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Sequestration of carbon dioxide in red mud

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ABSTRACT

In Bayer process of obtaining alumina from bauxite, the insoluble product generated after bauxite digestion with sodium hydroxide at elevated temperature and pressure is known as "red mud" or "bauxite residue." It is an alkaline residue with a high pH of 10.5–12.5. It is disposed as dry or semi dry material in red mud pond or abandoned bauxite mines. The environmental concerns which are related to the disposal of red mud are its large quantity and causticity resulting in soil and groundwater pollution. At present, there is no viable process developed for utilizing the bauxite residue, consequently, this large quantity of waste material is required to be disposed economically and safely to the environment. In this view, a pH-reduction processing step is incorporated to ameliorate the red mud by sequestrating it with CO_2. By mixing carbon dioxide into the bauxite residue, the compound's pH level can be reduced to levels normally found in alkaline soil. It was seen that full neutralization of red mud takes place following CO_2 carbonation, but the pH rebound of carbonated mixtures takes place and the pH drifts upward to a value of 9.4–9.7 with time when solution and solid remains in contact. Chemical, mineralogical, and morphological analysis of neutralized red mud is also studied in the paper.

Keywords: Bauxite residue; Red mud; pH; pH rebound; CO_2 neutralization

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1. Introduction

The capture and storage of carbon dioxide (CO_2) could play a significant role in reducing the release of greenhouse gases to the atmosphere. Approximately, one-third of all CO_2 emissions due to human activity come from fossil fuels used for generating electricity, with each power plant capable of emitting several million tons of CO_2 annually. CO_2 is released annually from stationary sources such as electric power plants, refineries, cement plants, and other industrial facilities. Capture of CO_2 for sequestration from stationary sources is easier than CO_2 capture from mobile sources, such as motor vehicles. Carbon sequestration (storage) is the isolation of CO_2 from the Earth's

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atmosphere which can play a significant role in preventing continued CO_2 increase in the atmosphere. CO_2 is already being captured in the oil and gas and chemical industries. Aluminum is commercially produced from bauxite ore through two main process steps. In the first, alumina (Al_2O_3) is extracted from the bauxite ore by the Bayer process (invented by Karl Bayer in 1887) and in the second, the Al_2O_3 is electrolyzed in a Hall–Heroult cell to produce the pure metallic aluminum. Global warming is of special interest to the aluminum industry as approximately 14 MW h/ton of energy is required for the aluminum refining process. Assuming that this energy will be met through the captive coal fired power plant, it would result in associated high levels of CO_2 greenhouse emissions. The process of capturing CO_2 and mixing it with bauxite residue (red mud), a waste from Bayer process would lock up large amounts of the greenhouse gas that otherwise would be released into the atmosphere. The process of carbon capture and storage, where CO_2 is removed from flue gases before being stored in underground reservoirs is called as carbon sequestration. Carbon sequestration describes long-term storage of CO_2 or other forms of carbon to either mitigate or defer global warming.

An enormous quantity of red mud is generated worldwide every year posing a major environmental problem. The present worldwide generation of red mud is ~75 million tons of which ~4 million tons of red mud is generated annually in India. Red mud mainly consists of Fe_2O_3 (35–55 wt %), Al_2O_3 (16–22 wt %), SiO_2 (4–16 wt %), Na_2O (3–6 wt %), TiO_2 (2–19 wt %), CaO (0.8–4.5 wt %), and other oxides in trace amount depending upon the mineralogy and process parameters used. In India, red mud is disposed of in land-based impoundments [1]. Stored bauxite residue represents a long-term environmental liability for aluminum manufacturers. Safe storage of these materials requires engineered impoundments with leachate collection and treatment to prevent contamination of soil and groundwater. The storage impoundments typically occupy hundreds of acres of land at processing sites. The dust (predominantly Na_2CO_3) formed on the dry residue surface can pose a health risk to wildlife or humans when dispersed by wind [1]. The physical and chemical properties of the residues, especially the high pH and large fraction of fine, silt-sized particles, are the major constraints limiting reclamation efforts on residue storage areas [2]. Revegetation cannot be done due to the high pH, exchangeable sodium, and fine-grained characteristics of red mud, which result in a low hydraulic conductivity and a high impedance to the penetration of roots [3,4].

Red mud is disposed as dry or semidry material in red mud pond or abandoned bauxite mines. It is also disposed as slurry having a high solid concentration of 30–60% and with a high ionic strength. Research and development work for the utilization, storage, and disposal is being carried out all over the world. Problems associated with the disposal of red mud waste include its high pH (10.5–12.5), alkali seepage into underground water, safety in storage, alkaline air borne dust emissions, and the vast area of land required for disposal. Up to 2 tons of liquor with a significant alkalinity of 5–20 g/L caustic (as Na_2CO_3) accompanies every ton of dry mud. Red mud is a very fine material in terms of particle size distribution. Average particle size of red mud is less than 10 μm. A few particles of greater than 20 μm are also available. The specific surface area (Brunauer, Emmett and Teller) of red mud is between 10 and 30 m^2/g depending upon the grinding of bauxite. The specific surface of the red mud used for experimentation is 20.26 m^2/g.

At present, there is no viable process developed for utilizing the bauxite residue, consequently, this large quantity of waste material is required to be disposed economically and safely to the environment. This can be achieved by residue neutralization which will convert the highly caustic state of red mud to a state which is no longer highly caustic and is less hazardous. Neutralization will also open opportunities for reuse of the residue which to date have been prevented because of the high pH. Amongst the several pH-reduction steps to be incorporated to ameliorate the red mud, one of the methods for neutralization is treatment with CO_2.

The feasibility of treating red mud with acid has been studied [5,6]. Various aqueous acidic solutions have been considered for this application, including acidic industrial wastewater [4]. The use of carbonic acid has also been considered. A number of studies have been done for the feasibility of treating bauxite residue with acid on Kwinana red mud slurry. Treating red mud with acidic spent pickling solutions, derived from the steelmaking process, provides a coagulant—a mixture of aluminum and iron salts for waste water treatment [7]. Neutralization using acids is reasonably cheap if sufficiently large quantities of waste acid are present. Red mud has been treated with hydrochloric acid to dissolve only the sodium content [8] and the treated material is mixed with kaolinitic clay and formed into construction bricks. The potential of acidic fly ash to neutralize red mud slurry has also been studied [9].

The use of CO_2 from the atmosphere or from industrial emissions is another potentially significant source of acid for neutralizing red mud [10]. By mixing CO_2 into the bauxite residue, the compound's pH level is reduced to levels normally found in alkaline soil. Carbonation produces a product, which can potentially be used in other industrial and agricultural activities to remove toxic metals and nutrients. This new mixture can be used as road foundation, building material, or an additive to improve soil. India ranks 145th in the world in CO_2 emissions amongst 214 listed countries and emissions are 1.4 tons per capita [11].

Gas phase CO_2 or CO_2-containing flue gas can be bubbled through aqueous slurries to form carbonic acid in the aqueous phase [12]. The carbonic acid would react with basic components of the red mud, lowering its pH. Carbonation of red mud was carried out to study the capacity of this waste to capture CO_2. Experiments were conducted to explore the concept of beneficially utilizing mixtures of caustic bauxite residue slurry and produced oil-field brine to sequester CO_2 from flue gas generated from industrial point sources [13].

CO_2 treatment has offered quite a unique process fit solution for Alcoa. Dosing of the residue slurry with the CO_2 gas can be accomplished without any significant changes to the current dry stacking process [14]. Khaitan et al. [15] measured the extent of neutralization of bauxite residue by CO_2 as a function of CO_2 partial pressure to determine the geochemical reactions responsible for carbon sequestration. The investigators have stated that CO_2 sequestration in red mud is much smaller (0.029–0.057 million metric tons (MMT) CO_2 per year) compared to annual CO_2 emissions (110 MMT per year) associated with bauxite refining and red mud production of 30 MMT per year.

Although, the pH of the aqueous phase drops rapidly upon exposure to CO_2 gas, it soon rises again to unacceptable levels as additional alkaline material leaches from the mud. Hence, Shi et al. [16] have investigated the use of high-pressure liquid CO_2 rather than vapor phase CO_2 for the pH reduction of red mud. The initial red mud pH of 12–13 was lowered to 7–8 immediately after treatment and then slowly rose to values of 9.0–9.5 due to reactions of the treated red mud.

According to the investigators [16], the approximate amount of sequestration is about 2.3 g CO_2 per 100 g of dewatered red mud. Studies carried out by other investigators [17] show CO_2 sequestration of 7.02 g CO_2 per g of red mud. According to the values calculated by [13], a total bauxite residue/brine slurry volume of approximately 1.42×10^{11} L of reactive mixture would be carbonated per year resulting in sequestration of 1.3 million tons per year of CO_2. A detailed review of the neutralization processes and utilization of red mud is presented by researchers [18,19]. Modification and utilization of dried red mud for construction of vegetation cover and neutralization of red mud with pickling waste liquor using Taguchi's design of experimental methodology has also been studied by them [20,21].

In view of this, the focus of the investigation was to study the neutralization of red mud with CO_2 and pH rebound phenomena which occurs when the solid and liquid phase of carbonated mixtures are in contact after neutralization of red mud with CO_2 gas. Neutralization and pH rebound phenomena have been studied for a prolonged duration (for seven weeks). Red mud has been neutralized in single cycle (neutralization only once) and multiple cycles (i.e. neutralization of red mud several times after the pH rebound). The results are presented in terms of reduction in pH with time (one week) and cycles.

2. Experimental

2.1. Materials

Red mud from an Al_2O_3 refinery located at the eastern coast of India (18°49′9′′N,82°57′52′′E) which uses gibbsitic bauxite, distilled water, and CO_2 cylinder gas (Research grade, 99.99% pure) was used for experimentation. Typical chemical composition of red mud is given in Table 1.

Mineralogically, these red muds have phases of undigested Al_2O_3, aluminosilicates, phases of iron, and titania. These phases are hematite (Fe_2O_3), goethite $Fe_{(1-x)}Al_xOOH$ ($x = 0.33$), gibbsite $Al(OH)_3$, boehmite $AlO(OH)$, calcite ($CaCO_3$), calcium aluminum hydrate ($x.CaO.yAl_2O_3.zH_2O$), rutile (TiO_2), anatase (TiO_2), $CaTiO_3$, Na_2TiO_3, kaolinite $Al_2O_3.2SiO_2.2H_2O$, sodalites, aluminum silicates, cancrinite $(NaAlSiO_4)6CaCO_3$, and hydrogarnet $Ca_3Al_2(SiO_4)n(OH)_{12-4n}$.

Sodium is present mainly in two forms in red mud, free soda as ionized sodium aluminate and bound soda as desilicated product. Free soda is the caustic soda in the entrained liquor of red mud slurry which gets incorporated during digestion process and remains with red mud in spite of repeated washings. Free soda is in the form of $NaOH$, Na_2CO_3, $NaAlO_2$, etc. The pH of the red mud is due to the presence of these alkaline solids in red mud. Inclusion of caustic soda in bound form in the red mud is due to the desilication step carried out in the Bayer process for removal of kaolinitic silica in bauxite. Bound soda is in the form of sodalite complex which can be stated

Table 1
Typical composition of red mud

Constituents (%)	
Al_2O_3	16–18
Fe_2O_3	54–57
SiO_2	6.0–7.0
TiO_2	5.0–6.0
Na_2O	4.0–5.5
CaO	2.0–3.0

as "NAS" phases: $3(Na_2OAl_2O_32SiO_2)Na_2X$ ($X = CO_2^{2-}$, $2OH^-$, SO_4^{2-}, $2Cl^-$) [22]. In red muds, about 20–25% is the free soda while the rest is in the form of sodalite complex.

2.2. Methods

Red mud from the refinery was ground to 100 mesh size and used for the study. The pH of the slurry was measured after each test using LabX Light Titrator (Potentiometric Titrator), Mettlor Toledo GmBH, Switzerland. Analysis of chemical constituents of red mud has been carried by a wet chemical method in which the caustic soda has been determined by using a flame photometer (Systronic, Ahmedabad, India). Red mud obtained after neutralization was analyzed for its chemical constituents and scanning electron microscopy (SEM) was used to study the morphology of neutralized red mud using Electron Probe Analyser (JEOL, Japan, JXA-840A). Neutralized red mud was analyzed mineralogically using X-ray diffractometer (PAnalytical X-Pert Pro) using Cu Kα radiation ($\lambda = 1.54060$ Å).

Two experiments were carried out to study the pH rebound after neutralization of red mud with CO_2. Red mud slurry was made having 30% slurry solids in 100 mL distilled water with a pH of 10.71–10.77. The slurry was taken in one liter stainless steel vessel fitted with an agitator to ensure proper mixing of CO_2 gas with the slurry. Gaseous CO_2 at 0.1 atm pressure was injected into the red mud slurry for a contact time of 15 min. A gas bubbler was used to inject the gas in the slurry. The final pH was measured after neutralization. The neutralized slurry was stored for a week. The pH of the slurry was measured and again CO_2 was injected. The pH of the slurry was measured after CO_2 injection and the slurry was then stored for another week. Seven cycles of experimentation were carried out for seven weeks in this manner.

In another experiment, CO_2 was injected only once in the red mud slurry (30% solids) for 15 min time duration. The pH of the slurry was measured and the slurry was stored as it is for a week. After one week, the pH of the slurry was measured. The slurry was again kept as it is and pH was measured subsequently in each week for seven weeks.

The extent of pH rebound was assessed for both the carbonated red muds. After seven weeks, the red mud slurry was filtered through Whatman paper No. 40. The filtered mud was dried and analyzed for its chemical and morphological composition. The mineralogy of the neutralized red mud obtained after multiple cycle carbonation was determined.

3. Results and discussion

3.1. Neutralization of red mud and pH rebound

Fig. 1 shows the pH rebound phenomena in multiple cycle neutralization. Rate of neutralization is very fast i.e. within 15 min of contact time the red mud slurry is getting neutralized completely from an original pH of 10.77–7.466. But each time the red mud slurry is neutralized, it again rebounds back to a pH of about 9.0–9.50 within a week. The same phenomenon was observed for seven weeks even after neutralizing it to a pH of about 7.0 each time. A comparison of the pH values obtained before and after neutralization in each week is given in Fig. 2. This means that only the pore water (caustic soda liquor adhered to the red mud particles) is getting neutralized. This was due to the reaction of carbonic acid and OH^- in the pore water [15]. The basicity of the slurry again increases as more caustic soda adhered to the red mud particles was being slowly leached in one week. The driving force for the extraction also increases once the slurry was neutralized.

In another experiment, red mud slurry is neutralized only once wherein full neutralization takes place within 15 min as the pH value reduces from 10.71 to 6.7. But the pH rebounds back in subsequent weeks and gradually increases and stabilizes to a value of about 9.7 as shown in Fig. 3.

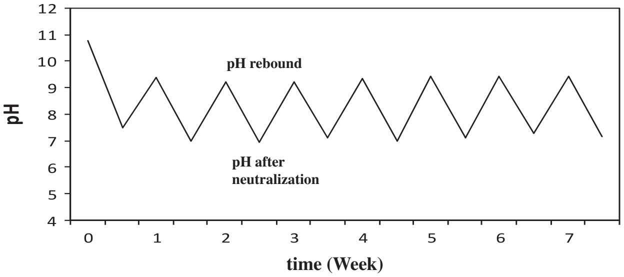

Fig. 1. pH rebound phenomena in multiple cycle neutralization of red mud with CO_2.

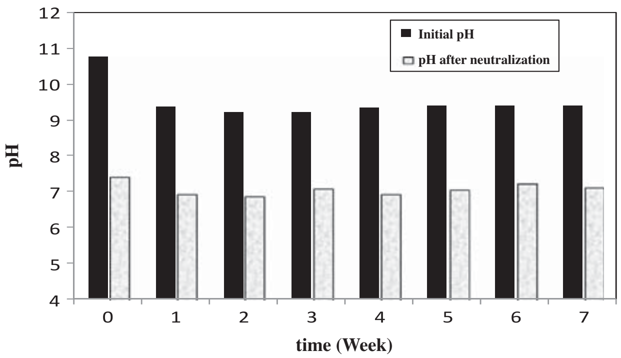

Fig. 2. Comparison of pH in each week in multiple cycle neutralization of red mud with CO_2.

The experiments show that more CO_2 can be sequestered in the red mud to reduce its pH but the processing cost would increase for more cycles. The same value of pH is achieved by injecting CO_2 only once indicating that less CO_2 could be sequestered in red mud but the processing cost would decrease. The experiments suggest that pH rebound occurs irrespective of the number of cycles the red mud slurry is neutralized. The pH rebounds back to quite a high value of 9.4–9.7 in both the cases which is also in accordance to the data reported by Cardile et al. [23]. Also according to investigators [13], the reactive mixture pH indicates full neutralization due to CO_2 carbonation and the pH of the carbonated mixtures has been observed to drift upward with time when solution and solid remained in contact. This strongly suggests that solid phase alkalinity associated with the by-product solids were released over time and drives the solution pH up. This is due to the phenomenon that gaseous CO_2 is reducing only the pH of pore water in the red mud due to the short contact time. The pore water carbonation occurs by the rapid absorption of CO_2 that forms carbonic acid and neutralizes the excess base in the form of NaOH, Na_2CO_3, and $Al(OH)_4{}^-$ in the pore water according to the mechanism which involves the following liquor reactions:

$$CO_2 + 2OH^- \longrightarrow CO_3^{2-} + H_2O \tag{1}$$

$$CO_3 + CO_2 + H_2O \longrightarrow 2HCO_3^-(aq) \tag{2}$$

$$HCO_3(aq) \longrightarrow CO_3^-(aq) + H^+\ (aq) \tag{3}$$

$$[Al(OH)_4]^- + Na^+ + CO_2 \longrightarrow NaAl(CO_3)(OH)_2 + H_2O \tag{4}$$

$$3Ca(OH)_2 \cdot 2Al(OH)_3 + 3CO_2 \longrightarrow 3CaCO_3 + 2Al(OH)_3 + 3H_2O \tag{5}$$

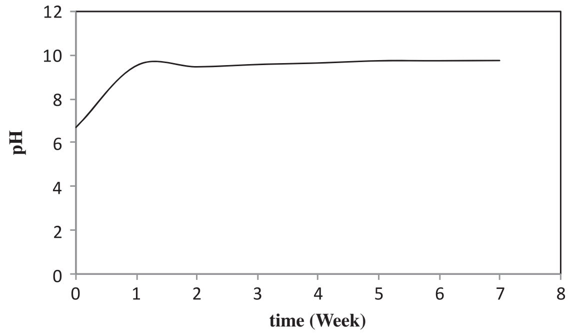

Fig. 3. pH rebound in single cycle neutralization of red mud with CO_2.

This suggests that the neutralization reaction is taking place with pore water dissolved species and the solids did not contribute significantly due to the short contact time. Solid phase reaction of tricalcium aluminate dissolution and $CaCO_3$ precipitation would occur after long-term carbonation which would actually reduce the pH of red mud slurry [15]. Storage of carbon in the form of carbonate is the most important factor for sequestration of CO_2 in red mud as stated in chemical reaction (5). Whether this sequestration is permanent or not needs further investigation because with time, these carbonates might dissolve to release CO_2. Smith et al. [24] hypothesized that dissolution of tricalcium aluminate in the neutralized slurries causes this pH reversion. It results in the formation of amorphous aluminum hydroxide and dawsonite ($NaAlCO_3(OH)_2$) that agglomerate together.

3.2. Red mud analysis after neutralization

3.2.1. Chemical composition of the red mud after neutralization

All the constituents except Na_2O content in red mud change in the red mud after treatment with CO_2 as can be seen from Table 2.

Nearly 23–24% soda reduction is observed in red mud from first experiment which was neutralized for several cycles while in another experiment, 19–20% reduction is observed wherein the red mud slurry was neutralized only once. This shows that only washable soda is being leached out from the red mud

Table 2
Chemical composition of neutralized red mud

Constituents (%)	Multiple cycle neutralization	Single cycle neutralization
Al_2O_3	16.85	16.70
Fe_2O_3	54.27	54.18
SiO_2	6.34	6.59
TiO_2	5.37	5.08
Na_2O	3.56	3.76
CaO	2.25	2.38

into the liquor phase and neutralized as explained by the previously described mechanism. Sodium carbonate is formed by the addition of CO_2 and this carbonate is an impurity to the Bayer circuit, however, processes (known as causticization) can be employed to convert this sodium carbonate back to sodium hydroxide.

3.2.2. Mineralogy of the neutralized red mud

Fig. 4 shows the mineralogy of the carbonated red mud obtained from multiple cycles. Neutralized red mud consists prominently phases of calcium carbonate or calcite ($CaCO_3$), sodalite or sodium aluminosilicates ($NaAlSi_3O_8$), iron (Fe_2O_3), titanium oxide (TiO_2), carbon along with undigested Al_2O_3, and other phases of red mud. $Ca(OH)_2$ has reacted with CO_2 to form $CaCO_3$ which has precipitated in red mud.

3.2.3. Morphological structure of red mud

Fig. 5 shows the morphological structure of untreated red mud which indicates scattered fine particles of about 1 µm size whereas Fig. 6 of neutralized red mud shows large agglomerations of 10–20 µm found after neutralization in multiple cycles. The agglomerates are circular with fine particles of iron, aluminum, and aluminosilicates attached to it. For single cycle neutralization, the SEM photograph shows some insoluble compounds having hexagonal structure being trapped inside the dense mass (Fig. 7).

3.3. Use of carbonated red mud

Carbonated residue can be used in brick and tile making, in fertilizer, as a filer in plastics or concrete, and as a replacement for construction sand. The waste might find an application as a soil amendment to also

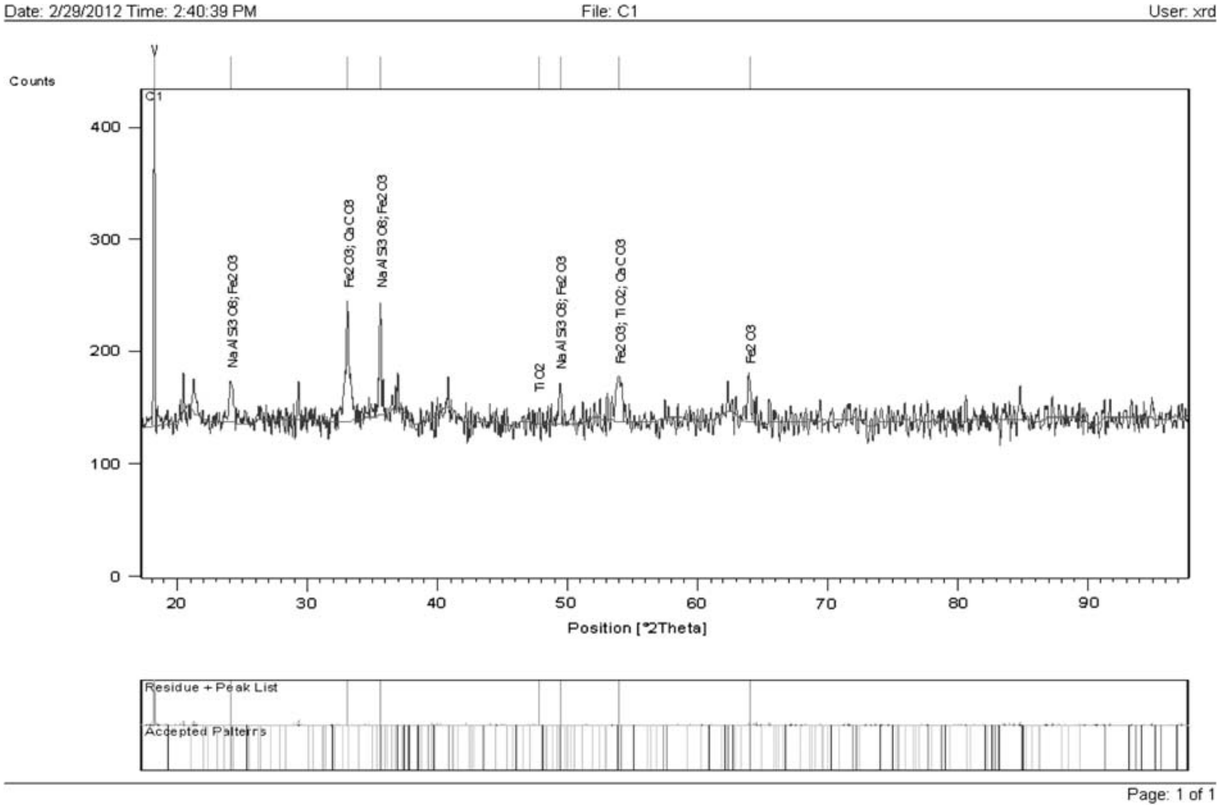

Fig. 4. Mineralogy of the neutralized red mud obtained from multiple cycle carbonation.

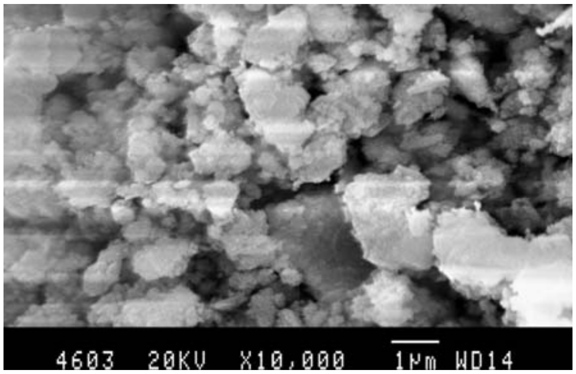

Fig. 5. SEM photograph of untreated red mud.

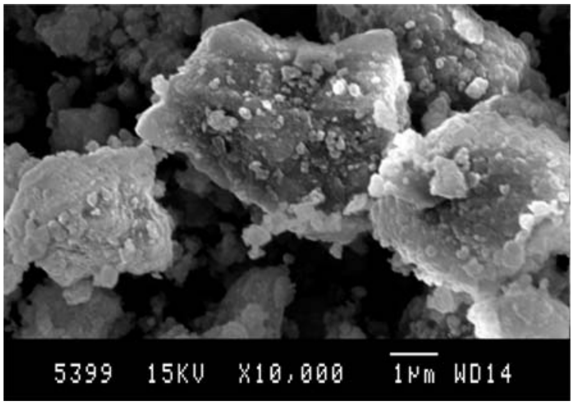

Fig. 6. SEM photograph of neutralized red mud (multiple cycle).

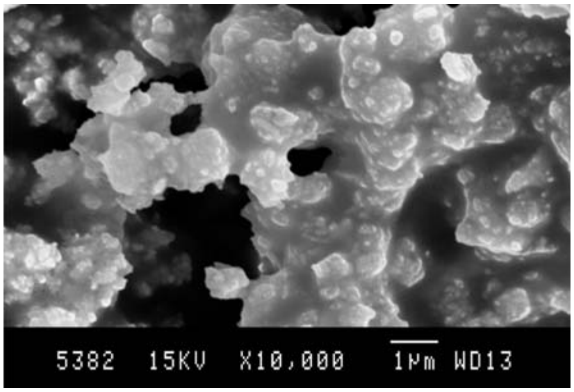

Fig. 7. SEM photograph of neutralized red mud (single cycle).

improve the soil pH. The initial cost of processing CO_2 in the red mud would be quite significant; the long-term benefits of carbonation cannot be ignored including entrapment of CO_2 from the environment to neutralize an alkaline waste. It will simplify storage and handling of red mud and also will open several reuse options.

4. Conclusions

It is seen that the pH of the red mud treated with gaseous CO_2 was close to 7.0–7.5 immediately after treatment; it soon rose again as additional caustic soda leached from the red mud. The stable pH of 9.4–9.7 is reached irrespective of the number of cycles CO_2 is injected. At the short contact times, which industrial process rates demand, only a fraction of the alkaline material in red mud is neutralized using gaseous CO_2. Hence, although the pH of the aqueous phase drops rapidly upon exposure to CO_2 gas, it soon rises again to unacceptable levels as additional alkaline material leaches from the mud. But CO_2 from the flue gases can be utilized for treating the soda in red mud by sequestrating it in multiple cycles which would ultimately reduce the alkalinity of the red mud. Also red mud will be a sink to sequester CO_2 from flue gases, thus reducing the pollution in air caused by greenhouse gases.

The CO_2 rapidly reacts with the pore water in the red mud. Dissolution of solid material back into the pore water causes the pH to rapidly increase again. The simplicity of the process suggests that impure CO_2 such as raw flue gas from the power plant might be used. Purifying CO_2 from a power plant gas may be the most expensive part of sequestration as the purity of CO_2 should be higher (more than 85%) for geologic sequestration. A small amount of fly ash or lime could be added to red mud to remove any residual sulfur in the flue gas as gypsum. Also, if more contact time is given to the reaction, more environmentally acceptable pH can be achieved. This would greatly improve the economics of CO_2 sequestration. Moreover, its use may be limited as the CO_2 source should be close to the alumina plant and also a significant amount of energy would be required to mix CO_2 into red mud slurry.

CO_2 can be recovered from various industrial sources such as ammonia plants, limestone calcination kilns, cement plants, thermal power plants, iron reduction plants, etc. Residue carbonation has been proven to be commercially viable by ALCOA and the carbonated residue can be used as soil amendment for acidic soils. Carbonation produces a product, which can potentially be used in other industrial and agricultural activities. Though, there may be limitations to make the process commercially implementable and further studies need to be carried out, the primary goal of carbonation to make the waste less toxic, easier to handle, and store would be fulfilled.

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