smelters to integrate into their alumina manufacturing plans and policies to ensure management systems for these residues emitted by these companies.

## 5.ACKNOWLEDGMENTS

In this work, we thank the CEA-MEM, the Guinean Ministry of Higher Education, Scientific Research and Innovation in general and in particular the General Directorate of the Higher Institute of Mines and Geology of Boké, for their invaluable support in this work, our thanks go to my thesis supervisors and all the staff of the INP-HB for their frank collaboration.

## 6.CONFLICTS OF INTEREST

This article presents no conflict of interest, and the authors favor the publication of their works in this journal.

## 7. AUTHOR CONTRIBUTIONS

Our contributions in this work were focused on literature reviews of the different extraction methods of Fe2O3 and TiO2 in red mud and showed the usefulness of their valorization for a question of circular economy.

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How to cite this article:
Kandas Keita et al. Ijsrm.Human, 2025; Vol. 28 (1): 25-32.

Conflict of Interest Statement: All authors have nothing else to disclose.