Extraction of Iron values from Red mud

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Abstract

Red mud is an aluminum industry waste generated by Bayer's process. It contains around 30-50% iron oxide depending upon the quality of bauxite ore used in Bayer's process Due to its highly caustic-corrosive nature and fine particle size it is considered toxic to the surrounding. In this work, an attempt has been made to achieve a suitable concentrate for iron making. It was found that the carbonation step helps in alkali recovery from red-mud in the form of dissolved sodium aluminate which can be recycled in Bayer's process. X-Ray diffraction of the carbonated residue of the red-mud system showed that the complex structure of the desilication products i.e., cancrinite and sodalite formed in Bayer's process dissociates into sodium aluminate, sodium silicate and sodium alumino silicate which improves the iron recovery. Specific pretreatment process with an incorporation of a primary magnetic beneficiation prior to reduction roasting followed by secondary magnetic beneficiation manifest optimal conditions for iron recovery and proved economical in terms of specific consumption of reductant. Carbonated red mud concentrated with magnetic separation followed by muffle furnace roasting using suitable reductant produced a 50.5% iron-rich concentrate with 70% iron recovery. This technique of iron recovery from red-mud consumes two harmful industrial wastes and shows promising results.

1. Introduction

Aluminum is the most abundant metallic element found in the earth's crust and is primarily extracted from bauxite ore. It is often present in Gibbsite or diaspore mineral phase in bauxite. Bayer's process involves digestion of grounded bauxite ore in caustic solution at high temperature and pressure in autoclaves. The digestion leads to selective precipitation of iron and other impurities present in bauxite ore. This fine precipitate is separated in the

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form of a slurry which has reddish appearance due to the presence of significant amount of iron oxide. The reddish appearance of precipitate gives it the name red mud whereas the aluminum dissolved in the solution in the form of sodium aluminate is further processed to form alumina which is transformed into aluminum by the Hall-Heroult process. The modified process flow sheet of Bayer's process is shown in Figure 1. The amount of red mud generated per ton of alumina depends upon the quality of the bauxite ore used and on the process parameters. Generally, 1 – 2.5 ton of red mud is produced per ton of alumina processed. In 2016, approximately 4 million tons of Red mud was produced in India only and ~90 million tons of red-mud was produced worldwide [1]. There is no wide-scale application of this abundantly generated toxic waste except stock-pilling. Red-mud waste produced in aluminum industries are most commonly discharged in engineered impoundment reservoirs. The physicochemical properties such as high alkalinity, corrosiveness, fine particle size and poor settling tendency of red mud make it toxic to humans and environment. Various industrial accidents related to red-mud eventuated over the time such as Akja aluminum refinery in Hungary, 2010 in which thousand million liters of red mud sludge was spilled which destroyed the flora and fauna of the nearby localities [2]. The significant presence of iron oxide along with the occurrence of a trace amount of rare earth elements (REE) makes red mud a potential source for iron production. Dedicated research work carried on red-mud to recover iron values is tabulated in Table 1. Briefly, authors have preferably opted smelting, sintering process and reduction roasting in presence of sodium salts as a flux to extract iron values in the form of iron nuggets and Ferroalloys [3]. These techniques consume a substantial amount of thermal energy as associated temperatures attained in these techniques are very high.
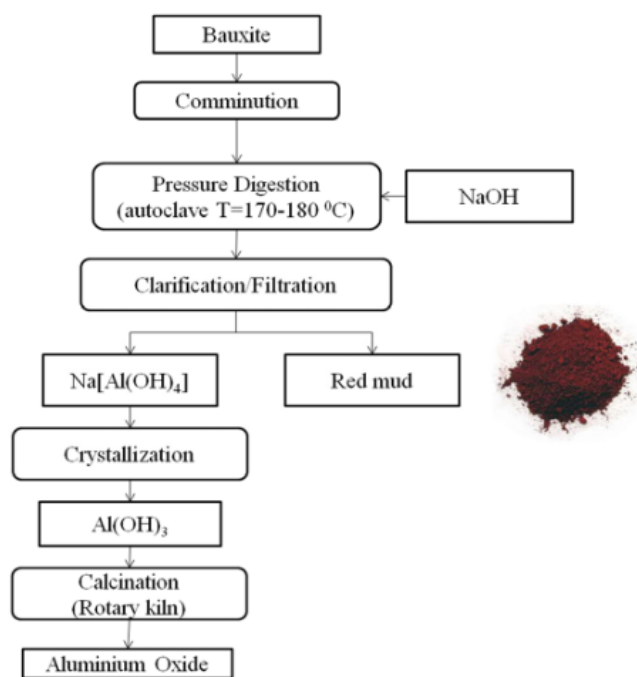

Fig. 1. Flow sheet of Bayer's process for bauxite processing.

Apart from pyrometallurgical process, researchers have also attempted hydrometallurgical process to recover iron values. Red-mud is dissolved with oxalic acid to selectively leach iron and further leach liquor was processed to extract dissolved iron values [11, 12]. Some efforts has also been made to separate iron from red mud by physical beneficiation steps such as multistep magnetic separation and High gradient Superconducting Magnetic Separation [13]. Several neutralization techniques were investigated to neutralize red mud like seawater neutralization, carbon-dioxide neutralization and acid treatment measuring the pH change, concentration of leach solution produced, alkalinity and solution aluminum concentration [14]. The carbonation process refers to chemical process of binding carbon dioxide to substrates. Alkali oxides and metal hydroxides react with carbonic acid formed by the bubbling of carbon dioxide in red-mud slurry to give respective carbonates and bicarbonates which are neutral in nature and

reduce the pH value of the substrate. A 500 MW thermal power plant generates approximately 3 million tons of carbon dioxide in a year which would abruptly damage the environment [15]. Atmospheric carbon dioxide or CO_2 containing flue gases can be a significant source of carbonation. It is believed that the gas phase CO_2 can be bubbled through aqueous slurries to form carbonic acid which can react with the basic components of red mud thus breaking the structure of complex phases in red mud. Some work is also reported of neutralizing the red mud and as a potential source of raw material in construction materials like bricks and ceramics [16].

Table 1. Literature review on iron extraction from red-mud

S. No	Procedure description	Key findings	Reference
1	Iron is separated from red mud using hydrogen gas in a tube furnace followed by magnetic separation.	Optimum conditions for conversion of hematite to magnetite were 2.5 times stoichiometric supply of hydrogen, 480 ^{0}C and 30 minutes reduction time.	[4]
2	Pellets of mixture of red mud mixed with graphite powder and wollastonite were prepared and roasted in a vertical tube furnace in Argon environment (0.4L/min). Slag formed in the experiment was crushed and leached in HCl, HNO_3 and H_2SO_4 at 90 ^{0}C for REE extraction.	Prolonged reduction and excessive amount of reductant degrades the slag metal separation which reduces iron recovery. Increase in temperature and acid concentration shows better recovery of REE during leaching.	[5]
3	Fine red mud powder was roasted in a vertical tube furnace with soda, lime and coal char in the molar ratio of CaO/SiO_2 and Na_2O/Al_2O_3 as a target. Experiment was performed under N_2 environment (0.5L/min).	The optimized roasting conditions were molar ratio of Ca/Si around 2 and Na/Al around 1, 0.5gm coal char per 100gm red mud, 1000^{0}C for 3 hrs. The leaching conditions were 50gms sample per 100ml water, at 60^{0}C for 15 min.	[6]
4	The smelting reduction was carried out in a 35kW DC extended arc thermal plasma reactor. Red mud was mixed with flux and graphite as per stoichiometric requirement and flow of plasma gas (1L/min) was maintained.	Optimum conditions were using 12% dolomite as flux and graphite as reductant which converts into carbon monoxide above 1000^{0}C and 15 minute reduction time.	[7]
5	Recovery of iron by direct reduction roasting followed by magnetic separation and preparing bricks from the residue. Calcium carbonate and magnesium carbonate were added in 1:1 ratio along with activated carbon as reductant in the furnace.	At 1150^{0}C FeO, SiO_2 and Al_2O_3 react to form low melting point substance which hinders the reactivity of the reactant. With increasing temperature the low melting substance cumbers and enhances the reduction of iron oxide. At 1100^{0}C the iron recovery was maximum and 18% carbon.	[8]
6	Calcification with lime is performed in autoclave at 180^{0}C. The leached solution contains sodium and the leach residue is further treated for carbonation in jet flow reactor at 80-120^{0}C at 0.5 − 1.2 MPa for 1-4 hrs. The resultant is alkali leached at low temperature for alumina recovery.	By calcification technique the silica is transformed into hydrogarnet which was further decomposed into calcium silicate, calcium carbonate and aluminium hydroxide by carbonation. The aluminium hydroxide was recovered by alkali leaching of the residue.	[9]
7	$2Fe_2O_3+C\rightarrow4FeO+CO_2(g)$, $FeO+Al_2O_3\rightarrow FeAl_2O_4$ (Hercynite), $2FeO+SiO_2\rightarrow Fe_2SiO_4$(Fayalite), $2SiO_2+3Al_2O_3\rightarrow3Al_2O_3.SiO_2$ (Mullite) Two crucibles one inside the other were used to ensure complete reduction atmosphere the outer crucible was coated with carbon layer.	The reduced ferrous oxide preferentially reacts with aluminium oxide to form hercynite and surplus ferrous oxide reacts with silicon dioxide. Above 900^{0}C, reduced ferrous oxide reacts with aluminium oxide and silicon dioxide to form hercynite and ferrous silicate. Hercynite phase is paramagnetic and insoluble.	[10]

2. Raw Material

The red mud sample used in this study was procured from Hindustan Aluminum Corporation (HINDALCO) situated in Uttar Pradesh, India. The received red-mud sample was homogenized in ball mill and sieved below 100 microns. The representative sample was dried in an oven at 110^{0}C for 10 hours prior conducting the experiments to

remove the moisture from the sample. X-Ray diffraction was carried on the oven dried sample to identify the mineralogical phases of red-mud as shown in figure 2a. Elemental composition of red mud was determined using standard wet chemical analysis. The red mud sample mainly composed of Fe_2O_3, Al_2O_3, TiO_2, SiO_2 and Na_2O as shown in Table 2.

Table 2. Wet chemical analysis of red mud sample.

Constituent	SiO_2	TiO_2	Al_2O_3	Fe_2O_3	CaO	Na_2O	LOI
Weight %	8.32	17.10	16.58	36.26	1.43	6.00	9.10

The major mineral phases occurring in red mud are Cancrinite [$Na_8Al_6Si_6O_{24}(OH)_2(H_2O)_2$], Quartz[$SiO_2$], Hematite[$Fe_2O_3$], Anatase[$TiO_2$], Sodalite[$NaAlSiO_4$], Gibbsite[$Al(OH)_3$] and Diaspore[$Al(OH)_3$]. The Thermo gravimetric/ Differential Thermal Analysis (TG/DTA) of red mud feed is shown in Figure 2b. Several steps in the weight loss over different range of temperature can be observed from the thermal decomposition profile. The first weight loss of 3.1% from room temperature to 200^0C started with the evaporation of the moisture present in the red mud followed by the steep fall in weight of 3.8% from 230^0C to 330^0C which is attributed to the structural water loss from the constituents (Cancrinite, Sodalite). The increment in weight above 1200^0C is due to re-oxidation of iron present in the red mud.
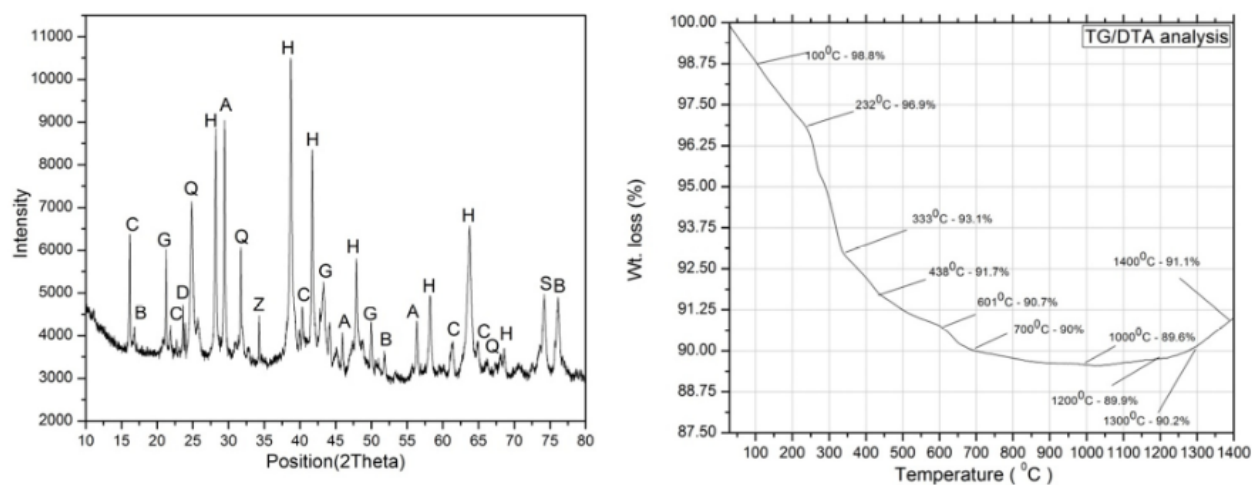

Fig. 2(a) XRD plot, (b) TG-DTA analysis of red-mud in air (H-Hematite, A-Anatase, G-Gibbsite, C-Cancrinite, S-Sodalite, B-Bohemite, Q-Quartz, D-Diaspore, Z-Calcium Carbonate).

3. Experimental

In this research, a novel experimental approach is designed to stepwise recover alkali content of the red-mud by carbonation followed by iron recovery. The carbonation process reduces the pH value of the red mud from 12 to 7. The slurry obtained from carbonation was vacuum filtered to separate residue and solution. The residue obtained after carbonation was further processed for iron extraction and the solution can be recycled back to Bayer's process. Combined roasting process is designed along with magnetic separation to extract iron values. The carbonation process was carried in a custom made carbonation setup which comprises a 2000ml Erlenmeyer flask and industrial grade CO_2 gas as a carbon dioxide source. All the carbonation experiments were carried at ambient conditions in a standard magnetic stirrer solid to liquid ratio 1:25 and 600 rpm stirring speed at atmospheric pressure and room temperature. pH of the red mud slurry was strictly checked during the initiation and completion of the carbonation process. The progress of carbonation treatment on red mud is monitored by studying the mineralogical phase changes occurring in carbonated red mud by XRD (Fig. 4a) analysis of residue. Also the dissolution of alkaline

elements like sodium and aluminum in carbonated solution was measured by Flame Photometer and ICP-MS (Inductively Coupled Plasma Mass Spectroscopy). Carbonated residue was then enriched with the help of primary magnetic separation step, reduction roasting step and combination of both pretreatment steps. In combined pretreatment step, reduction roasting was performed on iron rich concentrate obtained post magnetic separation. Reduction experiments were carried in a silica crucible in standard muffle furnace and domestic microwave at 900W power. A layer of calcium oxide was coated in all the reduction experiments to ensure trapping of carbon dioxide gas formed for sufficient time. It is important to mention that Calcium oxide is inert at experimental temperatures. All the reduced samples were water quenched post reduction to prevent re-oxidation of reduced sample. Reduced samples were pulverized below 100 micron followed by secondary magnetic separation. The magnetic separation of reduced red mud is carried at 0.8 kA/m or 2000Gauss. The effect of carbonation time on the sodium recovery and effect of magnetic field intensity of WHIMS (Wet High Intensity Magnetic Separation), roasting temperature and roasting time on extraction of iron from carbonated residue of red-mud were investigated in series of experiments. The experimental plan followed in this study is shown in Figure 3.
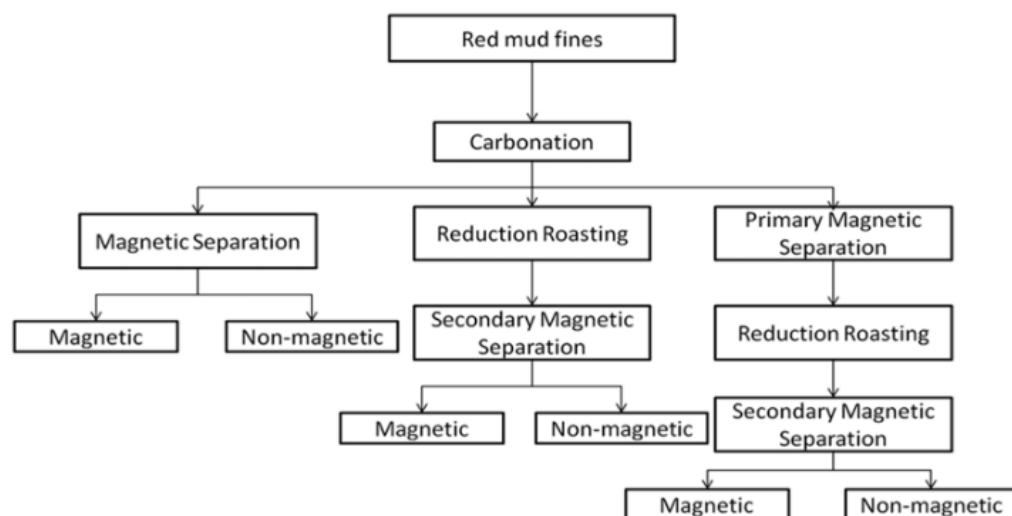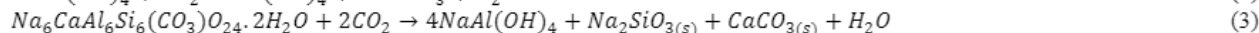

Fig. 3. Experimental plan for beneficiation, pyrometallurgical treatment and combined pretreatment for recovery of iron values from red mud.

4. Result & Discussion

It is evident from Figure 4a that cancrinite and sodalite phases disappear and carbonate and bicarbonate of sodium and calcium along with sodium aluminate and sodium silicate are formed. It was expected that the carbonation of red mud aids in breaking the complex structure of sodium and calcium and thus liberates the trapped alumina and alkali from the red mud which imparts better separation tendency of iron from the red mud. The reactions occurring in the system during carbonation process are shown in equation (1)-(3).

$$CO_2 + OH^- \rightarrow HCO_3^- \tag{1}$$
$$NaAl(OH)_4 + CO_2 \rightarrow Al(OH)_4 + NaHCO_3 + H_2O \tag{2}$$
$$Na_6CaAl_6Si_6(CO_3)O_{24}.2H_2O + 2CO_2 \rightarrow 4NaAl(OH)_4 + Na_2SiO_{3(s)} + CaCO_{3(s)} + H_2O \tag{3}$$

4.1. Effect on alkali recovery with variation in carbonation time
Post carbonation the pH value of the red mud slurry was reduced to 6-7 as compared to initial pH 12. This is due to the formation of neutral alkali carbonates and bi-carbonates by reaction of carbonic acid with the cancrinite and sodalite phases in red mud slurry. As shown in Figure 4a comparatively less stable cancrinite breaks down first on 30 minutes carbonation whereas sodalite remains as such in the system. On further increasing the carbonation time sodalite structure gets dissociated into neutral compounds. It is also observed that the settling time of red-mud particles in the slurry decreases by manifold (feed hours; carbonated in minutes). With increase in carbonation time,

aluminum as well as sodium was found to dissolve significantly (30minutes; Al: 8.9%, Na: 17.4% and 60minutes; Al: 16.4%, Na: 72.3%).

4.2. Magnetic beneficiation of red-mud

The effect of carbonation on recovery of iron was studied by incorporating a physical beneficiation step in the carbonated residue of red-mud and feed red-mud. It can be observed from Figure 5a that iron grade increases with increase in magnetic separation intensity and the carbonated product always shows a significant increase in the enrichment of iron in the magnetic concentrate. Hence, it can be inferred from the experiments that breaking of cancrinite and sodalite phases present in the red-mud system enhances the iron extraction. Also, the trend of increase in iron values with increase in magnetic intensity can be explained as more paramagnetic iron containing phases will get trapped with increase in magnetic intensity. Although, there was significant improvement in iron enrichment in concentrate still it is not suitable for iron making.

4.3. Pyrometallurgical treatment of red-mud

4.3.1. Muffle furnace roasting

Similarly, the effect of carbonation was studied at different reduction temperatures for 90 minutes reduction time with 10% activated charcoal as shown in Figure 5b. The stoichiometric amount of reductant required in the reduction roasting was calculated from the reduction reaction of ferric oxide to ferrous oxide as shown in equation (4)-(5).Theoretically carbon is stable as carbon monoxide gas above 1000^0C therefore reduction reaction takes place with carbon monoxide gas above 1000^0C and with carbon dioxide gas below 1000^0C. The increase in iron grade with increase in temperature was due to increase in partial pressure of CO which results in faster reaction kinetics. Also the carbonated red mud shows a significant enrichment in iron compared to red mud feed. Hence, it can be concluded that carbonation process is successful pretreatment step in recovering iron values from red mud.

$$3Fe_2O_3 + C \rightarrow 2Fe_3O_4 + CO \quad (<1000\ ^0C) \tag{4}$$
$$3Fe_2O_3 + CO \rightarrow 2Fe_3O_4 + CO_2 \quad (>1000\ ^0C) \tag{5}$$

4.3.2. Microwave roasting

In order to improvise the roasting process, radiation heating was incorporated with the help of microwave system instead of conduction heating as in case of muffle furnace. The radiation heating has a particular edge over conduction heating because the rate of temperature rise is very fast in radiation heating and entire sample gets reduced at same time as compared to layer wise reduction in conduction heating. As shown in Figure 6 the reduction characteristics of hematite present in red-mud system is much better in microwave roasting than that of muffle furnace roasting. The concept of carbonation remains valid in this case also. The carbonated red-mud gives better grade and recovery compared to uncarbonated red-mud. The roasting conditions were performed at 10% activated charcoal as reductant and roasting time of 10 minutes. Similarly, it was observed that on increasing the reduction time the grade and recovery falls as the elements present in red-mud fused together resulting in formation of some paramagnetic iron phases which reduces the grade and recovery. Further XRD analysis was required to substantiate this fact.

4.4. Combined pyrometallurgical – physical beneficiation treatment of red-mud

Although, carbonation pretreatment proved to be a successful step in recovering iron rich concentrates from red-mud, the current grade achieved is not suitable for metallurgical application. Hence, a combined pretreatment technique was designed to achieve desired grade concentrate. A primary magnetic separation step is incorporated before reduction roasting of the carbonated residue. It can be beneficial from economic point of view as reductant consumption per unit mass of the product and the specific energy consumption in roasting decreases. Various parameters that influence the pretreatment process are magnetic separation intensity, reduction temperature and reduction time. The effect of each parameter on iron grade and recovery of the carbonated residue is investigated by a series of experiments and optimized conditions are stated. At first the effect of primary magnetic separation

intensity is pursued on iron grade and recovery from carbonated red mud. Series of experiments were conducted on carbonated red mud at intensity varied between 10,000-16,000 Gauss.
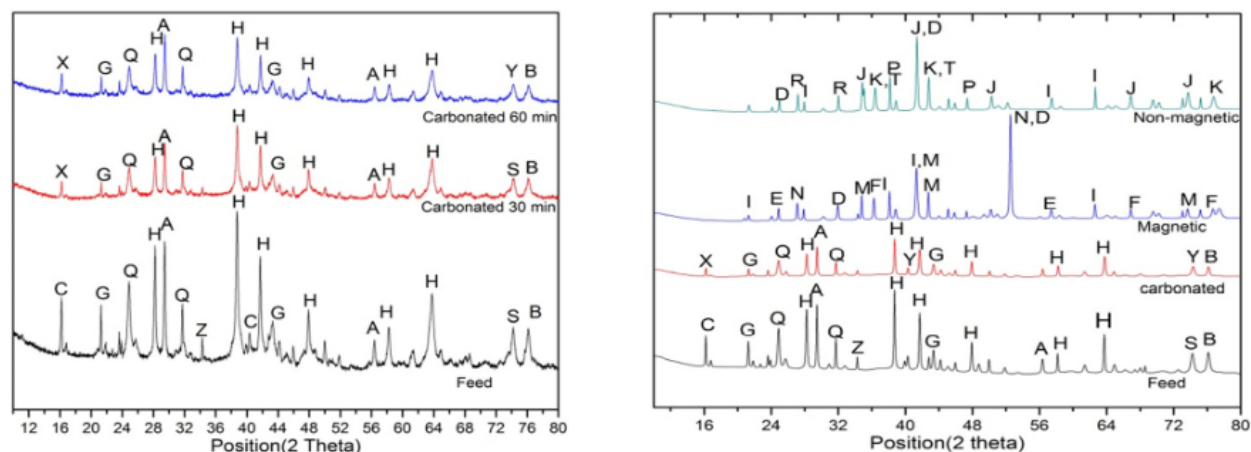

Fig. 4. XRD plot of (a) red-mud feed and carbonated sample at 30 and 60 minutes (b) feed, carbonated, magnetic and non-magnetic concentrate at optimal conditions (A-Anatase, B-Bohemite, C-Cancrinite, D-Cristobalite, E-Goethite, F-Hercynite, G-Gibbsite, H-Hematite, I-Illeminite, J-Magnesium Titanium Oxide, K-Spinel, M-Magnetite, P-Perovskite, Q-Quartz, R-Rutile, S-Sodalite, T- Gehlenite, X-Sodium Silicate, Y-Sodium Alumino Silicate, Z-Calcium Carbonate)
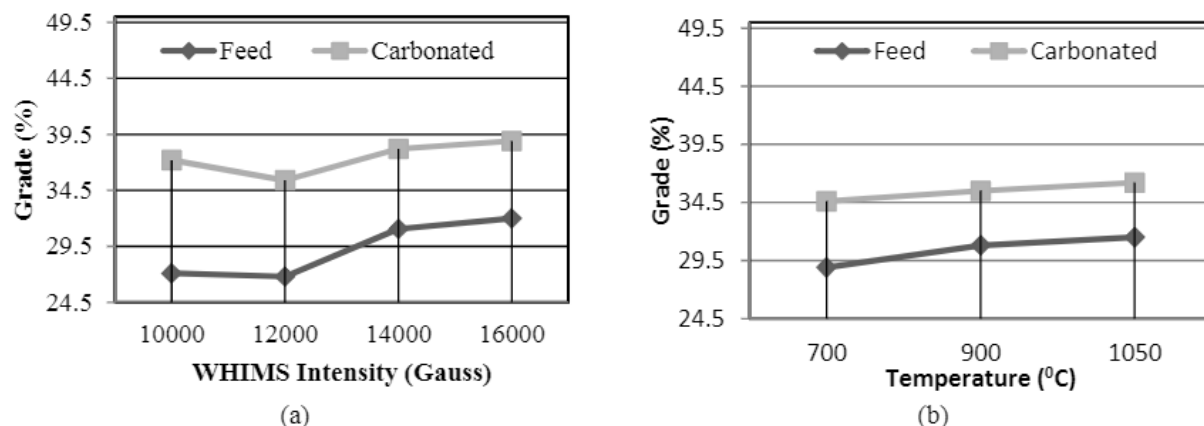

Fig. 5. Variation in grade of red-mud and carbonated red-mud at (a) different intensity of magnetic separation (b) different reduction temperature in muffle furnace.

The reduction temperature in all the experiments was fixed at 1050^0C and reduction time for 90 minutes. A graph was plotted for grade and recovery at different WHIMS intensity as shown in Figure 7a. The trend in increase in grade with increasing WHIMS intensity can be explained as with increase in intensity more and more iron phase minerals will get accumulated in magnetic concentrate along with other impurities which increases the grade whereas recovery of iron has mixed trend. Similarly, the reduction time is varied from 30 min to 120 min in the muffle furnace to study its effect in iron grade and recovery as shown in Figure 7b. During the reduction period of 30 min the complete conversion of hematite to magnetite does not take place which results in decrease of iron grade. With increasing time duration for reduction, more fraction of hematite gets converted and thus the grade increases but prolonged reduction leads to interaction of aluminum and titanium with iron present in red mud to form hercynite and illeminite which reduces the iron grade. Next, the effect on grade and recovery of iron with varying

reduction temperature is investigated. Three different temperatures (700^0C, 900^0C & 1050^0C) are investigated for the reduction of iron phase in red-mud into magnetite or other ferromagnetic phases. As shown in Figure 7c, the grade and recovery of iron increases till 900^0C and then falls at 1050^0C.
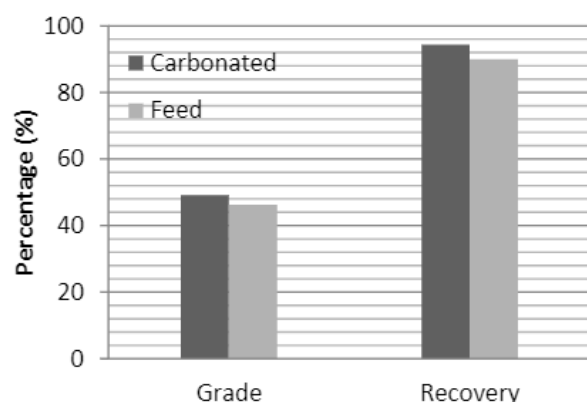

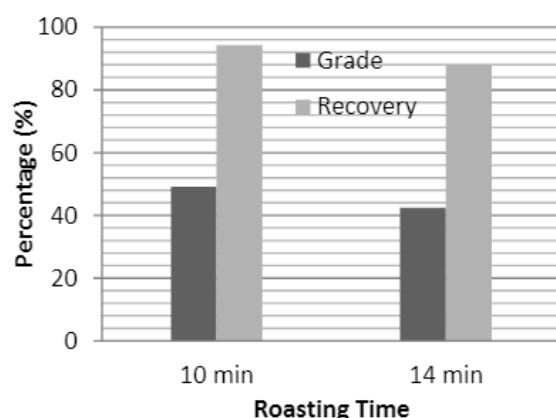Fig. 6a. Comparison of feed and carbonated red-mud in microwave roasting for 10 minutes with 10% charcoal

Fig. 6b. Effect of microwave reduction time on grade and recovery of carbonated red mud
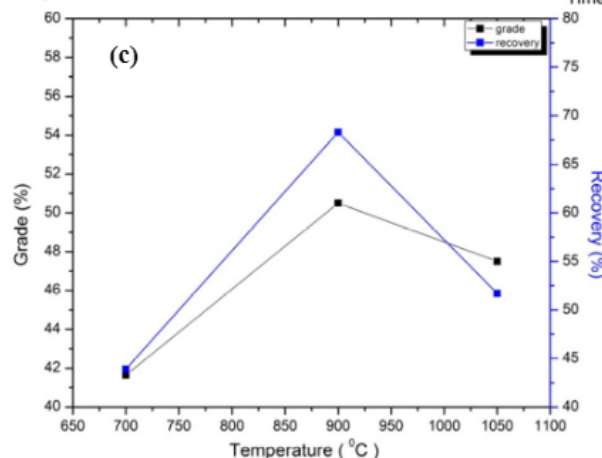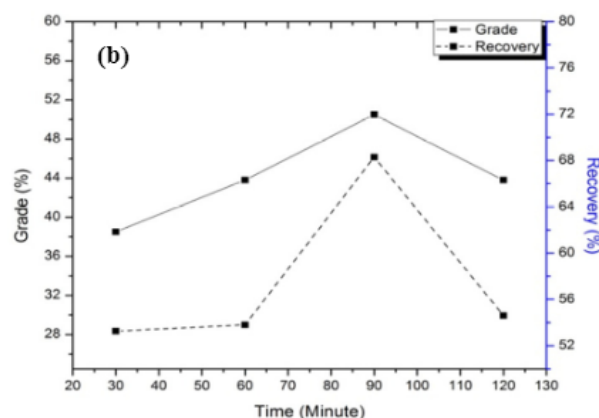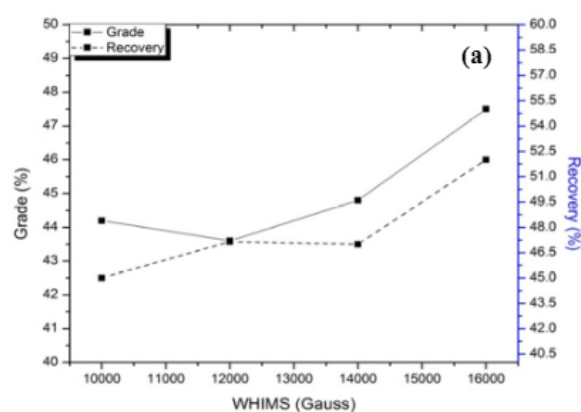

Fig. 7. Variation in grade and recovery at (a) different magnetic intensity at 1050^0C for 90 minutes, (b) changing reduction time at 900^0C temperature at 16000 gauss, (c) varying roasting temperature for 90 minutes at 16000 gauss.

It is also observed that color of red-mud doesn't change at 700^0C and unburnt carbon is also present in significant amount which can be due to incomplete reduction with increase in temperature the amount of unburnt carbon decreases. At higher temperature, reduced ferrous compounds can be physically observed by color difference in the lower and upper half of the reduced mass in crucible. The lower portion has a grayish tint probably due to conversion of hematite to magnetite. The XRD plot of final concentrate (magnetic and non-magnetic) at optimized conditions is plotted along with feed and carbonated residue as shown in Figure 4b. Magnetic concentrate contains magnetite and goethite along with iron-aluminum phase (hercynite) and iron-titanium phase (illeminite) whereas non-magnetic concentrate contains mainly titanium (rutile, perovskite, magnesium titanium oxide) and silicon (cristobalite, gehlenite, spinel) phases. Based on the phases present in the final concentrate, it can be concluded that iron preferentially interact with aluminum over titanium which results in fall in grade and recovery of iron in magnetic concentrate.

5. Conclusion

Based on the experimental work carried out in this study, following enumerated conclusions are considered to be significant for the processing strategies of iron recovery from red-mud.

(a) Carbonation step was found to be successful pretreatment step to dissociate complex desilication phases i.e., cancrinite and sodalite and in dissolution of aluminum and sodium. Thus reducing the pH value of the red-mud and improving the settling of red-mud by manifold.

(b) Carbonation pretreatment step helps in achieving an enriched iron concentrate from red-mud.

(c) Based on few experiments in domestic microwave, it appears that microwave roasting of red-mud can be proved beneficial as compared to conventional muffle furnace roasting.

(d) Carbonated red mud concentrated with magnetic separation at 16,000 Gauss followed by roasting of iron enriched concentrate in muffle furnace at 900^0C for 90 minutes using 10% charcoal produced a 50.5% iron rich concentrate with 70% iron recovery.

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