



METHODS FOR EXTRACTION OF VALUABLE ELEMENTS (Fe, Al, Na, Ti) FROM RED MUD

N. V. Vasyunina¹, I. V. Dubova², K. E. Druzhinin³, A. V. Alekseev, O. Yu. Shubkina⁴,
 T. R. Gilmanshina⁴ and S. I. Lytkina⁵

¹Department of Metallurgy of Non-Ferrous Metals, Institute of Non-Ferrous Metals and Materials Science, Siberian Federal University, Krasnoyarsk, Russia

²Department of Fundamental Natural Science Education, Institute of Non-Ferrous Metals and Materials Science, Siberian Federal University, Krasnoyarsk, Russia

³Irkutsk National Research Technical University, Irkutsk, Russia

⁴Department of Engineering Baccalaureate CDIO, Institute of Non-Ferrous Metals and Materials Science, Siberian Federal University, Krasnoyarsk, Russia

⁵Department of Materials Science and Materials Processing Technologies, Polytechnic Institute, Siberian Federal University, Krasnoyarsk, Russia

E-Mail: tgilmanshina@sfu-kras.ru

ABSTRACT

Red mud is solid waste received in the process of obtaining alumina by the Bayer method and is a potentially valuable secondary resource. Currently, the global scientific community is faced with the task of developing environmentally friendly technologies for red mud recycling. Numerous publications discuss both the methods of extracting valuable metals (aluminium, iron, titanium, scandium, rare earth elements), and the use of red mud in building; cement, mineral wool, ceramics manufacturing, etc. This paper summarizes the results of a systematic review of the researches in one specific application area - the extraction of valuable elements (Fe, Al, Na, Ti) from red mud. A total of 162 scientific publications were examined, and 46 of them have been analyzed in the paper. The analysis showed that, depending on the composition of the processed bauxite and the technological scheme for alumina producing, the content of iron oxide (Fe_2O_3) varies from 7 to 70% by mass, aluminium oxide (Al_2O_3) - from 2 to 33% by mass, titanium oxide (TiO_2) from 2.5 to 22% by mass, sodium oxide (Na_2O) to 12.5% by mass. The achieved maximum percentage of iron extraction is 97.5%, aluminium - 89.7%, sodium - 96.4%, titanium - 97%. In conclusion, it should be noted that almost all studies have been carried out only in the laboratory conditions. Technological proposals for the integrated processing of red mud are highlighted as the most effective, including the processes of reduction melting, magnetic separation, leaching with mineral (HCl , H_2SO_4 , HNO_3) and organic ($\text{H}_2\text{C}_2\text{O}_4$) acids. Current researches propose to use microwave, ultrasonic radiation, or plasma technologies in recycling. Considering the results and conclusion in the publications observed it seems difficult to conclude about the cost-benefit methods. As a result of the work, a systematizing table of the methods for extracting Fe, Al, Na, Ti from red mud has been proposed. The authors have made some suggestions on the criteria of a technology that would have environmental, energy and economic benefits.

Keywords: red mud, recycling of alumina production waste, environmentally friendly recycling technologies, red mud processing, recovery of metals from red mud, leaching of metals from red mud.

INTRODUCTION

The problem of processing and utilization of red mud is one of the most important in the metallurgical industry because of its annual obtaining in the production of alumina from bauxite ores in the amount of about 120 million tons per year. Red mud is a product of the Bayer process, which is the main industrial method for the production of alumina from bauxite in the world (it accounts for 95% of the production of alumina) [1, 2]. Bauxite is a mixture of minerals rich in aluminium hydroxide oxides. As a rule, it consists mainly of gibbsite minerals $\text{Al}(\text{OH})_3$, boehmite minerals $\gamma\text{-AlO}(\text{OH})$ and diaspora minerals $\alpha\text{-AlO}(\text{OH})$, composed of two iron oxides, goethite $\alpha\text{-FeO}(\text{OH})$ and hematite Fe_2O_3 , a small amount of rutile / anatase TiO_2 , clay mineral, kaolinite and other impurities in small quantities. The amount of red mud obtained varies greatly depending on the type of bauxite and the method used in the production of alumina, however, as a rule; red mud yield exceeds the alumina yield.

Red mud is a fine material the particles size of which is less than 100 microns, depending on the grain size of the primary minerals. The specific surface area of bauxite is from 10 to 25 m^2/g [2, 3]. Red mud is removed from the process in the form of a pulp with L (liquid) : S (solid) = 2.5-3-3.0, with a Na_2O content in water of 1.0-3.0 g/l. The chemical and mineralogical composition of the sludge is very complex. It is determined by the composition of the initial bauxite and the conditions of its leaching (temperature, dosage of CaO , etc.).

Red mud are stored in sludge fields near alumina refineries, it requires large land areas for sludge fields and entails environmental pollution (changes in the pH of groundwater, leakage, overflow of sludge fields, air pollution with dust). Until recently, it has been typically to dump it into the sea. The destructive effect of red mud is due to its high alkalinity, the presence of various heavy metals and some content of radioactive elements (Ra, U, Th) [3]. The ecological catastrophe regarding red mud ingress in Hungary in October 2010 represents the scale of



the problem of red mud waste recovery. Since 2010, ultrasonic warehousing technology has begun to be used in world practice, in which red mud is filtered using a press filter or a hyperbolic filter to produce cake with a moisture content of 25-30%. This provides not only effective red mud storage, but also reduces the cost of shipping it to the consumer, as well as preparing for the next stages of processing [4].

At the same time, red mud is a potentially valuable secondary resource due to the significant content of valuable metals (Al, Fe, Na, Ti, Si, Ca), as well as rare-earth elements such as Ga, La and Sc. Depending on the composition of the bauxite processed and the technological scheme for alumina production, the content of iron oxide (Fe_2O_3) in red mud varies from 7 to 70 % by mass, Aluminium oxide (Al_2O_3) - from 2 to 33 % by mass, Titanium oxide (TiO_2) from 2.5 to 22 % by mass, Sodium oxide (Na_2O) to 12.5 % by mass. Red mud consists mainly of numerous minerals, for example, hematite Fe_2O_3 , goethite $\alpha\text{-FeO (OH)}$, boehmite $\gamma\text{-AlO (OH)}$, quartz SiO_2 , sodalite $\text{Na}_4\text{Al}_3\text{Si}_3\text{O}_{12}\text{Cl}$, anatase TiO_2 and gypsum $\text{CaSO}_4\cdot 2\text{H}_2\text{O}$, with a small amount of calcite CaCO_3 , oxalate calcium $\text{CaC}_2\text{O}_4\cdot\text{H}_2\text{O}$ and gibbsite Al (OH)_3 . For example, in the processing of diasporite North Ural bauxites with $\mu\text{si} = 10\text{-}12$ at 240°C with the presence of 3-5%CaO, red mud of the following average composition is obtained, %: 6.3 loss by roasting; 14.5 Al_2O_3 ; 9.0 SiO_2 ; 44.5 Fe_2O_3 ; 12.0 CaO; 4.0 TiO_2 . This red mud is represented by sodium hydroaluminosilicate - sodalite (40%), boehmite (4%), hematite (15%), goethite (30%), calcite-dolomite (3%), pyrite (2%) and some other minerals [2].

10-20% of the alumina contained in the initial bauxite and 100-200 kg of Na_2O per 1 ton of alumina are irretrievably lost with dumped red mud. The annual loss of iron with red mud from a large plant is about 0.5 million tons. Therefore, red mud is to be considered as one of the potential sources of alumina, caustic alkali and iron [5].

Red mud is not a particularly hazardous waste due to the high content of caustic soda, but its corrosive activity greatly complicates the choice of processing technology. To reduce alkalinity, seawater treatment, heat treatment and acid neutralization are proposed [5].

In the review articles of recent years, an extensive literary analysis of more than 150 scientific papers has been carried out, which shows the particular relevance of red mud recycling [2-7].

At present, red mud is used after water washing in the manufacturing of construction materials (bricks, tiles, Metlakh tiles, cement, etc.), materials for land reclamation and paving, additives for the paint and colored glass, weighting agents in the oil industry [8, 9]. Adding of up to 10-15% of red mud to a blast furnace for smelting cast iron is also used in practice. Besides, red mud can be used for land reclamation. On the other hand, it is necessary to remove of valuable elements such as Fe, Al, Ti, Na, V, Sc and Ga. Considering the mineralogical diversity of bauxite ore and red mud, it is difficult to extract valuable elements entirely during its processing. These methods are promising and highly effective, however, only a few of them have been introduced on an industrial scale.

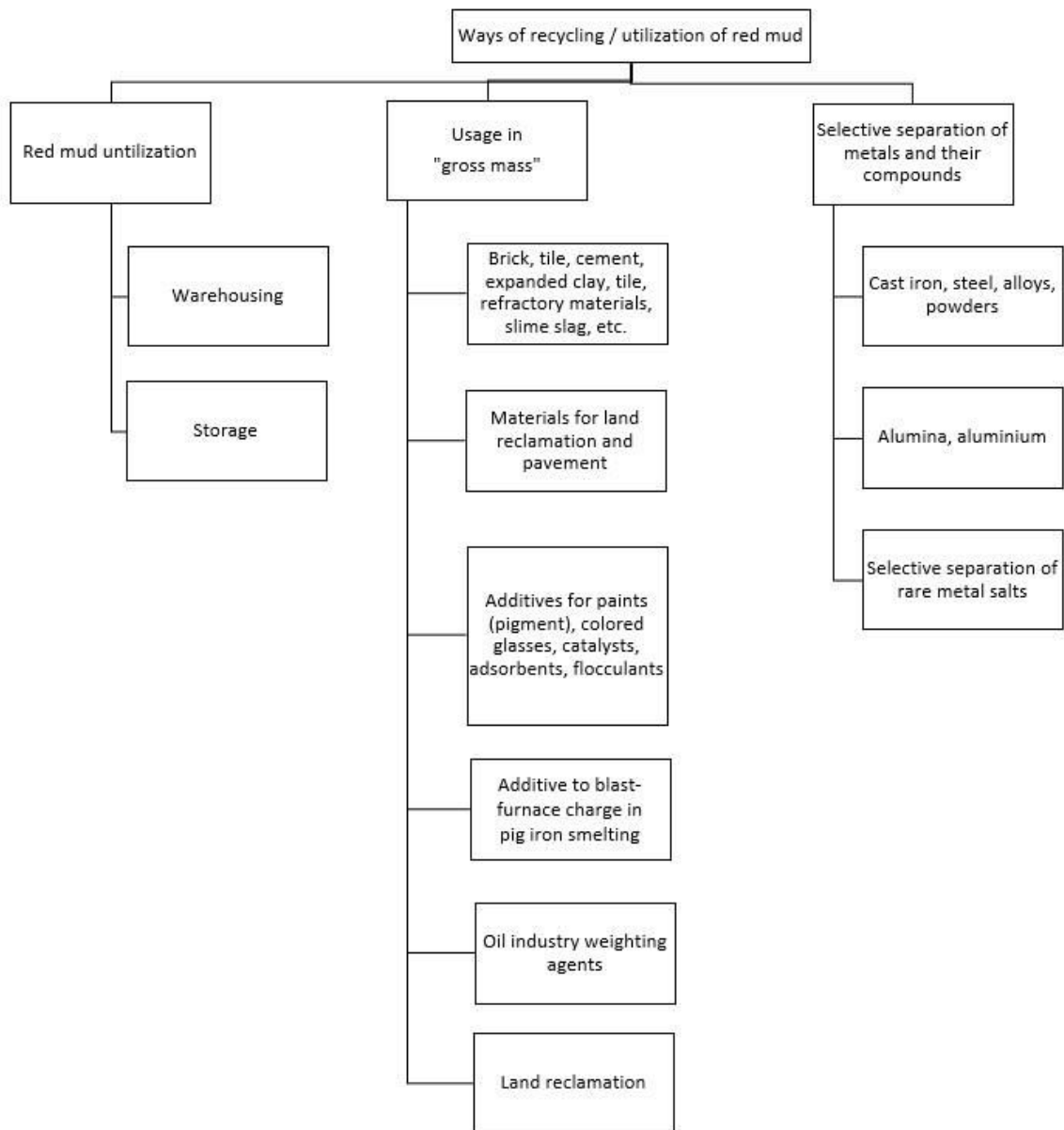


Figure-1. The ways of red mud recycling / utilization.

Basic Methods for Extracting Valuable Elements from Red Mud

Among the main methods for extracting valuable elements from red mud, the following ones can be

distinguished: magnetic separation (to extract iron), pyrometallurgical processes, hydrometallurgical (leaching processes with mineral and organic acids), biometallurgical processes (biosorption and bioleaching).

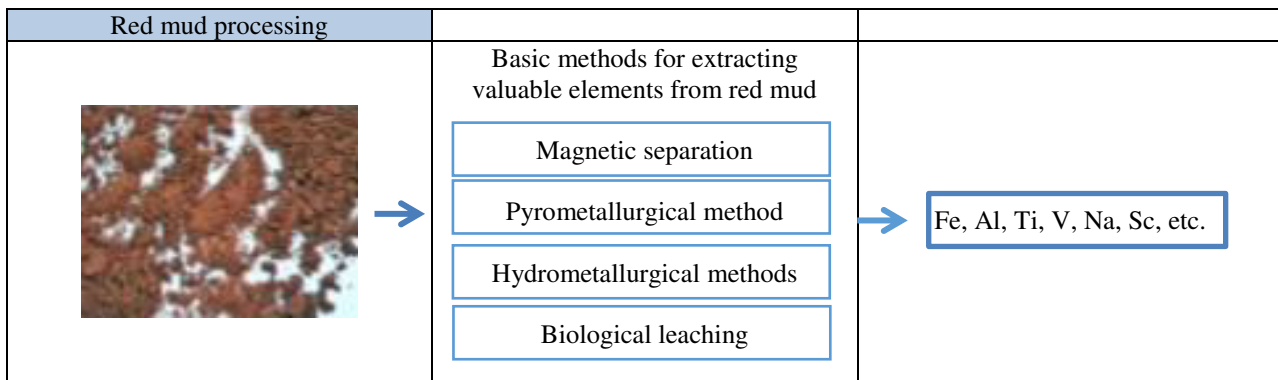


Figure-2. Methods of red mud processing.

Magnetic separation: As a rule, iron oxide in red mud is the main component, and the concentration of Fe in red mud is varied from 7 to 70 % by mass depending on the iron content in bauxite. The extraction of iron from solid waste can be carried out by a physical process, i.e. magnetic separation. The obtained magnetic products can be used for the production of iron by sintering or as a pigment for ceramics manufacturing [11, 12]. Magnetic separation system with a high-intensity alternating magnetic field provides an increase in recovery compared to conventional magnetic separation, especially for pre-processed and ground red mud [13]. The cost of superconducting magnets is economically acceptable from the point of view of industrial applications [14]. Different content of iron oxide in raw red mud also affects the separation result when using the HGSMS system for the separation of extremely fine red mud with a particle size of less than 100 microns [15]. The result of the experiments shows that the effectiveness of this method when using red mud, containing 29.79% Fe_2O_3 , is higher than that containing 58.74% Fe_2O_3 . The concentration of iron oxide in the remnants of the red mud with rich and poor iron content was 52.0% and 14.1%, respectively. The stronger the magnetic field intensity, the higher the degree of extraction of iron oxides in the process of magnetic separation.

However, iron agglomerates with a large number of poor magnetic and non-metallic materials will also be easily captured and recovered in magnetic concentrates regardless of the type of red mud, and this will lead to a decrease in the quality of the concentrate. If strongly magnetic materials of iron are primarily extracted from the red mud by a medium-intensity cylindrical magnetic separator, and then the obtained tail is processed using a vertical Slon ring and a high magnetic efficient separator with the creation of a pulsating magnetic field and production of a weakly magnetic iron concentrate, then an iron concentrate containing $\text{Fe} \geq 54\%$, the degree of iron extraction is 28–35% can be obtained [2, 10-15].

Pyrometallurgical method: As a rule, when metals are present in complex combination with other metals and non-metals, i.e. form complex compounds, they are often difficult to separate by the process of physical processing. Pyrometallurgical processes are widely used around the world to extract valuable elements

from red mud, and this requires a large energy consumption during sintering or roasting of raw materials. An example can be the extraction of iron from red mud by the pyrometallurgical method. Maintaining the optimum roasting or sintering temperature is critical to the quality of the cast iron and the composition of the slag. The stability of refractory materials is also determined by the composition and temperature of the slag and molten iron [18-22].

Hydrometallurgical methods: In most cases, the predominant method is hydrometallurgical processes, although these methods do not allow to process red mud completely and consequently, they are economically unprofitable.

The hydrometallurgical methods applied to recover metals from red mud are a modified version of the conventional methods used to obtain metals from primary ores. Leaching is carried out mainly with mineral acids to convert metals to soluble salts.

A high degree of iron recovery is equal to 97.46% can be achieved by calcining at 600°C, followed by leaching using H_2SO_4 with a concentration of 6M [23]. Moreover, the possibility of extracting aluminium (64.40%) was also investigated. The dissolution of aluminium was slower than the dissolution of iron. Oxalic acid can also be used as a leach for the extraction of iron from red mud [22, 24]. The degree of iron extraction in this case was about 96%.

Organic and mineral acids, such as sulfuric, citric and oxalic acids were used separately or as mixtures. Optimum aluminium recovery was obtained (leaching of 96% alumina) using a mixture of citric and oxalic acids in a 2:1 ratio at pH 1.5 adjusted by the addition of sulfuric acid. Compared to leaching using H_2SO_4 at pH 1.0 the mixed leach showed a higher extraction of aluminium but was less economical due to the high cost of organic acids.

Titanium can be easily recovered using hydrometallurgical method by sulphuric acid leaching recovery degree can reach 64.5% at leaching at 60° C in sulfuric acid with concentration of 6N and ratio S (solid) : L (liquid) equal to 1:20 [25-28]. The authors [27] attempted to extract alumina and alkali from the red mud of the Ural plants by leaching with pregnant liquor and recycled aluminate solutions as well as pure NaOH solution. With such leaching Al_2O_3 , Na_2O , SiO_2 are



transferred to the solution in ratios close to the composition of sodium hydroaluminosilicate. The resulting silica-contaminated aluminate solutions were desilified at a temperature above 220°C with the addition of lime to form a sodium-calcium hydrosilicate in the precipitate from which the alkali can be regenerated. Up to 60% of Al₂O₃, 80% SiO₂, 65% Na₂O can be extracted from red mud with the solutions of NaOH, KOH and recycled aluminate solutions at 200-240°C.

The use of a vast amount of highly concentrated acids is a potential impediment to the commercial use of direct acid leaching. In addition, the formation of secondary pollutants and by-products of hydrometallurgical recovery is also a limitation of this method of processing and imposes additional responsibility for potential environmental problems arising from the use of hazardous substances during processing at the industrial plants.

Compared to the process of leaching with mineral acids, a combined bio hydrometallurgical process is increasingly considered as an environmentally friendly process ("green" process) for the extraction of valuable metals from red mud. Green process attributes include environmental cleanliness, low energy consumption and costs, and flexible technology requirements [29-32].

The results of hydrometallurgical process of recovering of rare-earth elements are presented in [33, 34].

All the above-described results of the works with the references to authors are presented in [2]. The methods of waste flotation enrichment for the production of iron or aluminium containing concentrates have also been proposed [35-37].

Pyro-hydrometallurgical methods: Alumina can also be recovered from the iron-enriched red mud by sintering treatment [38]. In this method, the addition of sodium ferrite (Na₂O·Fe₂O₃) to 10-12% and the increase of sintering time to 30-40 minutes contributed to the formation of insoluble salts, for example, 3CaO·Al₂O₃, Na₂O·Al₂O₃·2SiO₂ и 4CaO·Al₂O₃·Fe₂O₃ which could improve the degree of alumina leaching. Based on the thermodynamic analysis such parameters as the influence of sintering temperature, reaction behavior and composition on the alumina extraction was also investigated [39].

Under favorable sintering conditions, the recovery of alumina and iron oxide can reach 89.71% and 60.67%, respectively, during the subsequent leaching and magnetic enrichment process (48 kA · m⁻¹). A high recovery rate of 75.7% Al and 80.7% Na can also be achieved by co-firing with soda and lime followed by water leaching of the calcined products. It was obtained that mixing red mud with a certain amount of calcium carbonate and then fusing this mixture at 1400°C makes it easy to achieve high recovery of aluminium and sodium due to the phase conversion of red mud to (Na, Ca)_{2-x}(Al, Fe³⁺)_{2-x}Si_xO₄ (0,2 ≤ x ≤ 0,5).

Combined pyro- and hydrometallurgical process can also be used to extract elements from aluminium, iron and titanium. The detailed description of these processes

with the references to the primary sources are presented in the review articles [2, 6].

Biological leaching: Bio metallurgical waste processing is a new and very promising technology especially for iron-oxide waste being an environmentally friendly, energy-saving method [29-32]. The mechanisms of action of microorganisms that contribute to the dissolution of iron minerals can be divided into two types. In one case, microorganisms emit a complex of different organic acids that support the dissolution of the corresponding iron minerals with Fe²⁺ and Fe³⁺; another group of microorganisms acts as reducing agents and facilitates Fe³⁺ transition to Fe²⁺ and bivalent iron compounds have higher solubility than trivalent. It was determined that the *Desulfuromonas palmitatis* bacteria are able to dissolve a part of the iron minerals from bauxite. In particular, 95% of amorphous ferrihydrite can be dissolved, while the degree of dissolution of crystalline hematite does not exceed 9%, and crystalline hematite is less than 1.2%. At the same time, high value of pH in red mud limits the application of a biological leaching method [31]. Biological leaching of aluminium from red mud was studied in [31]. The results of biological leaching experiments by microorganisms consisting of wastewater sludge bacteria, fungi such as *Aspergillus Niger*, *Penicillium notatum*, *Penicillium simplicissimum* and *Trichoderma viride* showed that 75% aluminium can be recovered at the concentration of the acids produced by *P. Simplicissimum* which composes 10 volume percent of initial red mud volume.

As for the future methods of extracting valuable metals from red mud they should be more environmentally friendly and cost-effective so that red mud is considered as a mineral resource or building material, and not as solid waste.

In the process of processing, it is not advisable to extract valuable metals from red mud separately or only one particular element. As a result, the production should be designed and developed for the processing of red mud with complex extraction of Al, Na, Ti with the main extraction focus on iron in order to make processing more economically feasible. The metal recovery waste can be used as a raw material for the preparation of building structures, catalytic materials, adsorbents, etc., which is advantageous in terms of minimizing environmental impact. In addition, it is not advisable to load red mud into a blast furnace with iron ore due to the presence of a large amount of alkali and alkaline earth metals in them which will cause lining erosion. The mineral and chemical compositions of red mud are crucial for the selection of mineral acids and the extraction of valuable elements. To recover Al, Na, Ti a hydrometallurgical method is widely used in practice usually involving pre-preparation and firing, acid leaching, solvent extraction, precipitation and calcination.

At the same time, when extracting valuable elements such as scandium, co-dissolving and co-extracting of valuable elements may affect the method efficiency. In addition to selectivity studies, it is also



necessary to develop a new extraction system with high selectivity and low acid consumption.

It is also interesting to note the emergence of new methods for processing red mud such as microwave heating, quenching in liquid nitrogen. Thus, the solution of the problem of extracting Fe, Al, Na, Ti and other rare earth elements from red mud is significant due to the increasing of iron ore prices and a shortage of rare earth resources worldwide. It is highly important and urgent to

develop an environmentally friendly and efficient technology for integrated processing of industrial waste [35-37] including red mud for which it is necessary to carry out fundamental research work.

The latest research in this field is given in [2, 6, 39].

The main results of the studies on the extraction of metals from red mud are summarized in Table-1.

Table-1. The main results of the studies on the extraction of metals from red mud.

Process	Metal	Recovery	Process parameters
Magnetic separation	Fe	77%	Magnetic separation with low magnetic field intensity - LIMS,
	Al	10%	Magnetic separation with high magnetic field intensity - WHIMS
	Fe	-	High-frequency alternating magnetic field for red mud after its processing and grinding
	Fe	28-35%	Magnetic separation system with high magnetic intensity of superconducting magnetic field
Pyrometallurgical	Fe		Reduction smelting in the range of 1200-1500 °C with the separation of cast iron/slag phases
	Al		
	Ti		The reduction of TiO ₂ , Al ₂ O ₃ and iron into cast iron by reduction smelting
Hydrometallurgical	Al	87,8%	45% solution of NaOH, CaO/red mud* = 0,25, L/S = 0,9, 0,8 MPa, 200 °C, 3,5 hours
	Na	96,4%	7% solution of NaOH, L/S = 3,8, 0,9 MPa, 170 °C, 2 hours
	Ti	97 ± 2%	1gr of red mud is leached by H ₂ SO ₄ /HF = 1/5 and some drops of the concentrator of HNO ₃ , Cyanex 301 and 302
	Fe	96%	1 mol of oxalic acid · 1-1 at 75 °C for 2 hours
	Ti	64,5%	Leaching by 6N H ₂ SO ₄ at 60 °C, S:L = 1:20
	Fe	46%	
Al	37%		
Pyro-hydrometallurgical	Al	80,7%	Calcination with soda ash: molar ratio Ca/Si ≈ 2,0, Na/Al ≈ 1.0 ~ 0.5mass. % of coal, 1000 °C, 3 hours; leaching by water L/S = 2; 60 °C, 15 minutes
	Na	75,7%	
	Fe	97,46%	Calcination at 600 °C followed by acid leaching by 6M H ₂ SO ₄
	Al	64,4%	
	Fe	93-94%	Reduction smelting with soda followed by leaching by H ₂ SO ₄
Ti	96%		
Al	89%		
Biological	Ti	84,7%	Smelting: 1/1/0,165/19,5= red mud/limestone/coke/bentonite, 1100 °C, 1 hour; 1550 °C, 30 minutes; acid leaching by 30% of H ₂ SO ₄ , 90 °C; recovery 5% of D2EHPA
	Al	75%	Acid leaching with <i>P. simplicissimum</i> , 3 hours
Complex (integrated)	Fe	95%	Leaching amorphous ferrihydrite by fungi <i>D. Palmitatis</i> , low pH
	Al	75,7%	Calcination with soda at 1000 °C for 3 hours, leaching, magnetic separation using Davis magnetic tube tester (XCGS-99, 50 mm ID)
	Na	80,7%	
Fe	dependence on the conditions applied		
Al	89,71%,	Reduction smelting of mixture of red mud with soda at 1300 °C with preliminary and subsequent holding at 800 °C and at 600oC, acid leaching, magnetic separation	
Fe	до 61,78%,		

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