

Materials sustainability for environment: Red-mud treatment

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Abstract Bayer's process revolutionized the extraction of aluminum from the bauxite ores. However, the hydrothermal extraction of alumina is associated with the generation of a byproduct, red-mud consisting of undissolved solids composed of iron oxides, sodium aluminosilicates, titania, silica and rare earth elements. The accumulation of red-mud (or bauxite residue) in the world is 30 billion metric tons produced at a rate of 125 million tons per annum (2013). Utilization of red-mud for constructional purposes, wastewater treatment, metallurgical products, and pigments are listed. Metallurgical processing efforts of red-mud to generate various value added products such as pig iron, direct reduced iron slag wool, magnetite, titania, iron carbides are presented in the article.

Keywords red-mud processing, waste management, sustainability, valorization

1 Background

Bauxite is named after a village, Les Baux, in southern France. It is the primary aluminum ore, a laterite is composed of aluminum hydroxides and compounds of iron, silicon and titanium [1]. The aluminum hydroxide comprises a mixture of gibbsite, böhemite and diaspor. These compounds generally have a variable crystal structure, hardness, dehydration temperature. Very rarely are all of these three mixed hydroxides present in a bauxite ore from a specific source. Other mineable sources for aluminum include a large variety of rocks and minerals such as aluminous shale and slate, aluminum phosphate rock, high alumina clays or kaolinities [2].

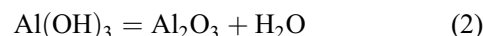
Bauxite deposits are found in all continents of the world due to their extensive methods of formation. Australia and Guinea have the largest reserves 2 billion tons. Jamaica,

Cameroon, China, Ghana, Yugoslavia, Surinam, Russia, Brazil, India, Greece, France, Guyana, Dominican Republic and Haiti [3] follow these. Bauxite processing is conducted in Australia, Brazil, Guinea and Jamaica. Very aptly, it is plausible to assume that it has a different composition in every source due to differences in origin (Table 1).

Many bauxite deposits are found near the surface; hence open-pit mining is used process them. Bauxite was originally ground dry, but now wet rod mills are being used in conjunction with a cyclone. Properly sized bauxite is transported to the digestion tanks where leaching of the aluminum hydroxides take place in concentrated sodium hydroxide solution [1,5] (Eq. (1)). The flowsheet for the process is shown in Fig. 1. The process is conducted in autoclaves at temperatures ranging from 140 to 270 °C and pressures in the neighborhood of 4137 kPa.



The pregnant leach solution is transferred to cyclones/settling chambers to separate the coarse precipitate from the leach solution. This process is called the counter current decantation where the underflow of each cyclone unit is passed on to the next cyclone while being elutriated in the solution from the next cyclone in series. The decanted slurry is then fed to the pressure filters to separate the fine precipitates. This precipitate is called the red mud, which is discarded, to properly managed tailings ponds. Red mud is mostly composed of constituents not dissolved in the leaching process mainly iron oxides. The solution is then left for precipitation of aluminum hydroxide. This hydroxide is calcined to generate smelting grade alumina (Eq. (2)). Alumina is reduced to aluminum in the Hall Heroult process cell employing a molten cryolite bath.



In order to completely precipitate silica in the bauxite process, finely ground lime is added during the digestion to precipitate a less soluble compound cancrinite, which

Table 1 The compositions (%) of some bauxite ores around the world [4]

Source	SiO ₂	TiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	H ₂ O (combined)	Total
France	0.8	2.8	58.6	26.2			10.9	99.30
France	0.8	3.3	76.4	4.8			14.3	99.8
France	0.29	0.8	60.6	26			10.4	98.09
France	13.3	2.4	63.7	5.5			14.3	99.2
Romania	1.49	3.12	59.66	23.66			11.81	99.74
Romania	0.8	2.8	65.5	21.3			9.96	100.36
Yugoslavia	0.89		51.85	26.82			19.97	99.54
Italy	2.79	1.27	57.6	26.55			11.71	99.92
Italy	7.91		58.85	18.62	0.30	0.37	13.27	99.32
US, Alabama	2.90	3.40	58.21	3.60			31.89	100
US, Arkansas	2	3.5	62.25	1.66			30.31	99.52
US, Arkansas	10.13		55.09	6.08			28.99	100.29
US, Georgia	0.62	1.05	64.91	0.28			33	99.86
British Guiana	2.73	0.1	64.38	0.5			32.29	100
British Guiana	1	1.1	70.90	0.8			26.30	100.10

eliminates the silica from the leach liquor. It is called as the desilication product (DSP). Additionally, lime also prevents the formation of sodium titanates, which form a gel on the aluminum compounds, lowering the aluminum extraction efficiency. Perovskite (CaTiO₃) is formed in this process. In addition, dissolution of some carbonate containing compounds and atmospheric carbon dioxide leading to the precipitation of Na₂CO₃. Addition of lime forms CaCO₃ and NaOH. Instances where high P₂O₅ is present, CaO is added to form carbonate apatite. Failure of addition of lime causes the buildup of elements in the recycled liquor and cause for the loss of caustic.

2 Nature of bauxite residue

Bauxite residue is composed of phases undissolved during the caustic digestion of bauxite ore. It is alkaline and consists of very fine particulates (< 10 μm). The amount of bauxite residue generated by an aluminum refinery depends on the source of the bauxite ore and the processing conditions. Typically, 0.3 to as high as 2.5 tons of alumina production can yield 1 ton of residue; 1.5 to 4 tons of residue is produced per ton of Aluminum. The residue essentially consists of hematite (20–45 wt-%), alumina (10–22 wt-%), rutile (4–20 wt-%), lime (0–14 wt-%), silica (5–30 wt-%) and oxides of sodium (2–8 wt-%). In addition to these products, a material called DSP which consists of unrecovered portions of alumina and silica is present. DSP usually consist of sodium aluminum silicates, cancrinite, kaolinites. In some red-muds, aluminum is also present as

gibbsite, diaspore, aluminogothite. Table 2 shows the compositions of red-mud produced at alumina processing plants in some locations around the world.

3 Utilization of bauxite residue

Large-scale utilization of red mud has been the focus of many researchers around the world but the feasibility of their proposed application have not been economically favorable. High costs associated with digging, transportation, processing the residue, logistics [7]. Additional, cost associated with storage neutralization, impoundments, disposal and cleanup have exacerbated the issue. Diverse applications such as cement, soil amelioration, base metal extraction, rare-earth meal extraction, ceramics, geopolymers, road fillers, brick production, heavy-metal adsorbents etc. However, very few of these applications proved to be economically feasible. Almost all of these applications rely on dewatering or drying, so an additional overhead cost becomes significant.

The application of bauxite residue in producing portland cement has been a matter of research since 75 years. The aluminum and iron content of red mud help the cement in terms of strength and settling characteristics. Sodium and chromium present in large amounts can be a problem. The similarity of red mud composition to that of calcium sulfoaluminate ((CaO)₄(Al₂O₃)₄SO₃) which gives ultimate strength to the cement matrix encouraged researchers to research on this topic [8]. Employing a proper composition of red mud, lime and bauxite, unusually high strengths could be achieved in the cement [9–12].

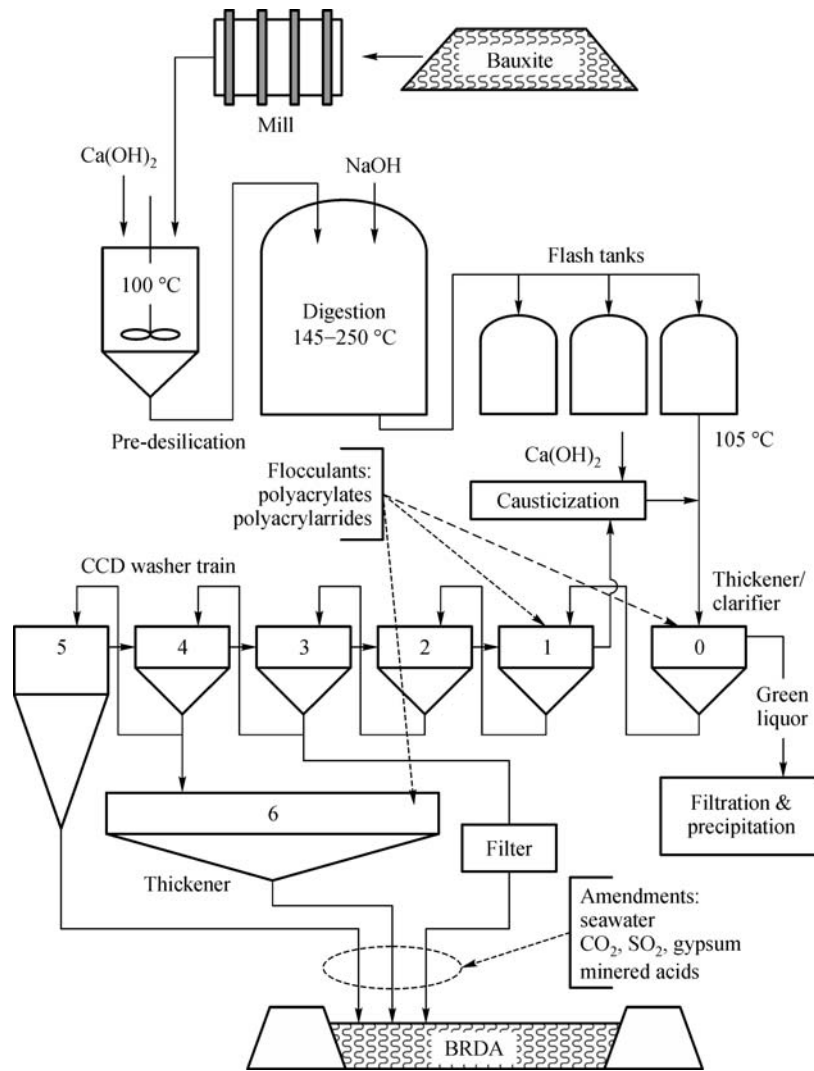


Fig. 1 Pictorial flowsheet of the Bayer process [5]

Table 2 Chemical compositions (wt-%) of various red-mud around the world [6]

Plant, location (country)	Fe ₂ O ₃	Al ₂ O ₃	TiO ₂	SiO ₂	Na ₂ O	CaO	LOI
Alcoa of Australia (Pinjarra, Australia)	36.2	17.1	3.9	2.8	1.6	3.9	10.4
Alcan, Arvida (Quebec, Canada)	27.4	28.4	9.8	14.3	8.8	1.3	9.9
Hungalu (Ajka, Hungary)	42.1	14.8	5.2	13.5	8.9	6.1	8.2
Alcan Jamaica (Kirkvine, Jamaica)	49.4	13.2	7.3	3.0	9.6	5.7	7.2
Kaiser (Gramercy, Louisiana, USA)	51.5	15.0	6.7	1.7	1.0	7.0	9.3
Alcoa alumino (Pocos, de Caldes, Brazil)	29.6	21.9	4.4	17.5	8.3	2.9	11.5
Indian plants							
Hindalco (a) causticised	28.1	21.9	15.6	7.5	4.5	10.2	12.2
(Renukoot) (b) uncausticised	33.1	18.2	19.6	8.8	5.8	2.7	8.9
Nalco (a) 1995	54.8	14.8	3.7	6.4	4.8	2.5	9.5
(Damanjodi) (b) 1991	68.5	12.5	2.8	3.0	2.5	0.1	10.2
Balco (Korba) causticised	27.9	19.2	16.4	7.3	3.3	11.8	12.6
Indal (Muri)	24.5	24.3	18.0	6.2	5.3	–	–
Indal (Belgaum)	44.5	19.2	13.5	7.0	4.0	0.8	10.0

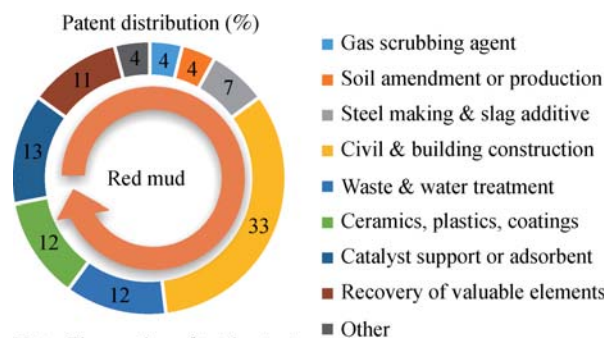
The dewatered red mud mixed with a suitable binder could be used in road construction. The red mud from Alcoa in Australia was converted to a suitable product called red sand that was used to construct a highway between Perth and Bunbury [4]. The bauxite residue mixed with fly ash could be used to make stable class I bricks. The presence of sodium ions reduces the weathering resistance, hence for high-level applications, the sodium ions were replaced by the addition of lime etc. In the mid 1990's, a sport pavilion was set up by the Jamaican Bauxite Institute using the residue from Ewarton.

The high alkalinity of red mud makes the material to have a good neutralization capacity. Addition of 5 % Gypsum to the soil increased its water retention and nutrient utilization capacity. Alcoa, made a carbonated residue from red mud using carbon dioxide. This was neutralized to below pH 8 under the trade name Alkaloam. This product ensured phosphorous retention capability and reduced the leachability of phosphorous. Red mud could also be used as an adsorbent for fluorides, arsenic and heavy metals [13,14].

The high iron content in red mud made it a viable component in pigments. They produced different reddish brown shades. Nalco red mud was used to produce paints that passed IS: 123-1962 and IS: 446-1969. Wood substitute was prepared using the Nalco red mud employing 50% natural fiber and polyester resin. This was used to produce wood of high strength, water resistance, weatherability and fire resistance.

Geopolymers have a number of potential advantages compared to conventional portland cement. Lower carbon-dioxide emissions are associated with geopolymers productions. The building block is a silico aluminate chain $(-\text{Si}-\text{O}-\text{Al}-\text{O}-)_n$ that is formed by the dissolution of silica and alumina in an alkali. Pontikes and Angelopoulos manufactured geopolymers of Fracture toughness $0.9 \text{ MPa}\cdot\text{m}^{0.5}$ and a compressive strength of 25.1 MPa on the addition of 10% sodium silicate to red mud [9]. The products manufactured had a high compressive strength, excellent water permeability, bulk density, excellent thermal stability at high temperatures. However, they were associated with low flexural strength and lack of resistance in freeze thaw cycles.

One of the main areas researched is metal recovery from red mud. The high iron oxide content in some red-muds, make it a potential source for iron production. Added to this the minor constituents of aluminum and titanium. Numerous work have been performed to recover iron as pig iron, DRI and the slag produced was used for extraction of Al, Ti and other rare-earth elements. A slag wool for roofing has been manufactured by NTUA in Greece but the commercial application has not been realized due to the ratio hindrance due to the radioactivity of the material. Figure 2 shows the percentage of research done in above areas.



Note: The number of total patents mentioned above is 734 (1964–2008).

Fig. 2 Research done by some authors in red mud utilization until date [15]

4 Metallurgical treatment of bauxite residue

4.1 Extraction of high volume low value products from red-mud

The bauxite residue contains many elements in extractable compositions as stated under the nature of red mud section in the paper. Iron, aluminum, sodium, calcium, titanium in their oxide form make up most of the red mud chemically. Extraction of these metals from red mud has been a focus of many aluminum companies. The high valuable elements niobium, gallium, scandium, vanadium, and thorium become significant in composition when the base metals are extracted.

Iron is invariably the predominant constituent in red mud. In almost all-major utilization of red mud, iron was recovered in an extractable form and the nonferrous portion was taken for further processing [16]. Logically, iron being the major component of most of the red muds; it was the first element to be extracted. There have been methods investigated on this subject [17]. The major methods tried in the past were magnetic separation of the reduced material from red-mud, addition of the material for blast furnace operation for iron extraction. Due to relatively high amounts of soda, alumina, titania, phosphorous and vanadium in the unprocessed red mud, direct charging of the residue for iron making was not an option [18]. A pre-concentration step was usually required to remove these undesirable constituents. Following the pre-concentration procedure, it was charged into the blast furnace through the tuyeres.

There have been many attempts to recover aluminum from red mud prior to removal of iron. Zhong et al. recovered alumina and alkali from red mud hydro chemically by the formation of “grossular hydrogarnet”. Sodium, aluminum and calcium are present as sodalite and cancrinite in the DSP [19]. Li et al. also generated the

hydrogarnet by destroying the structure of sodalite or cancrinite. In the hydrogarnet process, iron was substituted for aluminum in DSP [20]. The main factors influencing the reaction were reaction temperature, Na₂O to Al₂O₃ ratio, sodium concentration and reaction time. The authors claimed that this process was effective in treating a red mud (Fe 20%). Zhong et al. have employed an extra processing step in converting the DSP to NaCaHSiO₄ to recover alumina prior to hydrolysis of the recovered portion for alkali recovery. The authors succeeded in recovering 88% Al₂O₃ and 96% of Na₂O from red mud employing this method [19].

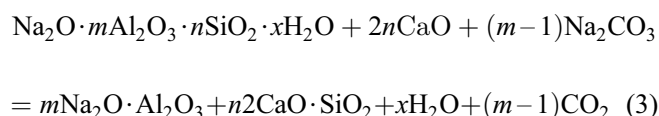
Chemical and biological leaching of aluminum from red mud by employing organic acids was performed by Vachon et al. Sulfuric, citric and oxalic acids were employed individually in leaching experiments. Solubilization of 96% was achieved using 2:1 citric to oxalic acid at pH 1.5, which was stabilized using sulfuric acid. Nevertheless, this process was not adopted commercially considering the high price of organic acids. Bioleaching was also carried out by Vachon et al. *Aspergillus niger*, *Penicillium notrum*, *Penicillium simplicissimum* and *Trichoderma viride* were used and 75 % aluminum was recovered using a pulp density of 10%. Al leached readily into the solution when under the influence of fungi compared to when using pure acids [21].

A hydrothermal process employing caustic was developed to extract Al₂O₃ in red mud to produce sodium-aluminate hydrate. It consisted of a high pressure leaching process. The optimum conditions of leaching for the first stage were 45% NaOH solution, CaO to red mud mass ratio 0.25 and L/S ratio at 0.8 MPa at 200 °C for 3.5 h. In the second stage, the residue used for extracting Al₂O₃ in the first stage was subjected to pressure leaching with 7% NaOH, L/S ratio 3.8, 0.9 MPa pressure at 170 °C for 2 h. In this dual stage, caustic-pressure leaching of red mud, 87.8% Al₂O₃ and 96.4% Na₂O were extracted. The final residues obtained contained less than 1% Na₂O hence could serve as a feedstock for construction materials [19].

Optimum fluxing, smelting and leaching practices were investigated by Bruckard et al. to maximize the oxidative smelting of bauxite residue and thus maximize alumina recovery while rejecting iron. The addition of calcium carbonate to the charge increased the CaO/SiO₂, which concomitantly increased the alumina recovery during water leaching of the slag [22]. Al and Na could be readily recovered due to the phase transformation into (Na, Ca)_{2-x}(Al, Fe³⁺)_{2-x}Si_xO₄. The slag was subjected to water leaching at 60 °C, recovery of 55% Al and 90% Na could be achieved at 50% pulp density. The leach-liquors were recycled back into the Bayer process. It was observed that addition of sodium carbonate to the melt made the formation of sodium aluminate more favorable thereby recovering most of the aluminum but they increased the possibility of attack and damage to

the refractory. Alternatively using the MPE model, reductive smelting was suggested which would produce pig iron and a slag of definite composition to allow recovery of Al, Na and Ti. This process was further improved by Li et al. Sodium ferrites were added to the melt and the sintering duration was increased to about 30–40 min. This allowed the formation of many insoluble salts, like 2CaO·Al₂O₃, Na₂O·Al₂O₃·2SiO₂ and 4CaO·Al₂O₃·Fe₂O₃, which improved the leaching efficiency of aluminum. The effect of sintering temperature, relevant chemical reactions, and formulation on the alumina recovery were investigated on the basis of the thermodynamic analyses [23].

Zhou et al. developed simultaneous soda lime reductive sintering process. This process was developed for the recovery of alumina as sodium aluminate (Eq. 3). The hematite was converted to metallic iron/wustite and partly to magnetite. Magnetic separation in the Davis tube was employed to separate the iron fraction. The aim of this study was to recover Al, Na and Fe from the red mud. This required a very fine particle size of red mud particles around 75 μm. It was found out that Al and Na recovery after water leaching was about 75.7% and 80.7% respectively. The phases after reduction include sodium aluminosilicate, larnite along with minor amounts of calcium aluminoferrite, melitite and wustite [24].



As seen from Eq. (3), the recovery of Na, Al was mainly due to the dissolution of sodium aluminosilicate phase. It was important to maintain an optimum Ca/Si ratio because the addition of CaO promotes the dissolution of Al and Na by reducing the Si content of the sodium aluminosilicate phase. It was observed that an inefficient magnetic separation was found because the ferrous phases were agglomerated together with non-ferrous phases such as larnite and sodium aluminosilicate phase. Agglomeration of these phases caused a reduction in phase liberation. Raspopov et al. carbothermically smelted red-mud to form cast iron, the slag produced was used to extract nonmetals and make sub-building components [25].

High iron (> 45% Fe₂O₃) red-mud was passed through a high-intensity magnetic separator, then deflocculated and neutralized with hydrochloric, or sulfuric acid. The filtered portion was dried and subjected to magnetic separation. The magnetic part was dried, pelletized and later used as a feedstock for iron making. The non-magnetic portion was utilized for blocks, bricks, tiles and mortars [26].

Jamieson et al. have tried magnetic separation on the red sand produced by Alcoa World alumina Australia. Red sand is the bauxite residue, which has a size greater than 90 μm. This coarse product represents a potential resource of

20000 tonnes per day. The authors researched on processing the red sand through the low intensity magnetic separator (LIMS) and wet high intensity magnetic separator (WHIMS) to produce some fractions that have a higher value than the red sand. Three fractions were produced; one, which is 40% Fe, one that had a high silica content (93% SiO₂), and a third portion which had iron and silica and can be used as a general fill [27].

Li et al. processed high iron containing red mud and employed it to recover alumina and ferric oxide by the process of reduction-sintering, leaching and magnetic beneficiation. Thermodynamic analyses show that iron oxide reduction using carbon to iron and conversion of alumina to sodium aluminate was theoretically possible. Higher the sintering temperature, higher is the alumina recovery. The sodium ferrite was converted into sodium aluminate. However, at temperature higher than 1320 K, calcium ferrite formed decreased the alumina recovery. Li et al. suggested that pulsating magnetic current utilization would provide better separations than the conventional magnetic current. The high magnetic separation system (HGMS) sets up a strong magnetic field than ordinary ferromagnetic core electromagnets. They were efficient in separating fine particles in liquid suspensions. HGMS could separate magnetic particles of size less than 100 μm [26]. The problem in using high magnetic fields was that the magnets carried some weakly magnetic substances including small intermetallics away. Zhu et al. recovered a strategy to recover metallic iron from red mud by conducting a carbon based sodium carbonate roasting. The metallization obtained was about 99.35% but the recovery of iron was low [28].

Liu et al. studied the direct roasting process followed by the magnetic separation to separate the iron out and building materials were prepared using the aluminosilicate residues. The authors proposed that the optimum reaction parameters were ratio of carbon powder: ratio of red mud at 18:100, ratio of additives: ratio of red mud at 6:100, roasting at 1300 °C for 110 min. The magnetic concentrate was found to contain 88.77% Fe, metallization ratio of 97.69%. This translates to the recovery of 81.40%. The non-magnetic portion essentially rich in aluminosilicates were cured for making bricks. These bricks have a compressive strength of 24.10 MPa on the addition of 13% lime. A phase transformation of the crystalline phase nepheline in the original residues to gehlenite in the cured bricks was observed. Zero emission of red mud wasted was achieved as the non-magnetic portion obtained was reused as building materials [29].

A combined pyro- and hydrometallurgical process could also be employed to recover aluminum, iron and titanium elements [30]. This process represented in Fig. 3 was a modified version of DLM process for sintering, pelletizing conventional ores. Red-mud was first dried to the desired consistency and mixed with coal, lime, and sodium carbonate. This mixture was subjected to reducing

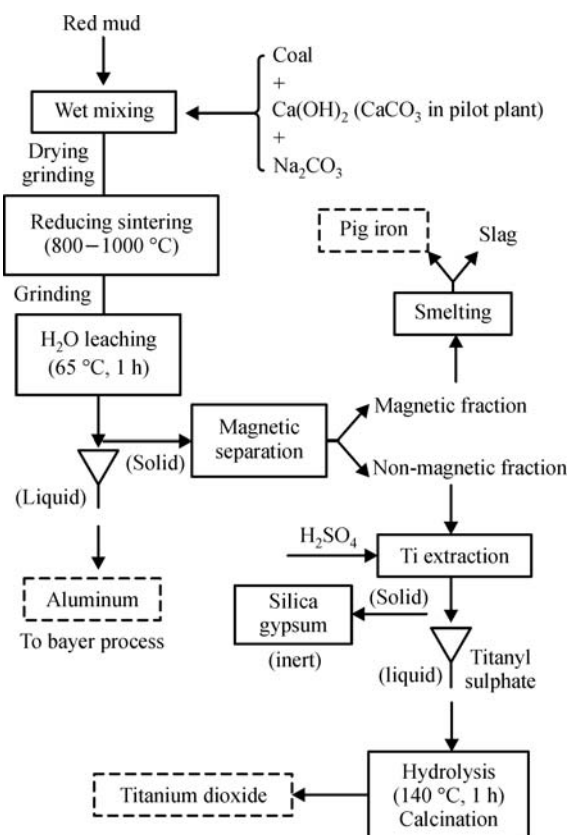


Fig. 3 Process flowsheet for metal extraction from red mud employed by Piga et al. [30]

sintering at 800–1000 °C. The resulting fine sintered products underwent water leaching at 65 °C for 1 h and 89% aluminum involved in the products were leached out. The filtrate obtained can be recycled in the Bayer process. The residue was subjected to high-intensity magnetic separation. The titanium in the non-magnetic portion was taken to the solution by leaching with sulfuric acid. The titanyl sulfate was filtered and then hydrolyzed to metatitanic acid. This acid was then roasted to form TiO₂ (87%–89% grade). Smelting of magnetic portion at 1480 °C yielded a product containing 93%–94% Fe, 4.5% C. Recovery of Iron was observed to be less due to the agglomeration of fine grains together, which decreased the efficiency [30]. The direct smelting of red mud produced a calcium aluminate slag. In such cases, the limestone content was carefully monitored to make a slag, which was soluble in sodium carbonate solution and could be recycled back into the Bayer process [31].

Kumar et al. investigated red mud from Nalco for two alternative namely the Elgai process showed in Fig. 4. This process was a patented process-to-process iron aluminous ores and used this as an addition to sintering process. In this process, testing the first alternative, red mud, charcoal and soda were pelletized in a rod mill, roasted at 700–925 °C and water leached. The parameters

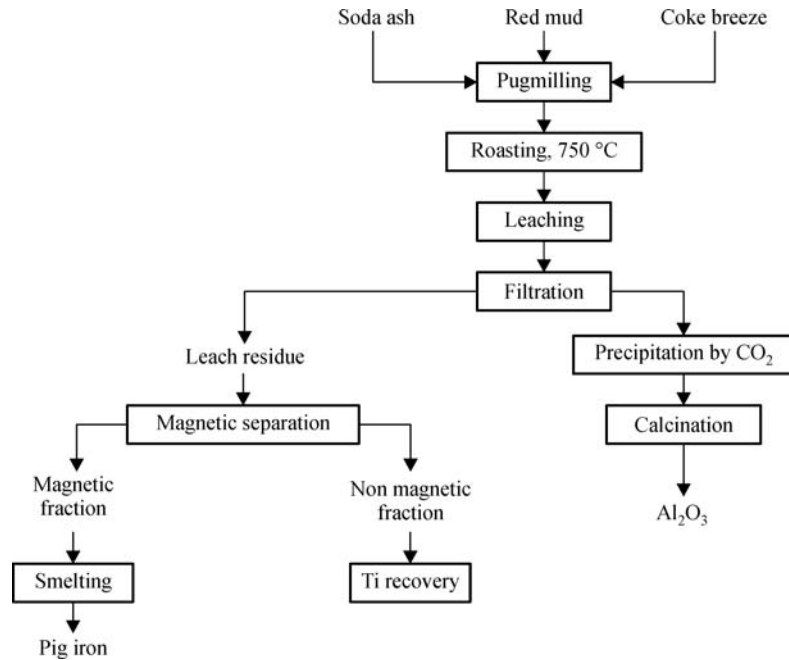


Fig. 4 Process for removal of iron from aluminous iron ores by Kumar et al. [32]

varied were amount of soda addition, milling time, roasting temperature and time, effect of water quenching of product, leaching temperature and time. The Davis tube experiments were conducted on the leach residue employing 2400 gauss and water flow rate 1 L/min. A clear separation was not obtained. Good results were obtained employing various amounts of red mud per ton of sinter produced. Similar values of tumbling index, reducibility, abrasion coefficient were observed [32].

There have been many attempts to find an economically feasible process to be practiced industrially. It was also told that addition of calcium, sodium or magnesium salt could improve the efficiency of carbon based direct reduction of iron in red mud. Jayasankar et al. has proposed that addition of 10%–12% dolomite would increase iron recovery to 71% [33]. A recovery of 94.5 wt-% Fe could be obtained by employing 6% Na_2SO_4 and 6% Na_2CO_3 in the mixture. This mixture was roasted to a temperature of 1050 °C for 60 min [34,35].

Iron nuggets were produced by direct reduction of carbon bearing pellets of red mud and coal. This work done by Guo et al. showed that the strongest influencing factor for metallization rate was temperature, reduction time, x_c/x_o in pellets and basicity. Iron nuggets with 96.25% Fe could be produced employing conditions of basicity 1, 1.6 $X_c:X_o$ ran at 1400 °C for 30 min. The pellets produced have a higher total iron than that present in pig iron with Si and Mn contents lower but have a higher concentration of S and P. The slag was essentially a fayalitic (Fe_2SiO_4) slag with some iron entrainment along with SiO_2 [36].

Red mud was treated with a 35 kW DC extended plasma reactor to carry out smelting. The smelting products

included pig iron and slag. Proper mixing of raw materials red-mud, graphite (99% fixed carbon) and fluxes (10% dolomite and lime) were achieved. The effect of various processing parameters like reductant, fluxes and smelting time were optimized. An optimal recovery of 71% was achieved in 15 min of smelting. This process was extended to direct reduction of red mud fines to achieve utilization of red mud [33].

Further to the above, Samouhos et al. have developed a microwave powered reduction roasting process of red mud using lignite coal. Wet magnetic separation was followed; the magnetic portion would constitute a raw material for the sponge iron production. Microwave associated heating is a clean source of heating, generates heat instantaneously in the sample by the vibration of the dielectric compounds and is not associated with any temperature drop on the periphery or in the solution. As a result, the experiments conducted reduced the reduction time by over 40% compared to conventional heating methods. A magnetic concentrate of 35.1% Fe, 69.3% metallization was obtained after wet magnetic separation (feed: 10 wt-% solids, intensity 0.3 A). All the radionuclides are considerably lower than the clearance levels ($1000 \text{ Bq}\cdot\text{Kg}^{-1}$) [37].

Li et al. carried out stepwise extraction of valuable components like Fe_2O_3 , Al_2O_3 , and SiO_2 . They employed magnetic separation and sulfuric acid leaching from reduced red mud. During the reductive roasting of red mud, sodium played an important role in reducing the dispersity of iron hence increased the efficiency of magnetic separation. They increase the growth of the metallic iron grains. The addition of sodium salts increased

the efficiency of acid leaching of Al, Na, and Si. Hercynite and quartz are converted to metallic iron and sodium silicate in the presence of sodium salts, which was favorable to the subsequent enrichment of TiO_2 . A magnetic concentrate containing 90.2% Fe at 95% recovery was obtained from a red mud containing 48.2% Fe. Red mud was reduced at 1050 °C for 60 min in the presence of 6% Na_2CO_3 and 6% Na_2SO_4 . In the enrichment of TiO_2 by sulfuric acid leaching, 94.7% Fe, 98.6% Al and 95.9% Si were extracted leaving behind a material having 37.8% TiO_2 . The ripening and neutralization process results in silica gel containing 64.2% SiO_2 and an $\text{Al}(\text{OH})_3$ precipitate containing 35.3% Al_2O_3 . The process is depicted in Fig. 5 [38].

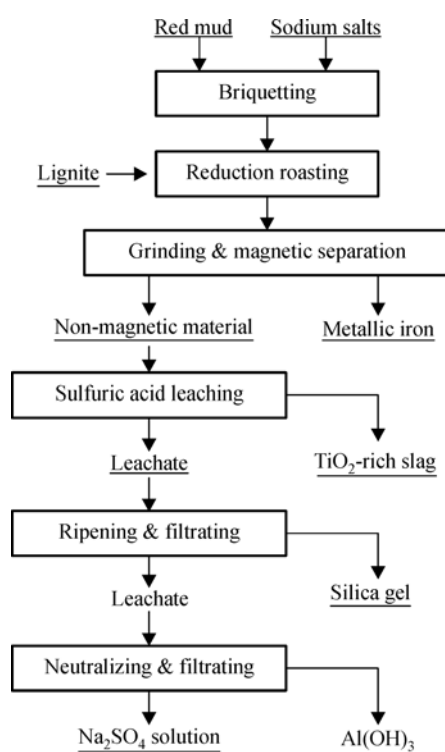


Fig. 5 Stepwise reduction, roasting and magnetic separation process for red-mud by Li et al. [38]

Teplov et al. studied the reduction of red mud in the presence of hydrogen atmosphere on the red mud obtained from the Alyum plant. It was observed that a complete conversion of hematite present to magnetite was achieved. This reduction was carried out at a temperature of 300 °C and a heating rate of 500 K/h. The complete reduction of magnetite to metallic iron takes place at 420 °C. The magnetite formation was associated with a 0.5 mm thick RM at 275 °C and this layer ends at 350 °C. The rate of reduction to iron decreased two fold if the reduction was conducted at a higher temperature range (above 900 °C). When the hydrogen gas was replaced by natural gas to heat the same 5 mm red-mud layer at 400 K/h resulted in a delay

of magnetization and the reduction temperature increased from 350 to 375 °C. In a same temperature range, the reduction of the pellets in natural gas is 30–90 times faster than that would occur in hydrogen gas. In fact, as the size of the pellets decrease, time taken for iron oxides to transform into magnetite decreased [39].

Staley et al. explored the possibility to extract iron from red mud by carbothermic reduction, followed by either magnetic separation, electrowinning or electro refining [7]. The carbothermic reduction aimed at converting red mud to metallic iron either fully or partially. The reduction occurred at temperatures 975 to 1050 °C for 2 h using petroleum coke or graphite as reductants. Complete reduction at 1050 °C using petroleum coke maintaining a layer of lime to maintain a reducing environment yielded metallic iron containing 92 wt-% Fe. Partial reduction experiments were carried out to produce magnetite using pine, poplar, petroleum coke and graphite as reducing agents. 92 wt-% of iron phase was formed in the reduced product using pine as carbon source and employing a lime layer.

Davis tube wet-magnetic separation was conducted on fully and partially reduced samples. Scanning electron microscopy (SEM)/energy dispersive X-Ray spectroscopy analysis showed that Fe, Ca and Ti seemed to have intimate affinity with each other. Grinding of reduced material to separate magnetic and non-magnetic materials, sieving and subsequent magnetic separation of several particle size fractions resulted in a decrease in separation effectiveness. It was suggested that the perovskite (CaTiO_3), formed during the Bayer process, occurred in red mud and its content in the product, obtained after the reduction experiments, increased. Perovskite has the ability to substitute for iron in the lattice. Consequently, calcium, iron and titanium are carried with iron to the magnetic section.

Electrowinning was preceded by digestion of reduced red mud in hydrochloric acid and sulfuric acid with varying concentrations. The digestion of reduced red mud was effective in dissolving the iron and producing ferrous ions. The electrolyte at 30 °C consisted of digested material with a pH of 5 ± 0.5 adjusted using NH_4OH . Adjustment of pH was necessary to prevent any hydrogen evolution at low pH's and precipitation of iron bearing minerals [7]. Continuous flow of digested material was assured because of the fear of anticipating low iron concentration in the tank due to the electro deposition of Fe. A chromium coated on high stainless steel is used as a cathode and a platinum-coated tungsten wire is used as an anode. Current density of $10 \text{ mA} \cdot \text{cm}^{-2}$ is maintained using an applied voltage of 12 V. The duration of current application was 2 h. Purity of the iron product obtained was 90 wt-% at a current efficiency of 30%.

Caupain has conducted a low temperature gas carbidization of iron constituents of red mud. Theoretically, it was proposed that the magnetic iron carbides be separable from

the bulk material by magnetic separation. Carbide formation experiments were carried out for a range of processing temperatures and times, 560 to 650 °C and 15 to 45 min. The gas composition was maintained at $\text{CO}:\text{H}_2 = 3:1$. It is observed that soot formation is not observed. It is accounted to the fact that some sulfur bearing compounds in red mud restricted the nucleation and growth of carbon to soot. Some SEM micrographs depicted the presence of decrepitated particles of red mud in the reduced sample, which indicated the presence of many fine particles, which are agglomerated together in the original sample of red mud. The composition of the reduced material is 7–60 wt-% cementite (Fe_3C), 0–79% Hägg carbide (Fe_5C_2), 0–11 wt-% Fe_2O_3 , 0–8 wt-% Fe_3O_4 , 0–12 wt-% Fe and 0–12 wt-% of an unidentified (nanosize) non-magnetic iron oxide phase [7,40].

The reduction products contained more Hägg carbide than that of cementite. It has been observed that in the initial period of quenching the reduced sample, CO formed appeared to contribute to the formation of the condensed carbon phase that subsequently reacted with the Hägg

carbide to form cementite. It was observed that the magnetic separation on the samples was unsuccessful. Even the comminution of the reduced sample did not improve the outcome. A conclusion was made stating that the carbide particles are associated with other constituents/carbides¹⁾.

Piga et al. [30, 41] performed beneficiation of red mud. Red mud was leached in hydrochloric acid. Titanium is soluble in sulfuric acid but not in Hydrochloric acid. This process increased the recovery of TiO_2 content in the residue from 31% to 58%. The solids were then leached with sulfuric acid at 270 °C, followed by hydrolysis and roasting. The TiO_2 content obtained was 96%. The product can be used directly as TiO_2 pigment or chlorinated to form TiCl_4 . The solids are treated in an alternative route, in which mill scale, aluminum powder, compounds such as CaO, CaF_2 and NaNO_3 are added to the residue, which is then subjected to aluminothermy as shown in Fig. 6. This process yields a ferrotitanium alloy. The solution containing FeCl_3 and AlCl_3 are roasted to produce the respective oxides.

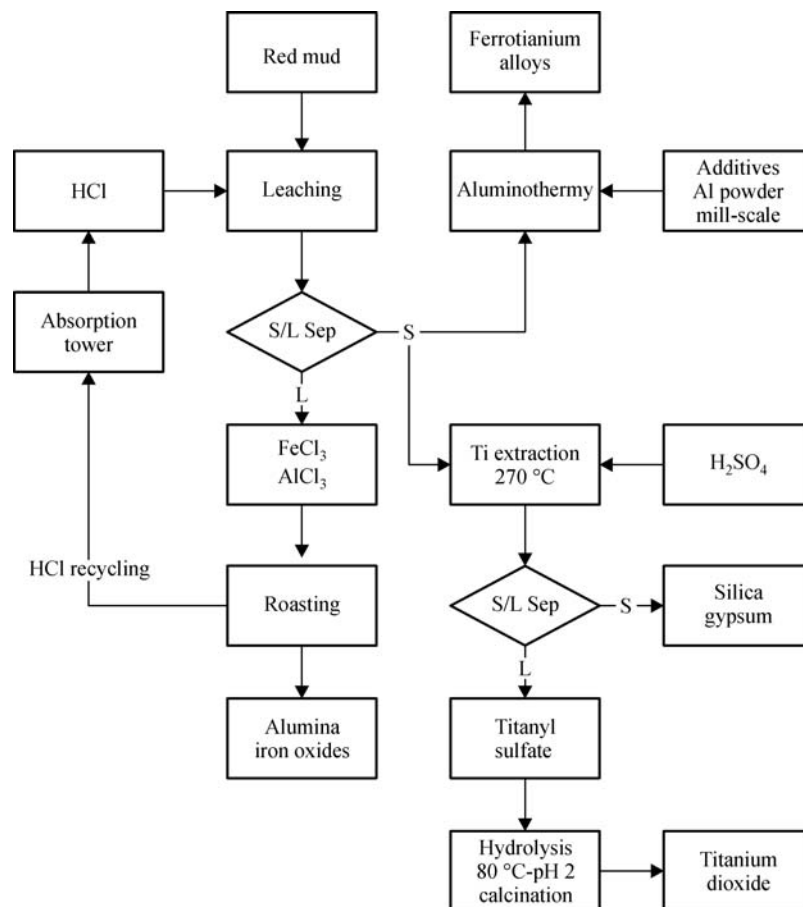


Fig. 6 Process flowsheet for metal recovery from red mud adopted by Piga et al. [30]

1) Mishra B, Gostu S. Opportunities for high volume commercial products conversion from bauxite residue. Bauxite Residue Valorisation and Best Practices Conference, 2015. <http://conference2015.redmud.org/wp-content/uploads/2015/10/Brajendra-MISHRA-secure.pdf>

In addition to recovery of metals, it was shown that it was possible to obtain sodium aluminum fluoride compounds like cryolite from red mud. The cryolite can be used as a flux in the Hall Heroult process and can be used in the glass making. Leaching of red mud with HCl yields a residue of titanium and some iron and aluminum containing compounds. To the filtrate, some Hydrofluoric acid is added, which contains sodium, aluminum and silicon, which causes the precipitation of silicic acid. The liquid is then evaporated simultaneously adding sodium, which gives rise to the formation of cryolite. The hydrofluoro-silicic acid, hydrochloric acid mixture together with silicic acid are added to the residue from the first stage causing dissolution of iron and aluminum as chlorides to precipitate a titanium rich solid [42].

Acid leaching of recovering iron also was investigated, a high recovery of iron 97.46% Fe can be obtained at calcination 600 °C and subsequent leaching in 6 mol·L⁻¹ H₂SO₄ and aluminum was recovered around 64.4%. Flow sheet of the process is show in Fig. 7. Oxalic acid was also used to dissolve iron. Using photocatalysis ferric oxalate was transformed into ferrous oxalate. Al³⁺, Na⁺ were recovered using cationic membranes using H⁺ as stripping phase [42].

Red mud was mixed with H₂SO₄ (20–30 g/L) at 30–110 °C with solid:liquid ratio of 3–6 and mixed at 200–500 r/min for 20–80 min. The mixture was then roasted (100–800 °C, 20–80 min) and washed (3–8 times). Recovery of iron was conducted using precipitation or resin adsorption from the leaching solution. The Na₂O content in the residue was significantly reduced while iron was recovered. By-product Na₂SO₄ was also obtained [43].

Bioleaching technology was also adopted considering the ecological effects and low energy metal consumption [44–46]. There are 2 kinds of bacteria one, which is formed by forming complexes with organic acids and iron ions. The other is by facilitating the transformation of Fe³⁺ to Fe²⁺ as ferrous has a high solubility compared to ferric forms. The high pH of red mud restricts this process of extracting iron through this route.

In a work done by University of Missouri Rolla, three red muds were treated using three carbon-based reductants: charcoal, coal, sawdust from local mill, and the residue from the sugarcane processing. Dried red muds and reductants were mixed in weight ratios 3:2 and 6:1. A 5 g mixture was placed in an alumina boat under Argon atmosphere. The duration of experiments were varied from 0.5 to 12 h. Firstly, red muds from three different sources

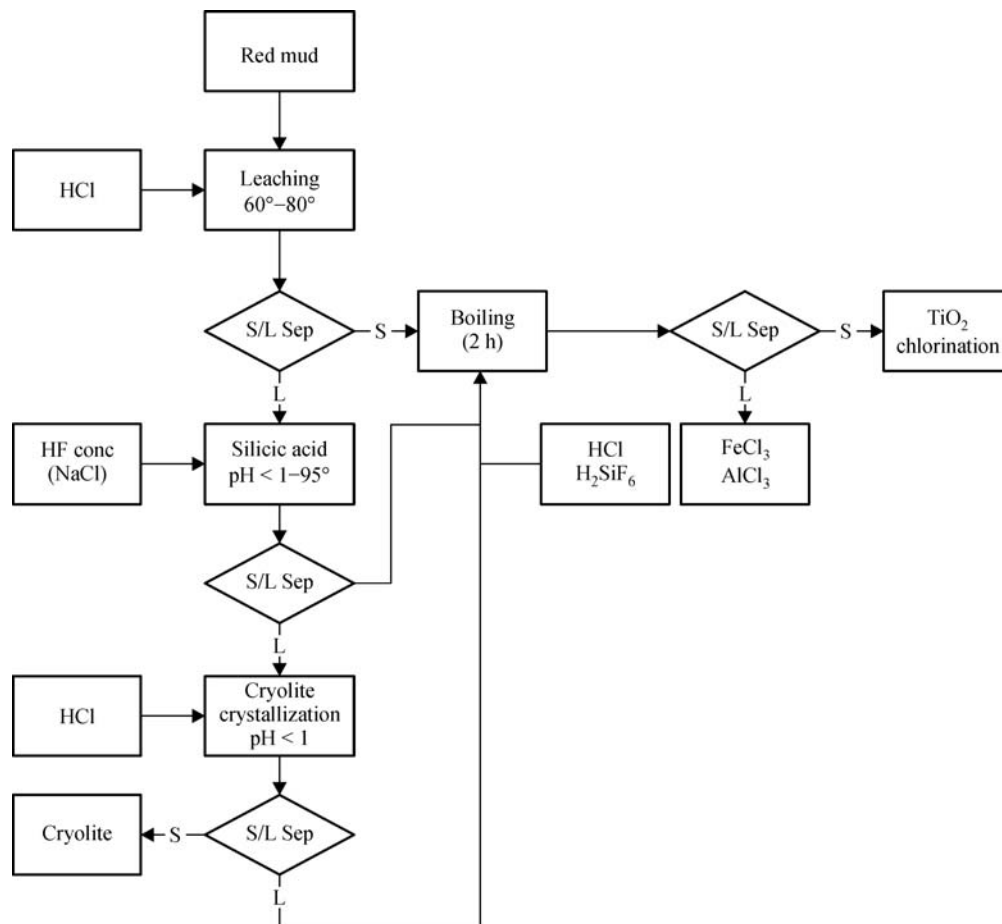
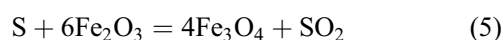


Fig. 7 Cryolite production from red-mud by HCl leaching [42]

were treated with the same reductant. It was reported that due to a lower iron content in the “Alcoa red mud”, higher reduction was achieved. It was also observed that in the small samples, the conversion was so rapid that the reaction comes to an immediate halt once it is done. This suggests that rate-controlling step of the reaction was the chemical transformation reaction. Then the feasibility of a reductant was assessed by conducting the reactions by changing the temperature at a constant time of 4 h and red mud/reductant ratio is kept constant at 3:1. It is observed that temperatures as low as 500 °C are enough to ensure complete reduction. The authors suggested the use of a partial reduction strategy to prevent the formation of nonmagnetic wustite. From this study it has been determined that baggase and saw dust are more effective than coal and charcoal in terms of reduction efficiency. It is seen that at reductions conducted at 500 °C, Ti forms perovskite, which reduces the magnetic separation efficiency. Hence, the need for a low temperature, 400 °C reduction is chosen. A large scale reduction is conducted utilizing these parameters, but It was observed that a higher temperature is needed for the same amount of reduction, this could be attributed to the fact that higher gas pressures are generated in the alumina tube due to an increase in the amount of starting red mud. Davis tube wet-magnetic separations were conducted at a field intensity of 2.73 kGauss, water flows varied between 60 to 500 mL/min. It is observed on comparison to the reduction of “Kaiser red mud” sample using charcoal at 600 °C, the nonmagnetic portion yielded low Iron but the recovery of magnetite in the magnetic portion was smaller. It was observed that the magnetite recovery of a sample reduced at 700 °C was not affected. It was demonstrated that the results from the reduction with these three reductants also were similar. It was observed that magnetic separation performed on the “Alcoa red mud” reduced at 600 °C, exhibited smaller [47].

Anaerobic coroasting of pyrite (FeS_2) with red mud was employed by Liu et al. Fe_2O_3 transforms to Fe_3O_4 (Eqs. (4,5)). The parameters varied were mole ratio of pyrite to hematite and the roasting temperature. The sintered red mud out of the desulfurization process was used for the experiment heated under the N_2 atmosphere. Pyrite thermally decomposed into iron monosulfide, pyrohotite and elemental sulfur.



The decomposition of pyrite to elemental sulfur takes place at about 525 °C. At 600 °C, 30 min of roasting maintaining 1:16 ratio of pyrite to hematite yielded complete magnetite. 30 g of red mud (13.24% Fe_2O_3) mixed with 0.74 g of pyrite was roasted at 600 °C under N_2 atmosphere. Magnetic separation was employed to yield a

nonmagnetic product having 2% Fe that is certified for refractory material manufacture [48].

4.2 Extraction of high value low volume materials from red-mud

If a complete rare earth metals extraction from red-mud (~1–1.5 wt-%) is achieved, it could achieve a major chunk of the total revenue, which could be recovered by extracting all the recoverable components of red-mud [49]. The typical rare earth metal extracted from red-mud was scandium owing to its abundance and sale price in most red-mud. Scandium composition in red-mud depends on the source bauxite ore used in the Bayer’s process. It varies from 390 ppm in Jamaican red-mud to as low as 130 ppm in Greek red-mud [50]. Due to the presence of high levels of impurities in red-mud, it is difficult to extract scandium directly from red-mud. Iron and scandium share some common chemical properties, so in attempting scandium recovery directly, iron interferes in its extraction.

Borra et al. have devised a strategy to leach scandium from Greek red-mud using different acids. They varied reactant concentrations, pulp densities, Kinetics of leaching to study the extraction of scandium. Scandium extraction in hydrochloric acid was found to be about 60%, but it was accompanied with the extraction of iron, aluminum [51].

A SX based process for scandium extraction from Australian red-mud was devised by Wang and Cheng. Red mud was leached with sulfuric acid using optimized devised leaching parameters for the hydrothermal treatment. Multiple stages of SX (with Shellsol-50, D2EPHA/TBP) was attempted by the authors to extract Sc, Zr and Ti [52]. A partial selectivity between iron and scandium was achieved in this process but a large fraction of residue (99%) was left out in the process untreated, which deems the process unsustainable. Yatsenko and Pyagai have devised an alkaline leaching strategy to hydrometallurgically treat scandium from the bauxite residue [53]. Sodium carbonate was used as a leachant to create a stable anionic complex compound with stable carbonate compounds. This complex could dissolve only $0.43 \text{ g} \cdot \text{L}^{-1} \text{ Sc}_2\text{O}_3$ in a solution containing $100 \text{ g} \cdot \text{L}^{-1}$ carbonate solution.

5 Conclusions

Variegated approaches to ameliorate red-mud in various branches of research in the past 30 years have failed to put a step forward into commercial utilization. The complex nature (elemental structure, basicity, fine particle size) is a critical aspect in the path of its commercialization. Added to this, the difference in chemical compositions of red mud is a function of the bauxite ore used in the Bayer’s process. This variation would prevent a universally acceptable process for red-mud valorization. Red mud manufactured

construction materials like cement, soil ameliorant applications, face the problem of Na, Cr, As leaching into the environment. The geo-polymers manufactured from red-mud are associated with low flexural strength. Weathering rate of sodium is a problem in using red-mud in brick production industries.

High costs are associated with transportation, drying the residue, dewatering the residue from red-mud ponds, maintenance of red-mud storage facilities, process economics. All these overhead costs have to be considered in devising an economic flowsheet for a valorization process. Metal recovery from red-mud would be a combination of mainly a pyrometallurgical and hydrometallurgical process. The pyrometallurgical processes are associated with a consumption of tremendous amounts of heat. Red mud cannot be used directly as an iron ore because of the presence of high alumina, the complex mineralogy associated with red-mud hinders its use to extract high-grade magnetic materials (magnetite, metallic iron, Hagg carbide), and the extraction of scandium from red-mud is associated with co-precipitation of iron.

A sustainably economic, efficient, value added valorization process for red-mud must be associated with the extraction of the major components from red-mud Fe, Al, Na, Ti, Ca, Sc and rare earth elements. Multiple prospective markets must be identified for the value additions achieved.

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