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Research Paper

Alkaline Roasting of a Red Mud–Sodium Carbonate Mixture After Milling

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***Abstract*:** After preparing a red mud sample from Iran Alumina Company (Jajarm) and determining its parameters, the chemical analysis and phase evaluations were undertaken using XRF and XRD methods. Mechanical milling of red mud-sodium carbonate mixtures was done for 15 minutes, 2 and 5 hours. STA analysis of the red mud sample and as-milled mixtures was undertaken under an air atmosphere up to 900 ºC with a heating rate of 20 ºC/min. The results showed the mass loss of red mud is about 13.25%, whilst it is in the range of 23-26% for milled mixtures. The alkaline roasting was done by heating the mixtures at the temperature range of 600-900 ºC under an air atmosphere for one hour. The traces of $NaAlO_2$, $NaAlSiO_4$ and $NaFeO_2$ phases were observed in the XRD patterns of the alkaline roasted samples. The signs of these phases were not observed in the milled mixtures of red mud-sodium carbonate; however the peak broadening and disappearing of the signs of sodium carbonate were the major events with increasing the milling time. Leaching of the alkaline roasted samples with water at 80 ºC for 60 minutes resulted in the dissolution of $NaAlO_2$ phase, and the traces of this phase are not observed in the solid residues after leaching. The iron concentration was trace in the leached solutions by the AAS method. The results indicated that alkaline roasting of red mud with sodium carbonate, followed by dissolving the roasted solid products in water, makes it possible to separate the contained aluminium from iron and iron compounds.

Keywords: Bauxite, Bayer process, Mechanical milling, Red mud, Sodium Aluminate.

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INTRODUCTION

Red mud is the by-product of the Bayer process in the production of aluminium oxide (alumina) from bauxite resources. The red mud normally contains iron oxides, silicon oxide (silica), titanium dioxide, alkaline oxides such as Na_2O and CaO and rare earth compounds. The traces of radioactive elements and unreacted aluminium oxides with caustic soda during the Bayer process have been found in the red mud. The pH of red mud is in the range of 10-13, its colour is normally red, and the mean particle size of the dried red mud is less than 100 μm [1-4]. Based on the chemical composition of bauxite resources, nearly 1-2 metric tons of solid red mud are produced from the production of one ton of aluminium oxide [5]. Therefore, the recovery of valuable elements and compounds from red mud is important. Some processes, such as acid leaching (acid washing), solid-phase reduction magnetic separation, and calcification–carbonation methods, are proposed to recover iron and/or aluminium oxide (alumina) from red mud [3]. Some research articles have also recently been presented about the alkaline roasting process to recover alumina from bauxite residue or low-grade bauxite resources [6,7]. Sodium aluminate ($NaAlO_2$) is formed by reaction (1) with soda ash (sodium carbonate) in the alkaline roasting process [8]. The endothermic reaction (1) is thermodynamically feasible at temperatures above 800ºC, and its theoretical mass loss due to the release of CO_2 gas is about 21.2% [8]. The produced phases, such as $NaAlO_2$ or $Na_{1.95}Al_{1.95}Si_{0.05}O_4$ from alkaline roasting of bauxite with Na_2CO_3, can be dissolved in water [9,10].

$$Al_2O_3 + Na_2CO_3 = 2NaAlO_2 + CO_{2(g)}$$

It is very hard to find research articles about the alkaline roasting process in the milled mixtures of red mud with sodium carbonate (Na_2CO_3) as well as the phase changes in this process. The possibility to recover the contained aluminium oxide from red mud and convert it to a water-soluble phase such as sodium aluminate is also the main objective of this manuscript. The mechanically milled mixtures of red mud-Na_2CO_3 have been used for the alkaline roasting method, and the phase changes have been studied after heating the milled mixtures at different temperatures. The leaching of the roasted samples was performed in water, and the phase changes of the solid residues were also compared with roasted samples in this work.

METHODS

After preparing the red mud sample from Iran Alumina Company (Jajarm) and determining its pH, the sample was dried in an oven at 80 ºC for 96 h. The density, BET surface area, PSA (particle size analysis), and chemical analysis of the dried red mud were determined. Table 1 shows the chemical analysis of red mud using the XRF method. The total amount of iron in the red mud sample was determined to be 17.57% by the ICP-OES method. The mixtures of red mud-sodium carbonate (Na_2CO_3, Merck grade) were prepared based on reaction 1 with the molar ratios of Al_2O_3:Na_2CO_3 = 1:2. The mechanical milling of the red mud-Na_2CO_3 mixtures was undertaken using a planetary ball mill (stainless steel cup/balls) with a BPR of 40:1 at different milling times (15 minutes, 2 and 5 hours). The STA analysis was done with a heating rate of 20 ºC/min under an air atmosphere. The alkaline roasting process of the milled mixtures was undertaken at different temperatures (600-900ºC) for one hour under air atmosphere in a tube furnace. The solid products of the alkaline roasted samples were leached with hot water at 80ºC for 60 minutes, then the solid residues of leaching were dried at 80ºC in an oven for 2 hours. The concentrations of Fe and Al in the leached solutions were determined via the AAS method, and the XRD patterns of the solid products were prepared under Cu radiation. The thermodynamics assessments were undertaken using HSC software [8].

Table 1. The chemical analysis (in weight percent) of red mud using the XRF method

SiO_2	Al_2O_3	CaO	MgO	TiO_2	MnO	P_2O_5	SO_3	Fe_2O_3	Na_2O	K_2O	SrO	ZrO_2	V_2O_5	L.O.I
15.15	18.14	17.84	1.16	4.61	0.08	0.72	< 0.01	22.38	5.52	0.79	0.05	0.1	0.09	13.37

FINDINGS AND ARGUMENT

The high pH of red mud in Table 2 is related to amounts of alkaline oxides such as Na_2O and CaO based on the XRF analysis in Table 1. The BET and PSA data in Table 2 indicate that the particle sizes of the red mud are very fine. The signs of phases such as boehmite ($Al_2O_3.H_2O$), iron oxide (Fe_2O_3, hematite), silica (SiO_2), Ca_2SiO_4, $CaCO_3$, Na_2O and TiO_2 have been detected in the XRD pattern of dried red mud.

Table 2. Data of pH, density, BET and PSA analysis of the red mud sample

D_{10} (µm)	D_{50}(µm)	D_{90} (µm)	BET($m^2\ g^{-1}$)	density($g.cm^{-3}$)	pH
~1	4.25	12.54	7.5753	2.97	11.7

Figure 1 shows the XRD patterns of red mud-Na_2CO_3 mixtures after different milling times. The peaks of sodium carbonate (*JCPDS-84-0176*) are observed with high relative intensities in the 15-minutes milled mixtures; meanwhile, the signs of the boehmite phase (*JCPDS-21-1307*) at 14.5, 28 and 45.8º, the hematite phase (*JCPDS-84-0311*) at ~24, 33.4 and 35.8º, and the traces of SiO_2 (*JCPDS-80-1288*) and $CaCO_3$ (*JCPDS-004-0637*) phases are also detected in this mixture. Most signs of Na_2CO_3 and boehmite phases disappear with increasing milling times to 2 and/or 5 hours, whilst the signs of the hematite phase (Fe_2O_3) at ~24, 33.4 and 35.8º are observed in the 2 h and 5 h milled mixtures. Peak broadening, decreasing in the relative intensities and disappearing of the softer phases are observed with increasing milling times in the milled samples. Figure 1 shows that these effects play significant roles in the softer phases (low Mohs hardness), such as boehmite and sodium carbonate (Na_2CO_3), compared to harder phases, such as hematite.

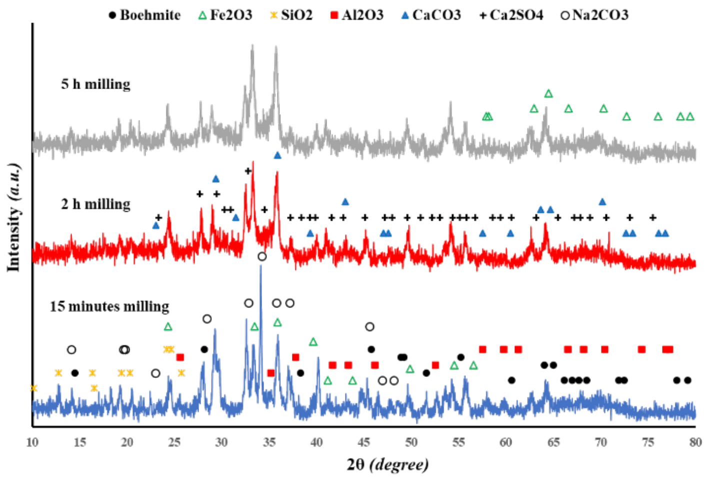

Figure 1. The XRD patterns of red mud-Na_2CO_3 mixtures after mechanical milling at different times

After roasting the milled mixtures of red mud-Na_2CO_3 at different temperatures (600-900 ºC) for 1 h under an air atmosphere, the roasted products were leached with hot water (80 ºC) for 60 minutes. Figure 2 shows the XRD graph of the alkaline roasted products in the 2 h milled mixture. The pattern of the dried solid residues after water leaching is also indicated in Figure 2. The signs of $NaAlO_2$ (*JCPDS-002-0999*), $NaAlSiO_4$ (*JCPDS-33-1203*) and $NaFeO_2$ (*JCPDS-30-1196*) phases are observed in the roasted sample at 800 ºC, and the signs of these phases are overlapped at ~21, 33 and 34º. The disappearance of the signs of $NaAlO_2$ phase and the phase changes in the ranges of 30-35º in the XRD graph of solid residues (Figure 2) indicate that the sodium aluminate phase ($NaAlO_2$) can be dissolved in hot water (80 ºC). The trace of the hematite phase at 33.5º and the signs of Ca_2SiO_4 and $CaCO_3$ phases indicate that these phases are not soluble in hot water. The previous research indicated that the acid washing (with HCl) process is not useful for separation of iron oxide from aluminum oxide (Al_2O_3) in the red mud sample [11]. The results of this work show that the water-soluble phase, such as $NaAlO_2$, occurs in the alkaline roasting of red mud. This phase can be dissolved in hot water and separated from the iron oxide impurities of red mud.

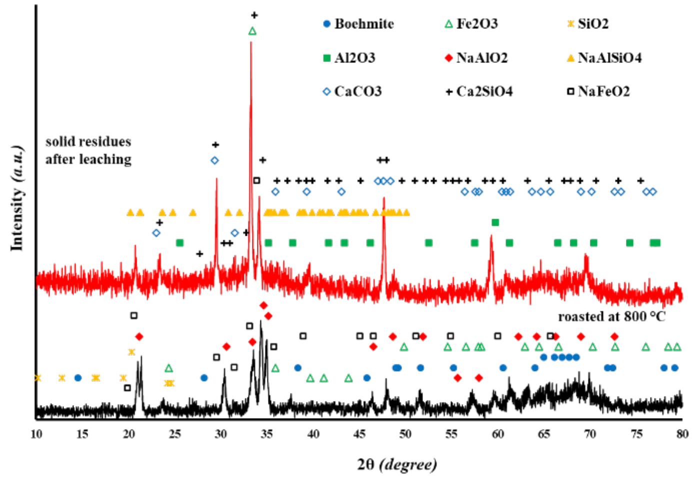

Figure 2. The XRD patterns of alkaline roasted products at 800 ºC and solid residues after leaching in hot water (80 ºC) in the 2 h milled mixture of red mud-Na_2CO_3

CONCLUSIONS

The chemical analysis using XRF and ICP-OES methods showed the high content of iron oxide (i.e., Fe) in the red mud sample. The high amount of pH in the red mud is related to the presence of high amounts of alkaline oxides such as Na_2O and CaO. The mass loss of red mud from STA analysis (TGA results) was about 13.25% at 900 ºC whilst in the red mud-Na_2CO_3 mixtures, it was in the range of 23-26%. The peak broadening of the softer phases, such as boehmite, and the disappearance of the sodium carbonate (Na_2CO_3) were observed with increasing the milling time. The alkaline roasting process occurred after heating the milled mixtures, and the signs of $NaAlO_2$, $NaAlSiO_4$ and $NaFeO_2$ phases were detected in the roasted samples. The leaching of the roasted samples with hot water (80 ºC) for 60 minutes was useful to dissolve the sodium aluminate ($NaAlO_2$) phase, and the signs of this phase were not detected in the solid residues of the leached samples. The minor traces of iron concentration (Fe) in the leached solutions indicated that the alkaline roasting of red mud with Na_2CO_3 prevents iron compounds (such as iron oxide) from dissolving in hot water. Therefore, it is possible to separate the aluminium content of red mud from the other impurities (such as iron oxide) by the alkaline roasting process.

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