EXTRACTION OF ALUMINA FROM RED MUD BY DIVALENT ALKALINE EARTH METAL SODA ASH SINTER PROCESS

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Abstract

A novel method for extraction of alumina from red mud using a divalent alkaline earth metal oxide soda ash sinter process followed by thermo chemical leaching in suitable caustic concentration at 105°C for different times is presented in this paper. During sintering Sodalite/Cancrinite, Gibbsite and Boehmite in red mud is converted to sodium aluminate as well as stable phases of divalent alkaline earth metal ferrite, titanate and silicate. In the above novel economic process an extraction of alumina from red mud of 97.64%, 98.7% and 99.5% was achieved using CaO, MgO and BaO respectively. The morphological study of sinter products and leached products was studied using SEM and XRD.

1. Introduction

Red mud is a by-product in the manufacture of alumina that contains mainly iron oxide (54-65%) with significant amounts of silica, alumina, calcium oxide and titania, dispersed in a highly alkaline and caustic liquor [1,2]. The treatment and disposal of bauxite residue is a major operation and may account for 30-50% of operations in an alumina refinery [1]. The red mud has been accumulating at a rate of 70 million tonnes annually throughout the world [3]. For a given aluminum production rate, the quantity of red mud generated during the alumina extraction process varies significantly depending on the original properties of the bauxite and the operating conditions of the Bayer process, and in particular, the process temperature [4]. Depending on the factors, between 1 and 2.5 tonnes of bauxite residue is generated per tonne of alumina produced [1]. The disposal of the red mud is associated with pollution problems. Disposal of any solid waste is associated with space or real estate near the industry, and the cost of disposal and pollution, which are now crucial factors. Obviously, these three factors are also associated with the red mud disposal. As the red mud contains a large amount of valuable chemicals, there is a need for developing a technology for the recovery of at least some of the important chemicals [3].

Sulfided red mud is active as a hydrogenation catalyst, due to its iron sulfide content, and has been used for the hydrogenation of organic compounds, and the liquefaction of coal and biomass [2].

In the literature, a lot of different articles that explain the properties and the investigation of the red mud tailings can be found. One such reference investigated the red mud under caustic concentrations of 10-30% to determine the extraction of soda and alumina as a function of caustic ratio, CaO: SiO_2 ratio, water content, temperature and reaction time. Hydrothermal treatment of the red mud with lime at high temperatures of 300°C was found to be an effective method for the recovery of

soda and alumina. Recoveries of 95% Na_2O and 70% Al_2O_3 were obtained [5]. Alyanak et al. [6] studied on the recovery of aluminium from the red mud of the Seydisehir Aluminium Company Limited. They prepared a mixture composed of 26.6 wt% red mud, 37.2 wt% $CaCO_3$ and 36.2 wt% Na_2CO_3. The mixture was sintered at 900°C for 2 hours then leached in water. Increasing the sintering temperature as high as 1150°C decreased the alumina extraction efficiency.

In another article, a gallium recovery method was applied to bauxite ores, during alumina production by the Bayer process. Gallium movement and possible locations of gallium recovery in the Etibank-Sedisehir Aluminium Plant and some results for gallium concentrate production by precipitation were presented [7]. S. Uday [8] had worked on the recovery of iron, titanium and aluminium values from red mud by calcining it, preferably between 800°C to 1350°C and smelting the material with a carbonaceous reducing agent in an electric furnace, thus obtaining molten iron and a slag containing substantially all the titania, alumina and silica.

The red mud has also been investigated for the making of artistic glazes in ceramic. For this purpose, no additives and some fluxing materials such as read lead, ulexide, cryolite, borax, soda ash, etc. were added to the red mud in different proportions. Finally, the optimum conditions forming novel glazes were determined [9].

The red mud was also investigated to find out whether it can be used as a construction material or not. For this purpose, the red mud, a waste material of Aluminum Plant in Seydisehir, was mixed with clay, which was taken from the same region. This mixture was pressed and sintered at between 1000 and 1150°C. It was stated that construction materials produced from the mixture had higher compressive strength and lower water absorption ratio compared with the related standards [10]. In another study, the structure of the red mud deposits of Seydisehir, Turkey was investigated by simple statistical analysis. It was shown that the ratio of the iron oxide % (D_1) in the red mud to the mean iron oxide % (D_2) was found to fit a linear function represented by plotting D_1/D_2 versus K_1/K_2 in a double logarithmic plot. The relation in the form of a power function was $D_1 = 1.0092D_2 K_1^{0.021} K_2^{-0.022}$ [11].

The thermal behaviour of the red mud was investigated in the literature. An extensive characterization was performed by thermal and X-ray diffraction analyses. Dried red mud was substantially inert up to 900°C, the loss of H_2O from aluminium hydroxides and of CO_2 from silico-alumino-carbonates being the only detectable effects [12].

Several methods for extracting alumina as well as for recovery of other metal values from alumina-bearing materials

have been studied [13]. The United States Bureau of Mines has been evaluating possible alternatives to the use of bauxite for several years. These include acid leaching processes and sintering processes applied to clays and anorthosites. Fly ash from burning regular coals has also been investigated extensively as a potential source for alumina. The processes studied for the extraction of alumina from fly ash include variations of existing sintering and leaching processes.

There are now a few millions of tonnes red mud stored in Red Mud ponds in India. These wastes have not been investigated yet in any industrial process or used for material production as an additive. The aim of this study was to investigate these muds for maximum alumina extraction by a divalent alkaline earth metal oxide soda ash sinter process followed by thermo-chemical leaching.

2. Materials and Methods

Red mud and crushed Lime (CaO) used in experimental studies were supplied from, Damanjodi, Dist.-Koraput. The MgO, BaO and Na₂CO₃ used in experimental studies were supplied from Qualigens fine Chemicals, Bombay. The major elements were analysed by classical methods as well as X-Ray Fluorescence Spectroscopy (Model PW 2400). The minor elements were analysed by AAS. The chemical composition of the red mud is shown in Table 1. X-Ray Diffraction (XRD) analysis of sintered red mud and leached red mud were carried out with a Diffractometer using CuKα (Kα=1.54186 Å) Rigaku Dmax 2200, Japan. The morphological behaviours of sinter red mud and leached red mud were studied by SEM (Model:430, Electron Microscopy, United Kingdom).

An amount of 100 g of red mud, 10 g of divalent alkaline earth metal oxide (i.e. CaO, MgO and BaO) and 15 g / 20 g/25 g of Na₂CO₃ was mixed thoroughly and pulverised in a pulveriser, then the above mixture was sintered at temperatures of 900, 1000, 1100°C for 1 to 4 hrs in a ceramic crucible in a high temperature muffle furnace. An amount of 100 g of red mud, 15 g of divalent alkaline earth metal oxide (i.e. CaO, MgO and BaO) and 10 g/20 g /25 g of Na₂CO₃ was mixed thoroughly and pulverised in a pulveriser, then the above mixture was sintered at temperatures of 900, 1000, 1100°C for 1 to 4 hours in a ceramic crucible in a high temperature Muffle furnace. An amount of 100 g of red mud, 20 g of divalent alkaline earth metal oxide (i.e. CaO, MgO and BaO) and 10 g/20 g/25 g of Na₂CO₃ was mixed thoroughly and pulverised in a pulveriser, then the above mixture was sintered at temperatures of 900, 1000, 1100°C for 1 to 4 hrs in a ceramic crucible in high temperature muffle furnace. After sintering the sinter products were leached with 80 gpl caustic at 105°C for 1 hr in an autoclave of 2.5-litre capacity. The leached red mud was filtered after the leaching. The amount of Al_2O_3 in the leach solutions was determined by using a titrimetric method.
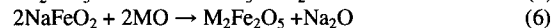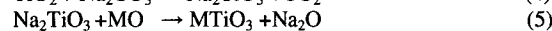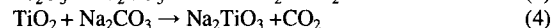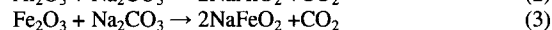
Table 1. Chemical Analysis of Red Mud

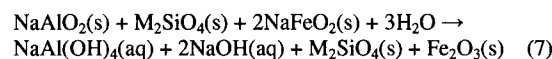| Chemical Constituents	Al₂O₃	Fe₂O₃	TiO₂	SiO₂	Na₂O	CaO	V₂O₅	MgO	MnO	LOI at 1000°C
(%)	16-18	51-57	3-5	8-12	4-6	0.03-2.30	0.14-0.21	0.13-0.18	0.157-0.250	11-13

3. Results and Discussion

Methods of aluminium and sodium recovery from red mud have been around for many years due to the requirement of some refineries to process very high silica bauxites. The most common method to process red mud is the "divalent alkaline earth metal oxide Na₂CO₃ sinter process". The process involves adding divalent alkaline earth metal oxide (i.e. CaO, MgO and BaO) and Na₂CO₃ to the red mud and sintering at temperatures of 900-1100°C [14]. The silica in the red mud reacts with divalent alkaline earth metal oxide to form the relatively inert divalent alkaline earth metal silicate as shown in equation:1. The sodium, which is in the red mud via soda ash or sodium carbonate, reacts with the alumina and forms the soluble sodium aluminate as given in equation:2 [15]. The Fe₂O₃ contents of red mud reacts with sodium carbonate to form sodium ferrite as given in equation: 3.The TiO₂ which is in the red mud reacts with sodium carbonate forms sodium titanate as given in equation: 4. The sodium titanate reacts with divalent alkaline earth metal oxide forms divalent alkaline earth metal titanate and free Na₂O as given in equation: 5. The sodium ferrite reacts with divalent alkaline earth metal oxide forms divalent alkaline earth metal ferrite and free Na₂O as given in equation: 6.

$$SiO_2 + MO \rightarrow M_2SiO_4 + 2H_2O \qquad (1)$$
$$Al_2O_3 + Na_2CO_3 \rightarrow 2NaAlO_2 + CO_2 \qquad (2)$$
$$Fe_2O_3 + Na_2CO_3 \rightarrow 2NaFeO_2 + CO_2 \qquad (3)$$
$$TiO_2 + Na_2CO_3 \rightarrow Na_2TiO_3 + CO_2 \qquad (4)$$
$$Na_2TiO_3 + MO \rightarrow MTiO_3 + Na_2O \qquad (5)$$
$$2NaFeO_2 + 2MO \rightarrow M_2Fe_2O_5 + Na_2O \qquad (6)$$
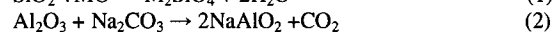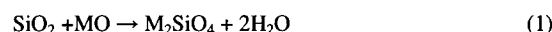
The product is then leached in an alkaline solution or water as given in equation: 7 and the sodium aluminate solution is directed to the precipitation stage of the Bayer process.

$$NaAlO_2(s) + M_2SiO_4(s) + 2NaFeO_2(s) + 3H_2O \rightarrow$$
$$NaAl(OH)_4(aq) + 2NaOH(aq) + M_2SiO_4(s) + Fe_2O_3(s) \qquad (7)$$

To produce high aluminium and sodium extractions the decomposition of divalent alkaline earth metal silicates must be as low as possible. In practice the amount of silica extracted in the leaching step is too high, as the M₂SiO₄ is not sufficiently stable under the leaching conditions [16] and silica extractions of around 15–20% [17] occur during leaching. This ties up sodium and aluminium with the formation of Mg₃Al₂(OH)₁₂, Ca₃Al₂(OH)₁₂ or Ba₃Al₂(OH)₁₂, Hydro garnet and DSP. The opportunity exists for a process to treat red mud to separate aluminium and sodium from silica, producing an adequately stable insoluble phase for leaching and therefore yielding greater extractions.

An alternative method of divalent alkaline earth metal oxide Na₂CO₃ sinter is presented in this paper to produce a M : Si ratio of one in the sinter product[16]. The potential benefits are to halve raw material costs and to give a more stable insoluble phase. Also the extra sodium needed could be provided by spent liquor creating the potential for organics removal through liquor burning in the sinter step. The removal of organic impurities in sintering followed by leaching at 105°C is due to formation of TCA or Layered Double Hydroxide [Hydrotalcite: Mg₆Al₂(OH)₁₆CO₃.4H₂O].

However, thermodynamic calculations and laboratory tests show that MSiO₃ does not form under sinter conditions [18]. From the present experiments it was thought that the following divalent alkaline earth metal oxide sinter reaction was feasible due to the presence of sodalite in the red mud formed by the reaction of kaolin and sodium aluminate liquor.

$$Na_2O.Al_2O_3.2SiO_2 (Sodalite) + 2 MO + 4Na_2CO_3 \rightarrow$$
$$2Na_2MSiO_4 + 2NaAlO_2 + 4CO_2 + 2H_2O \qquad (8)$$
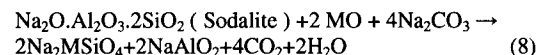
A similar lime sinter process is referred to in a paper discussing the integration of coal combustion with lime sintering [19]. Reaction (8) shows that soluble sodium aluminate and sodium alkaline earth metal silicates are produced. The sodium associated with sodium alkaline earth metal silicate must also be recovered in the leaching step. The objective of this project was to investigate the use of a M : Si ratio of one, in a divalent alkaline earth metal oxide, Na_2CO_3 sinter process for use as a red mud treatment for the recovery of aluminium and sodium and to compare this with the current lime soda ash sinter system of M:Si = 2.

The extraction efficiency of divalent alkaline earth metal oxide soda ash sinter process is shown in Table 2.

Table 2. Extraction Efficiency of divalent alkaline earth metal oxide soda ash sinter process at different condition .

Sl. no.	Condition RM+MO (CaO, MgO, BaO)+ Na_2CO_3	Temperature in ° C	Time in hours	Extraction Efficiency (%)
1	1:0.10:0.20	900	1	61.79
2	1:0.15:0.25	1000	3	66.66
3	1:0.20:0.25	1100	4	97.64
4	1:0.10:0.25	900	4	98.70
5	1:0.15:0.20	1000	4	97.22
6	1:0.20:0.25	1100	3	96.50
7	1:0.10:0.25	900	1	99.14
8	1:0.15:0.25	1000	4	99.50
9	1:0.20:0.25	1100	1	95.57

*Sl.No.1 to 3: RM + CaO + Na_2CO_3 sinter process, Sl.No.4 to 6: RM + MgO + Na_2CO_3 sinter process, and Sl.No.7 to 9: RM + BaO + Na_2CO_3 sinter process.

In the synthetic MgO Na_2CO_3 sinter stage, calcination at 900°C for 4 hrs with red mud, MgO and Na_2CO_3 in a ratio of 1:0.1:0.25 was found to produce the most stable Sodium Magnesium Silicates, Di-Magnesium Silicates, Magnesium Ferrite (SEM Fig.6) and Magnesium Titanate (SEM Fig.7). This was confirmed by XRD (as shown in Fig .1 and Table 3) and maximized the extraction of alumina at 98.7%. The lowest extraction efficiency of 47.84% obtained in the MgO Na_2CO_3 sinter process with red mud MgO Na_2CO_3 ratio (1:0.15:0.10) sintered at 1000°C for 1 hr is due to the formation of insoluble magnesium aluminium silicate hydrate and sodium aluminium magnesium silico titanate [NaAlMgSiTiOx].

In the synthetic CaO Na_2CO_3 sinter stage, calcination at 1100°C for 4 hrs with red mud, CaO and Na_2CO_3 in a ratio of 1:0.2:0.25 was found to produce the most stable Sodium meta aluminate, Sodium Calcium Silicates (Fig.4.a) Di-Calcium Silicates (Fig.4.a), Calcium Titanate (Fig.4.b) and Calcium Ferrite (Fig.5.a and 5.b) confirmed by XRD (as shown in XRD Fig. 2 and Table 3). This procedure maximized the extraction of alumina at 97.64%. The lowest extraction efficiency of 38.35% obtained in the CaO Na_2CO_3 sinter process with red mud CaO Na_2CO_3 in a ratio (1:0.1:0.15) sintered at 900°C for 2 hrs is due to the formation of insoluble calcium aluminium silicate.
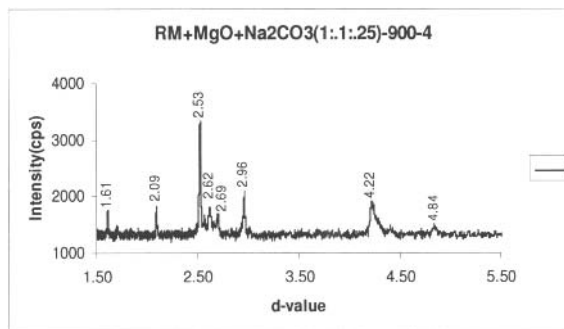

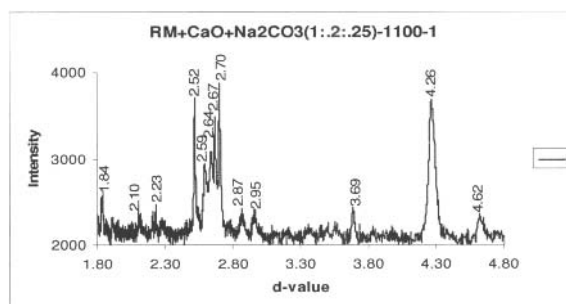**Figure 1: MgO Soda Ash Sinter Process with highest Extraction Efficiency 98.70% at 900°C.**

Figure 2: Lime Soda Ash Sinter Process with highest Efficiency 97.64% at 1100°C.

In the synthetic BaO Na_2CO_3 sinter stage, calcination at 1000°C for 4 hrs with red mud, BaO and Na_2CO_3 in a ratio of 1:0.15:0.25 was found to produce the most stable Sodium Barium Silicates, Di-Barium Silicates, Barium Ferrite (SEM Fig. 8) and Barium Titanate (SEM Fig.9) confirmed by XRD (as shown in Fig. 3 and Table 3) which maximized at 99.5% the extraction of aluminium and sodium. The lowest extraction efficiency of 32.56% obtained in the BaO Na_2CO_3 sinter process with red mud BaO Na_2CO_3 ratio (1:0.2:0.1) sintered at 1100°C for 3 hrs is due to the formation of insoluble Barium Aluminium Silicate Hydrate and Sodium Aluminium Silicate.
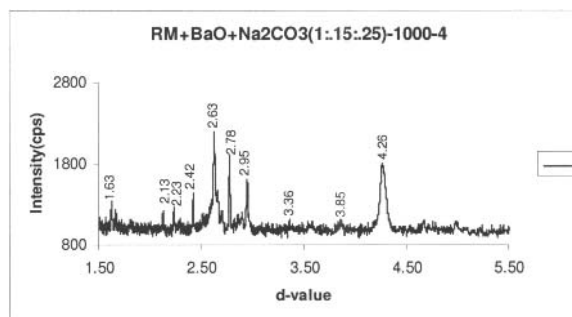

Figure 3: BaO Soda Ash Sinter Process with highest Extraction Efficiency 99.50% at 1000°C.

The existing CaO Na$_2$CO$_3$ sinter product ties some calcium up with iron due to the formation of Ca$_2$Fe$_2$O$_5$ (d value= 2.70,2.67,2.10,1.84, XRD Fig. 2 and Table 3), which have Whisker like structures as shown in the SEM images in Fig.5.a and 5.b. This meant that extra calcium was added to ensure the maximum amount of silica was reacted with the calcium and not with sodium or aluminium. At 1100°C, instead of sodium aluminate and sodium ferrite forming, Na$_2$AlFeO$_4$ formed in products with iron present.

For the synthetic new lime soda ash sinter process without iron it was found that a two stage caustic or water leach (105°C/60 min., 105°C/240 min.) was required to gain maximum Na and Al extractions due to the decrease in Al in solution over time as shown in Figure 10. It was found that in an MgO Na$_2$CO$_3$ sinter process sintering at 900°C for 4 hrs with red mud MgO Na$_2$CO$_3$ in a ratio of 1: 0.1:0.25 followed by leaching, formation of Sodium aluminate, MgTiO$_3$ and Mg$_2$SiO$_4$ maximise alumina extraction up to 98.7%. In a BaO Na$_2$CO$_3$ sinter process sintering at 1000°C for 4 hrs with a red mud BaO Na$_2$CO$_3$ ratio of 1:0.15:0.25 followed by leaching and formation of Sodium Aluminate, Ba$_2$Fe$_2$O$_5$ (SEM Fig.8) and BaTiO$_3$ (SEM Fig.9) give maximum alumina extraction efficiency of 99.5%.

This allowed the removal of the majority of the aluminium before the decomposition of the sodium divalent alkaline earth metal silicate and subsequent reaction to form DSP, Hydro garnet or Mg$_3$Al$_2$(OH)$_{12}$, Ca$_3$Al$_2$(OH)$_{12}$ or Ba$_3$Al$_2$(OH)$_{12}$. To ensure leaching conditions were the same for both systems the two-stage process was also employed for the existing lime sinter products. The results of extractions for the sinter products without iron are displayed in Figure 11.
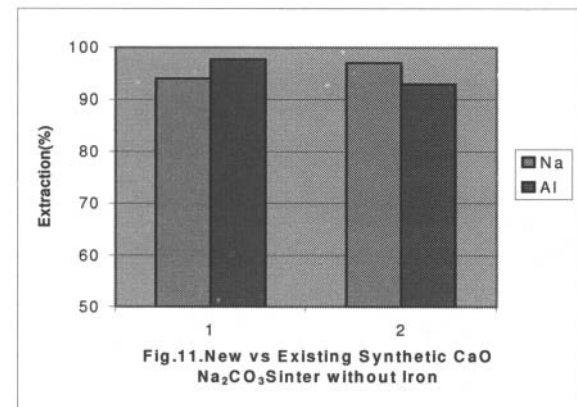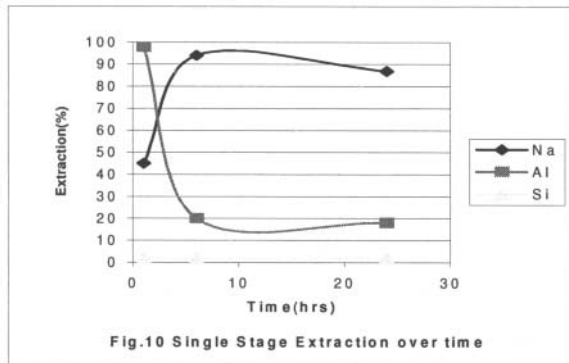
When Iron is introduced to the system there are effectively two soluble phases (NaAlO$_2$ + NaFeO$_2$) in which the ratio of soluble sodium to soluble aluminium increases. The results in Figures 12 and 13 show the benefit of the new alkaline earth metal Na$_2$CO$_3$ sinter process in terms of aluminium extraction. Much of the sodium and all of the aluminium is extracted from the existing alkaline earth metal sinter process in the first stage. The second stage merely completes the leaching of sodium. In the new red mud alkaline earth metal sinter extraction the resultant solids have a sodium content of 0.36 % compared to the 6.00 % sodium content of the red mud. The aluminium levels have been reduced from 17.8% to 0.42%. A more stable sodium alkaline earth metal silicate would increase these extractions and could be achieved with the optimisation of sinter temperature and leach conditions.
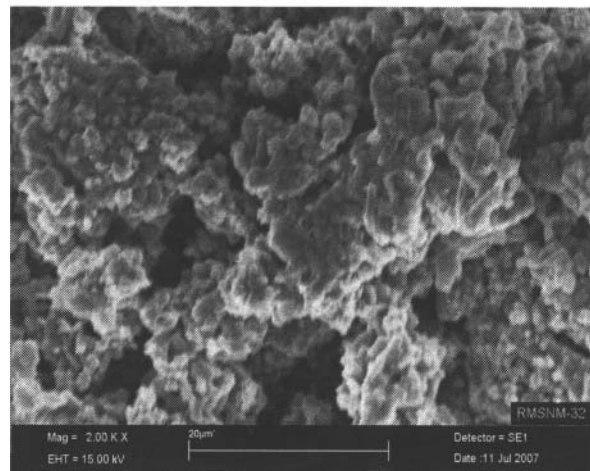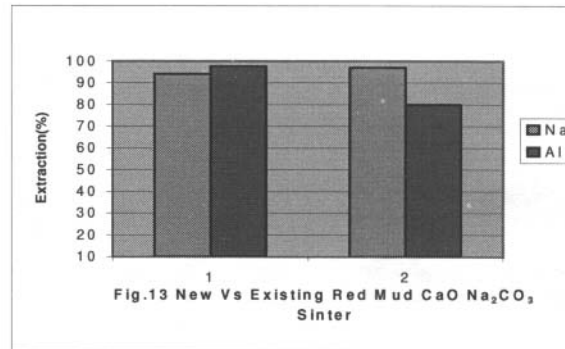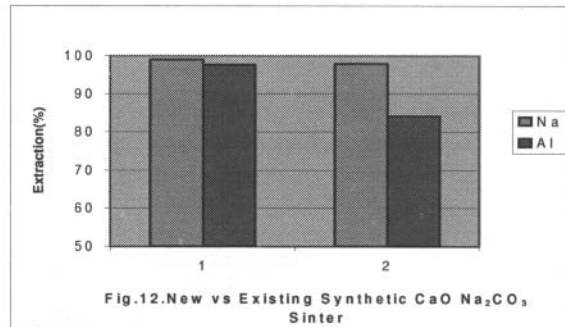

Fig.12.New vs Existing Synthetic CaO Na$_2$CO$_3$ Sinter

Fig.13 New Vs Existing Red Mud CaO Na$_2$CO$_3$ Sinter

Fig.10 Single Stage Extraction over time

Fig.11.New vs Existing Synthetic CaO Na$_2$CO$_3$ Sinter without Iron

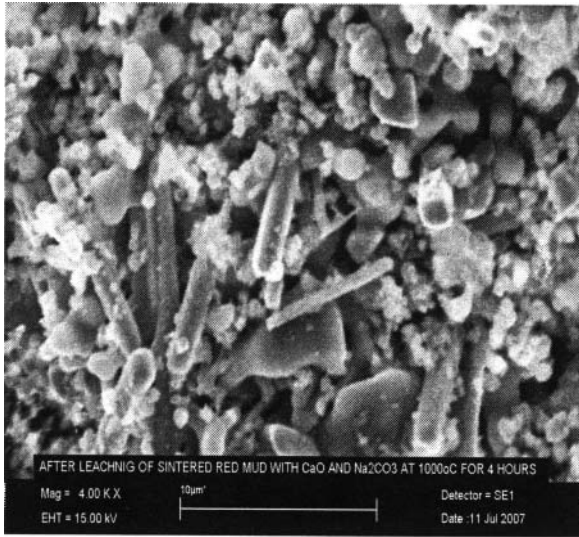Figure 4a: Formation of Sodium Calcium Silicate and Di-Calcium Silicate (2.00 KX)

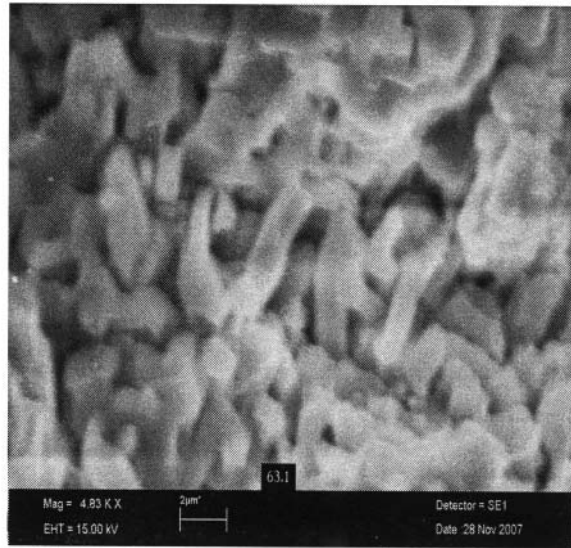**Figure 4b: Formation Calcium Titanate (4KX)**

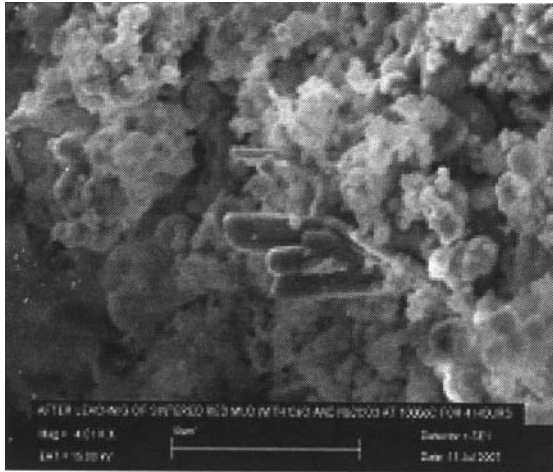

Figure 6: Formation of Magnesium Ferrite (4.93KX)

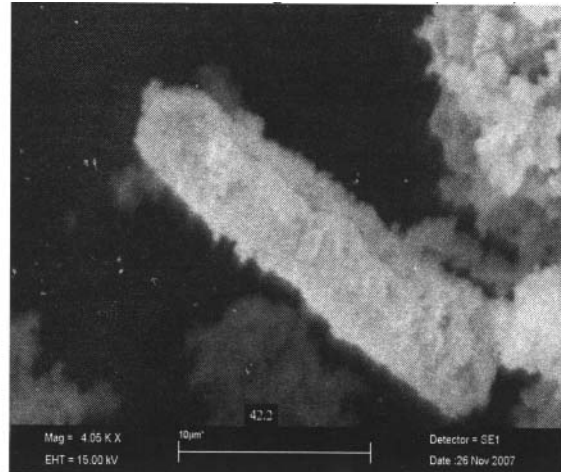**Figure 5a: Formation of Calcium Ferrite (4KX)**

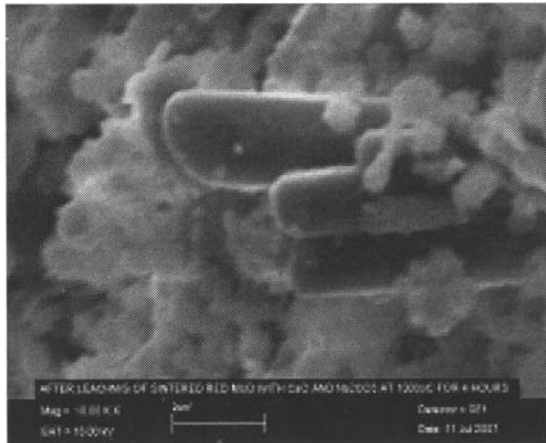**Figure 7: Formation of Magnesium Titanate (4.05KX)**

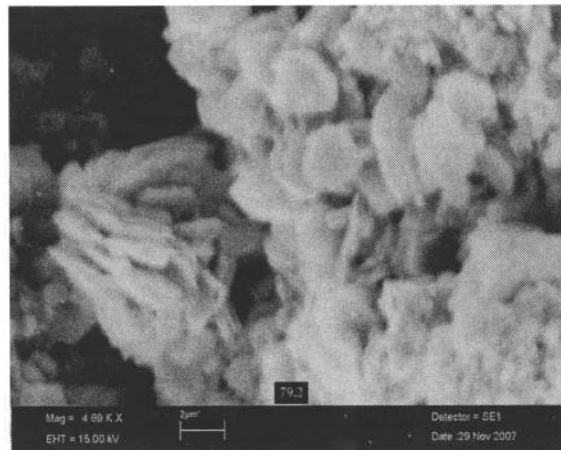**Figure 5b: Formation of Calcium Ferrite 10K**

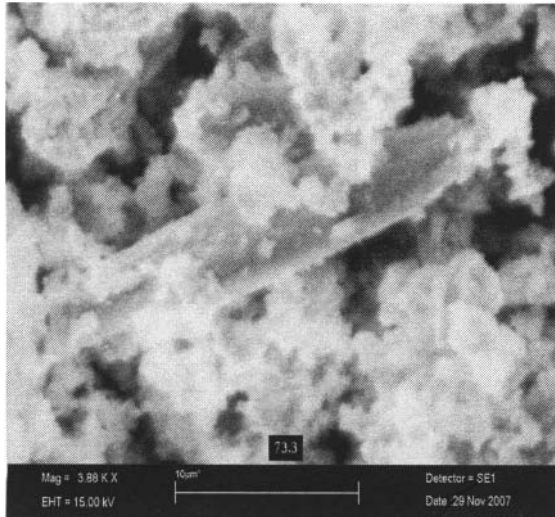**Figure 8: Formation of Barium Ferrite (4.69KX)**

Figure 9: Formation of Barium Titanate (3.88KX)

Table 3. Formation of major phases confirmed by XRD during sintering of red mud followed by leaching

Sinter Process	Formation of Major phases	Characteristic d spacing ($\mathring{A}$)
Red Mud + CaO +Na$_2$CO$_3$	Dicalcium Silicate	2.21
	Calcium Titanate	2.70,2.52,2.87
	Calcium Ferrite	2.70,2.67,2.10,1.84
	NaAlO$_2$	2.13,2.59,2.64,2.95
	CaSiO$_3$, CaV$_2$O$_6$	2.70,2.67,2.21
	CaMnO$_3$	2.67
	Calcium Aluminum Silicate	2.64
Red Mud + MgO +Na$_2$CO$_3$	Dimagnesium Silicate	2.53
	Magnesium Titanate	2.70,2.53,2.09
	Magnesium Ferrite	2.96
	NaAlO$_2$	2.96
Red Mud + BaO + Na$_2$CO$_3$	Dibarium Silicate	2.78,3.15
	Barium Titanate	2.13,2.24
	Barium Ferrite	1.63,2.43,2.63,2.78
	NaAlO$_2$	2.13,2.23

4. Conclusion

1. By adoption of the sintering method followed by alkaline leaching, the alumina extraction achieved was 97.64% in Lime-Soda Sinter process and impurities like SiO$_2$, V$_2$O$_5$ and MnO$_2$ are removed from the liquor. These impurities are removed from the crystal lattice of Hematite as well as Sodalite and Cancrinite to form insoluble materials like CaSiO$_3$ (d value= 2.70, 2.21), CaV$_2$O$_6$ (d value= 2.70, 2.67) and CaMnO$_3$ (d value= 2.67) confirmed by XRD.

2. In the MgO-Na$_2$CO$_3$ sinter process alumina extraction achieved was 98.7%.

3. In the BaO-Na$_2$CO$_3$ sinter process, the extraction efficiency achieved was 99.5%.

But from an economic point of view, the lime-soda sinter process is best and suitable method for extracting the remaining alumina from Bayer's process waste residues (Red Mud).

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