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Alumina Extraction by Lime-soda Sinter Process from Low-grade Bauxite Soil of Semirom Mine

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Abstract

As the mass ratio of alumina to silica (A/S ratio) in bauxite decreases, the cost of alumina production by the Bayer process sharply increases. With the increasingly fierce competition in the alumina industry and the gradual reduction in bauxite grade, when the A/S ratio drops to 3-4, the Bayer process is challenging to meet the market competition and production requirements. In such cases, the alumina production by sintering method has a vast development prospect and application potential for low-grade bauxite ores. The low A/S ratio and the high iron oxide content are the difficulties in the alumina production by the sintering process. This work adopts the lime-soda sinter process for extracting alumina from bauxite samples (A/S ratio = 1.34 and 20.80% Fe2O3) of the Semirom mine in Iran. The effects of sintering parameters are investigated. The maximum alumina extraction (88%) was obtained by a CaO/SiO2 molar ratio of 1.2, Na2O/Al2O3 molar ratio of 0.9, and sintering temperature at 1250 °C for 80 min. Also 83% of alumina is extracted by decreasing the N/A ratio to 0.66, to decrease the sodium carbonate consumption for a more economical process. The sintered materials are leached with sodium carbonate solution, and aluminum hydroxide [Al (OH)3] is precipitated. Finally, pure alumina (Al2O3) is obtained with a purity of 98 % after calcination at 1200 °C for 2 hours.

1. Introduction

Alumina is an important industrial raw material that has been widely demanded in many advanced technological applications, and is the primary source of aluminum metal [1]. Bauxite is the primary raw material of alumina among all natural mineral resources. The rapid growth of the global alumina demands with the diminishing of the high-grade bauxite resources has motivated new strategies for exploiting the global low-grade aluminum-bearing mineral deposits. These targeted deposits have become interesting as potential substitutes in alumina production [2]. The most common and widely distributed low-grade alumina resources worldwide include clay minerals [3], red mud [4], and solid wastes such as coal fly ash [5 &6]. Low-grade bauxites usually consist of a large quantity of kaolinite in company with hematite, quartz, and anatase. Among the different clays, kaolinite seems to be a more attractive candidate for alumina production and an aluminum resource alternative to bauxite due to its high aluminum content (20.90%). Kaolinite is a two-layer silicate mineral with the representative chemical formula $Al_2O_3.2SiO_2.2H_2O$ [7 & 8].

The widely used metallurgical processing methods in the alumina production are the Bayer and sintering processes. The Bayer process had not proven to be efficient with using the low grades of aluminum-bearing raw materials [9]. The low-grade bauxites should be pretreated to feed the conventional Bayer process [10 &11], while the sintering process is the most suitable in case of using such low grades. The sintering method is represented by two main types: lime-sinter and lime-soda sinter processes [5], [11-15].

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In the lime-soda sintering process, the purpose of sintering is to make the Al_2O_3, Fe_2O_3, SiO_2, and TiO_2 content of the bauxite, ultimately generating $Na_2O{\cdot}Al_2O_3$, $Na_2O{\cdot}Fe_2O_3$, $2CaO{\cdot}SiO_2$, and $CaO{\cdot}TiO_2$ at the appropriate sintering temperature. The addition of Na_2O-containing compounds to the lime sinter process decrease CaO addition and increase the alumina leaching property [15]. In principle, the highest leaching rate of Al_2O_3 and Na_2O can be obtained when the clinker meets the saturated formula. Some insoluble solid solutions $nNa_2O{\cdot}mAl_2O_3{\cdot}Fe_2O_3$ will form with low alkali formula, while the high alkali formula will generate some insoluble ternary compounds $nNa_2O{\cdot}mCaO{\cdot}pSiO_2$. In the low calcium formula, the insoluble $Na_2O{\cdot}Al_2O_3{\cdot}2SiO_2$ is formed in the clinker, and the high calcium formula may produce $4CaO{\cdot}Al_2O_3{\cdot}Fe_2O_3$ and free CaO. All of the above factors will lead to the loss of Al_2O_3 and Na_2O [8], [16- 21].

In general, the following main reactions can occurs in the sintering process [17 & 23]:

1. Al_2O_3 reacts with Na_2CO_3 to form sodium aluminate
2. SiO_2 reacts with $CaCO_3$ to produce dicalcium silicate
3. Fe_2O_3 reacts with Na_2CO_3 to form sodium ferrite
4. Fe_2O_3 reacts with $CaCO_3$ to form calcium ferrite or dicalcium ferrite
5. TiO_2 reacts with $CaCO_3$ to produce calcium titanate

There are many factors affecting the quality of clinker including the calcium ratio CaO/SiO_2 (C/S), the alkali ratio Na_2O/Al_2O_3 (N/A), the $Na_2O{\cdot}Fe_2O_3/CaO_2{\cdot}Fe_2O_3$ ratio (NF/C_2F), the sintering temperature, and the sintering time. The sintering temperature depends on the composition and proportion of raw materials, and if the compositions of raw materials change, the sintering temperature will change accordingly. The benefit of charcoal as a thermochemical fluxing agent in the lime-sintering process at 1260–1360 °C was reported [23].

Al-Ajeel et al. (2014) studied the lime-sinter method for recovering alumina from Iraqi kaolin [24]. It was noted that the conversion of silica content into dicalcium silicate was of great importance for self-powdering of the sintered material. The favorable temperature for the sintering of kaolin and limestone were 1350 °C for 1 h [24]. The lime-soda sinter process was also employed to extract alumina from a colored kaolinite ore, and the results showed that the optimum parameters for the sintering operation were CaO/SiO_2 molar ratio of 2.2, Na_2O/Al_2O_3 molar ratio of 1.2, and sintering temperature at 1213 °C for 90 min [9].

This work adopted the lime-soda sinter process for extracting alumina from low-grade bauxite samples of the Semirom mine in Iran. The optimum parameters for the sintering operation were studied. The sintered materials were leached with sodium carbonate solution, and sodium aluminate solution was obtained. By bubbling carbon dioxide gas into this extract solution, aluminum hydroxide [Al $(OH)_3$] was precipitated, and after calcination at 1200 °C for 2 hours, the alumina phase (Al_2O_3) was obtained with a purity of 98 %. The study tried decrease the N/A ratio due to the high price of sodium carbonate and its removal problems after alumina extraction.

2. Materials and Methods

2.1. Materials

The Semirom Refractory Mining Company supplied the raw material samples used in this work. The bauxite sample and limestone were brought from the Semirom mines. The chemical composition of the raw materials shown in Table 1 indicated the presence of high Al_2O_3 and SiO_2. The semi-quantitative X-ray diffraction analysis of the bauxite sample confirmed the presence of kaolinite (~52%), hematite (~21%), boehmite (~19%), cristobalite (~3%), and anatase (~2%). The analytical grade sodium carbonate (Na_2CO_3) was used in the sintering and leaching processes.

Table 1. Chemical composition of raw materials.

	Chemical Composition (% weight)								
Material	**SiO_2 (%)**	**Fe_2O_3 (%)**	**Al_2O_3 (%)**	**TiO_2 (%)**	**CaO (%)**	**MgO (%)**	**Na_2O (%)**	**L.O.I (%)**	**Total (%)**
Bauxite	27.50	20.80	37	1.80	< 0.50	< 0.50	< 0.30	11.00	99.40
Limestone	1.03	0.14	0.93	0.04	55.50	0.37	< 0.10	41.70	99.81

2.2. Analytical methods
2.2.1. Scanning electron microscope (SEM) analysis

The morphological characteristics of extracted alumina samples were studied with a scanning electron microscope (SEM). The analytical sample was cleaned with water, ethanol, and acetone sequentially, and then dried with hot air. After these pre-treatments, the prepared sample was mounted onto a monocrystalline silicon holder, coated with gold, and analyzed with an FEI Quanta 200 scanning electron microscope from the FEI Company.

2.2.2. X-ray fluorescence (XRF) analysis

XRF analysis was used to estimate the chemical composition in samples of raw materials and extracted alumina. The X-ray fluorescence used was a Magix model manufactured by the Philips Company.

2.2.3. X-ray diffraction (XRD) analysis

XRD analysis was conducted using Cu Kα radiation with 40 kV and 30 mA scan rate (in 1s). The X-ray diffractometer used was an Xpert model manufactured by the PANalytical Company.

2.3. Processing procedure
2.3.1. Sintering

The raw material for the bauxite sintering process was prepared in two forms, powder, and molded cylinder (Figure 1). The bauxite and limestone samples were crushed by a laboratory jaw crusher to pass 1 mm, then ground by a laboratory ball mill to a particle size of -100 µ. The separation of the proper size was carried out using a mechanical sieve shaker. The ground raw material samples of bauxite and limestone, plus the soda ash, were well-mixed in a pre-determined proportion using a drum. The powder sample was prepared by mixing lime, soda, and Semirom bauxite with known C/S ($n(Ca)/n(SiO_2+TiO_2)$) and N/A ($n(NaO)/ n(Al_2O_3)$) molar ratios. The molded sample was prepared by mixing the powder sample with a certain amount of water to form mud, pressing it into a cylinder, and drying it in an oven to remove moisture. These samples were sintered in a furnace for different durations.

Figure 1. Raw powder (left) and molded cylinders (right).

2.3.1. Leaching and calcination

The dissolution solution contained five g/L soda and 15 g/L sodium hydroxide in water. The alumina extraction leaching experiments were conducted by the mass ratio of the sintered Semirom bauxite and the dissolution solution equal to 1/13 and at 85 °C and stirring at a speed of 300 rpm for 15 min by a magnetic stirrer. Leaching was done in a laboratory glass beaker under atmospheric pressure. By bubbling carbon dioxide gas into this solution, aluminum hydroxide [$Al(OH)_3$] was precipitated [25], and after calcination at 1200 °C for 2 hours, alumina (Al_2O_3) powder was obtained.

3. Results and Discussion
3.1. Effect of parameters on alumina extraction efficiency
3.1.1. Effect of sintering temperature

Besides, the sintering temperature had a substantial role in the completeness of the phase transformations; the self-disintegration efficiency and, as a result, the particle size distribution of the sinters were mentioned to be significantly influenced by the sintering temperature. It was reported that the self-disintegration process initiated at temperatures ranging from 1300 °C to 1400 °C given to complete breakdown of the sintered material-forming briquettes into very fine powders, the sintered briquettes did not undergo any self-disintegration at lower temperatures. The breakdown of the sintered material-forming briquettes into very fine powders is predicted to be attributed to the formation of the dicalcium silicate phase ($2CaO{\cdot}SiO_2$) at high temperature [16 & 17]. Therefore, in this work, the sintering process of clinkers was studied with temperatures ranging from 1000 to 1300 °C. When the sintering temperature was above 1250 °C, the material entered the molten phase, and practically low leaching occurred (Figure 2 and 3). It was mentioned in the literature that the clinker strength increases with the increase of temperature when the sintering temperature is higher than 1250 °C. In addition, when the sintering temperatures are 1000-1300 °C, the standard leaching rate and actual leaching rate of alumina gradually increase, while the standard leaching rate and actual leaching rate of soda first increase and then decrease with the increase of sintering temperature [5, 18 & 19]. As shown in Figure 3, when the sintering temperature is 1250 °C, the highest amount of alumina is extracted.

(a) (b) (c)

Figure 2. Images of molded cylinder sintered in different temperatures, a) 800, b) 1250, and c) 1300 °C.

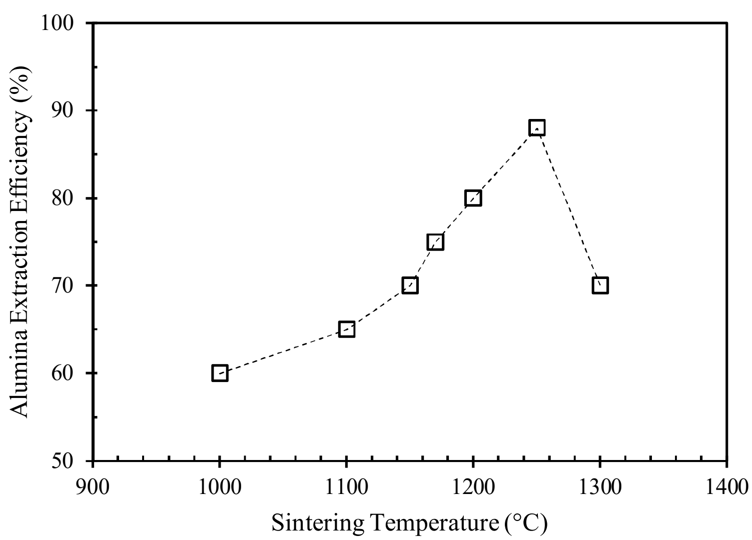

Figure 3. Effect of sintering temperature on alumina extraction efficiency
(N/A = 0.9, C/S = 1.2, Sintering time = 80 min, leaching temperature = 80 °C, leaching time = 15 min).

3.1.2. Effect of sintering time

As shown in Figure 4, when the sintering time is 80 min, the highest amount of alumina is extracted. When the sintering time is longer than 80 min, the molten phase is produced, similar to the effect of increasing temperature above 1250 °C (Figures 2 and 3). The stronger clinkers were produced as the sintering time increased from 30-80 min. The alumina leaching rate in clinker increases with time until the clinker strength increases sharply, and the soda leaching rate decreases [5].

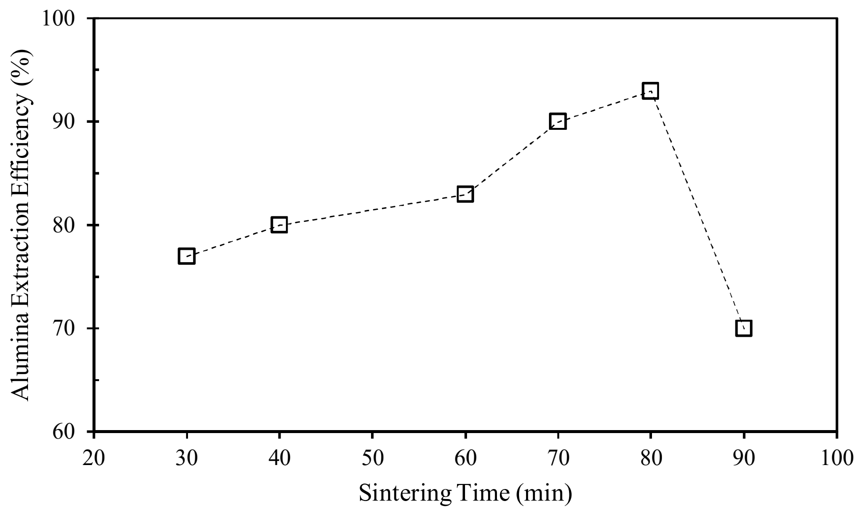

Figure 4. Effect of sintering time on alumina extraction efficiency. (N/A = 0.9, C/S = 1.2, sintering temperature = 1250 °C, leaching temperature = 80 °C, leaching time = 15 min).

3.1.3. Effect of C/S ratio on clinker sintering

The sintering process of clinkers with different C/S ratios ranging from 0.50-1.60 was studied. The sintering conditions are as follows: The N/A ratio of 0.9, the sintering temperature of 1250 °C, and the residence time of 80 min. The strengths of sintered clinker under different C/S ratios are kept at the same level. The alumina leaching rate first increases and then decreases with the increase of the C/S ratio, and the maximum is 88% when the C/S ratio is 1.2 (Figure 5). As shown in Figure 3, when the C/S ratio is 1.2, the highest amount of alumina is extracted. A low calcium ratio favored the formation of insoluble $Na_2O{\cdot}Al_2O_3{\cdot}2SiO_2$, while a high calcium ratio favored the formation of insoluble $4CaO{\cdot}Al_2O_3{\cdot}Fe_2O_3$. In both cases, the losses of Al_2O_3 and Na_2O occur [5 & 26].

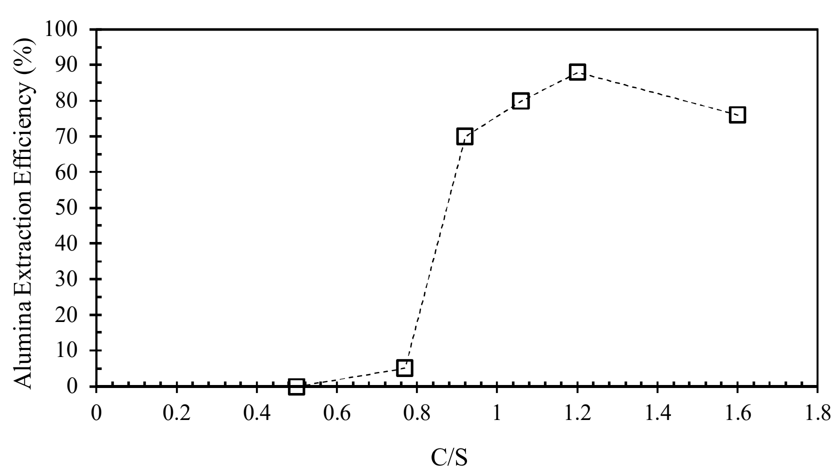

Figure 5. Effect of C/S on alumina extraction efficiency. (N/A = 0.9, sintering temperature = 1250 °C, sintering time = 80 min, leaching temperature = 80 °C, leaching time = 15 min).

3.1.4. Effect of alkali ratio

The sintering process of clinkers with different N/A ratios ranging from 0.50~0.95 was studied. The sintering conditions were the C/S ratio of 1.2, the sintering temperature of 1250 °C, and the residence time of 80 min. The results indicate that amount of liquid phase generated in the sintering process of raw materials with different N/A ratios is the same when other factors are fixed. Alumina leaching first increases and then decreases with the increase of the N/A ratio, and the maximum alumina leaching rate is 88% when the N/A ratio is 0.9. However, the soda leaching rate decreases with the further increase of the N/A ratio. As shown in Figure 6, when the N/A ratio is 0.9, the highest amount of alumina is extracted. Nevertheless, in this work, by reducing the N/A ratio to 0.66, an efficiency of 83 was achieved. It is mentioned in the literature that insoluble glassy $nNa_2O·mAl_2O_2$ was produced at a low alkali ratio because Na_2O was not enough to react fully with Al_2O_3 and Fe_2O_3 to form soluble $Na_2O·Al_2O_3$ and $Na_2O·Fe_2O_3$. Also the formation of calcium aluminates is controlled by the amount of Na_2O[21]. On the other hand, insoluble $nNa_2O·mCaO·pSiO_2$ was produced when a high alkali ratio was adopted. All these insoluble materials would contribute to the losses of Na_2O and Al_2O_3 [5, 26, 27]. It was reported that the amount of Na_2O has a vital influence on the transition and stability of dicalcium silicate during the sintering process [28].

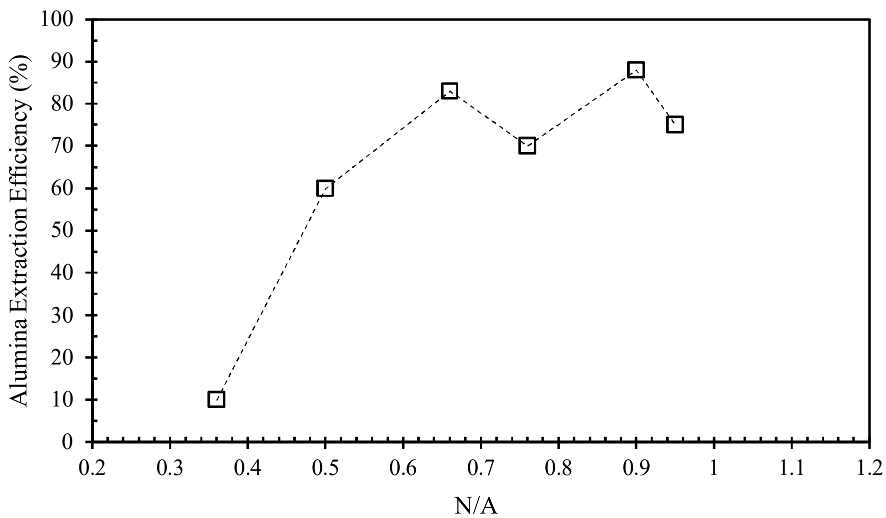

Figure 6. Effect of N/A on alumina extraction efficiency.
(C/S = 1.2, sintering temperature = 1250 °C, sintering time = 80 min, leaching temperature = 80 °C, leaching time = 15 min).

3.1.5 Effect of temperature on leaching performance

The temperature was the most critical factor affecting alumina dissolution from the sintered Semirom bauxite. As shown in Figure 7, alumina dissolution was accelerated at 60-80 °C. Raising dissolution temperature improved alumina extraction efficiency. At the same time, it also accelerated side reactions, which decreased the yield of alumina dissolution. At higher dissolution temperatures, the high amount of calcium silicate become more soluble and reacts with sodium aluminate and transfer aluminum oxide into insoluble calcium aluminosilicate hydrate ($3CaO·Al_2O_3·xSiO_2·yH_2O$). The formation of calcium aluminosilicate along with the precipitation of sodium silicate hydrate ($Na_2O·Al_2O_3·2SiO_2·nNaAl\text{-}(OH)_4·xH_2O$) led to significant loss of alumina [5 & 26].

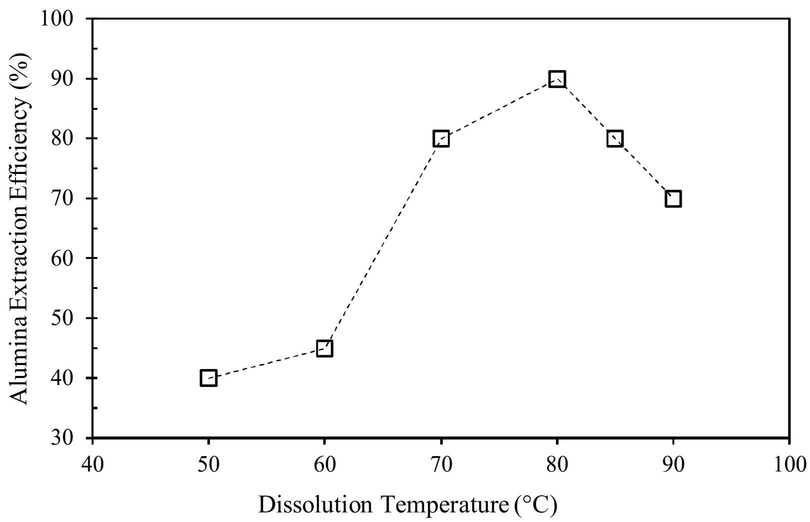

Figure 7. Effect of dissolution temperature on alumina extraction efficiency. (N/A = 0.9, C/S = 1.2, sintering temperature = 1250 °C, sintering time = 80 min, leaching time = 15 min).

3.2. Characterization of produced alumina

The sintered materials (CaO/SiO_2 molar ratio of 1.2, Na_2O/Al_2O_3 molar ratio of 0.66, sintering temperature at 1250 °C for 80 min) were leached with sodium carbonate solution, and sodium aluminate solution was obtained. By bubbling carbon dioxide gas into this extract solution aluminum hydroxide [Al $(OH)_3$] has been precipitated and on calcination at 1200 °C for 2 hours, alumina powder was obtained with purity of 98%. Table 2 shows the XRF analysis of the produced alumina powder. The XRF data shows a purity of 98%, and the presence of negligible calcium and sodium oxide impurities are inevitable. Figure 8 shows the XRD pattern of the product which confirms the production of pure tetragonal alumina crystals. Figure 9 presents the SEM images of the powder and the point and map EDS analysis, which confirms alumina particles.

Table 2. XRF analysis of produced aluminum oxide.

Material	SiO_2 (%)	Fe_2O_3 (%)	Al_2O_3 (%)	TiO_2 (%)	CaO (%)	MgO (%)	Na_2O (%)	L.O.I (%)	Total (%)
	0.80	0.02	98.00	-	0.06	-	0.70	0.30	99.88

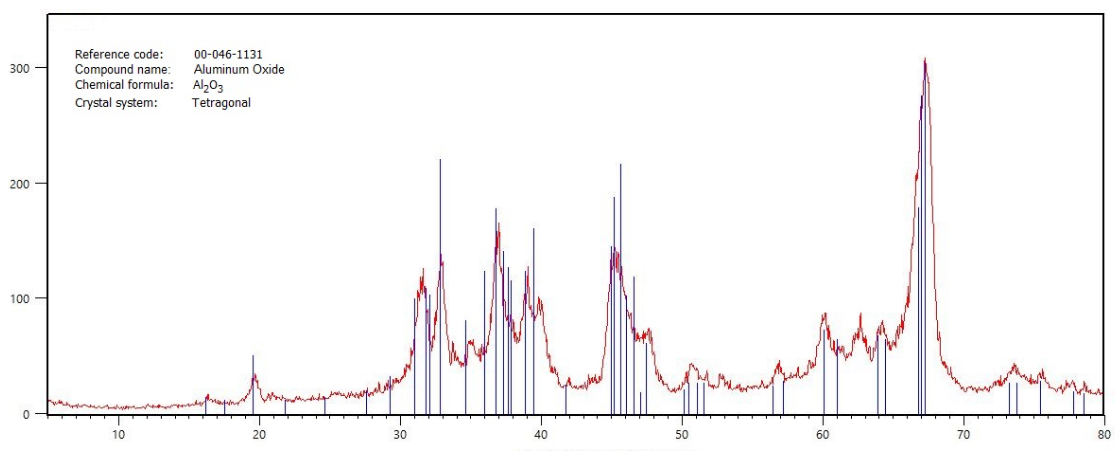

Figure 8. XRD patterns of produced alumina powder.

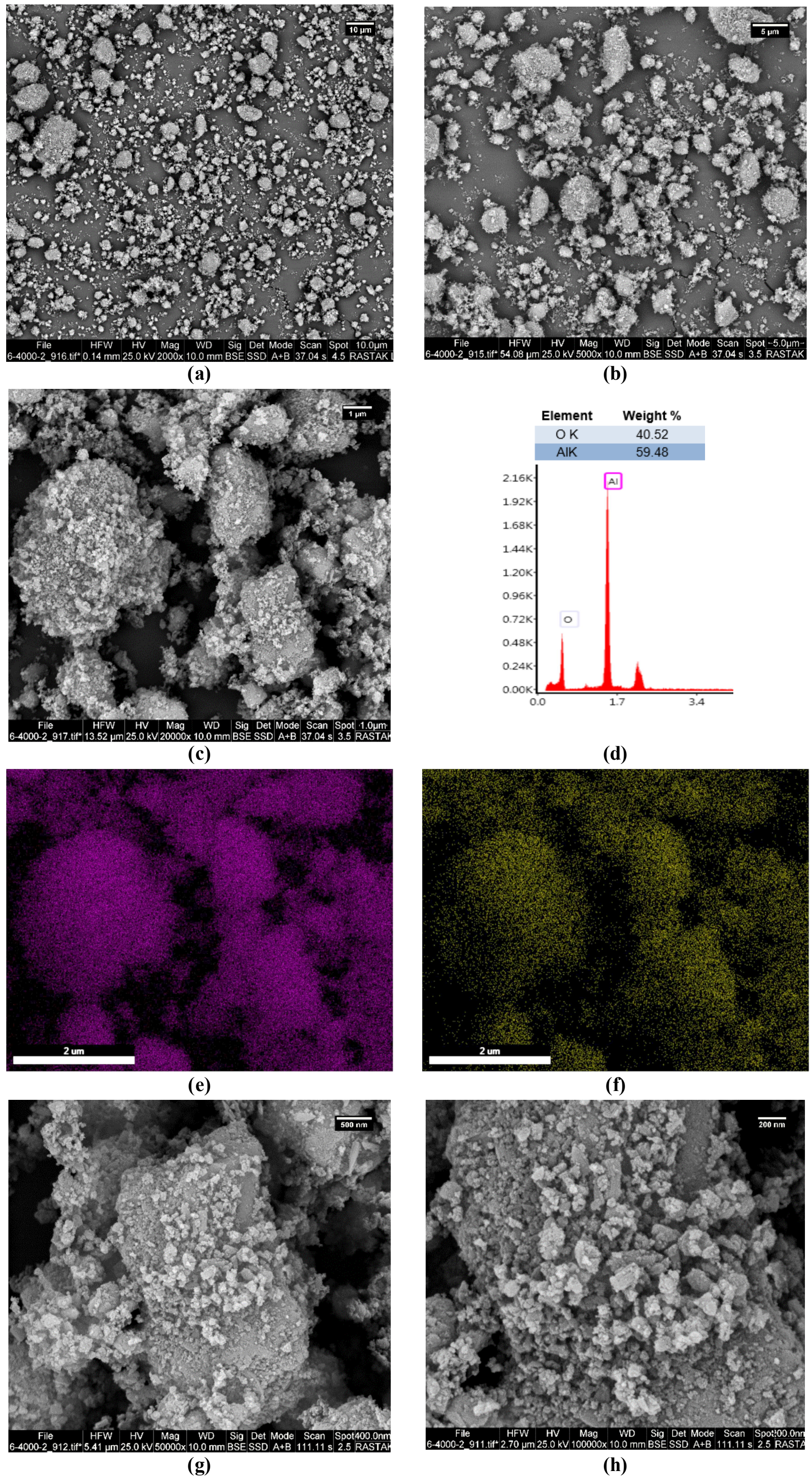

Figure 9. SEM Images of produced alumina powder, a,b,c,g and h are in 2000x, 5000x, 20000x, 50000x and 100000x magnification, d, e and f are map elemental EDS analysis, Al and O overlay plots for the c area.

4. Conclusions

Semirom kaolinite was used as a bauxite substitute in the lime-soda sintering process for alumina production. The clinker obtained by the sintering operation at CaO/SiO_2 molar ratio of 1.2, Na_2O/Al_2O_3 molar ratio of 0.66, and the sintering temperature at 1250 °C for 80 min. The sintered materials were leached with sodium carbonate solution, and sodium aluminate solution was obtained. By bubbling carbon dioxide gas into this extract solution, aluminum hydroxide [Al $(OH)_3$] was precipitated, and on calcination at 1200 °C for 2 hours, the alumina powder was obtained with purity of 98%. According to the obtained results, it was found that by reducing the amount of N/A to 0.66, the process was more cost-effective due to the high price of sodium carbonate, although the recovery of alumina decreased slightly (5%). Therefore, the extraction of pure alumina by the lime-soda sintering method from the low-grade bauxite ore of Semirom mine containing high amounts of hematite and kaolinite was confirmed as a substitution of the conventional Bayer method.

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استحصال آلومینا با استفاده از فرایند زینترینگ آهک- سود از خاک کم عیار بوکسیتی معدن سمیرم

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چکیده:

با کاهش نسبت آلومینا به سیلیس (نسبت A/S) در بوکسیت، هزینه تولید آلومینا با فرآیند بایر به شدت افزایش می‌یابد. بنابراین با کاهش تدریجی عیار بوکسیت و در مواردی که نسبت A/S به حدود ۳ تا ۴ کاهش می‌یابد، استفاده از این روش چالش برانگیز است. در چنین مواردی، تولید آلومینا با استفاده از فرایند زینترینگ، دارای پتانسیل کاربرد و چشم انداز توسعه برای بوکسیت‌های کم عیار است. از طرفی نسبت A/S پایین و محتوای اکسید آهن زیاد از مشکلات تولید آلومینا توسط فرآیند زینترینگ است. در این تحقیق، از فرآیند زینترینگ آهک- سود برای استحصال آلومینا از نمونه‌های بوکسیت کم عیار معدن سمیرم (۱٫۳۴ = A/S و % ۲۰٫۸۰ = Fe_2O_3) استفاده شده است. تاثیر عوامل موثر بر زینترینگ بررسی شد. حداکثر استخراج آلومینا (۸۸٪) با نسبت مولی CaO/SiO_2 ۱٫۲، نسبت مولی Na_2O/Al_2O_3 ۰٫۹ و دمای زینترینگ ۱۲۵۰ درجه سانتی‌گراد در مدت زمان ۸۰ دقیقه به دست آمد. همچنین مشخص شد که می‌توان به بازیابی استحصال ۸۳ درصدی آلومینا حتی با کاهش نسبت N/A به ۰٫۶۶ رسیده و درنتیجه با کاهش مصرف کربنات سدیم به فرایند مقرون به صرفه‌تری دست یافت. مواد زینتر شده با محلول کربنات سدیم شستشو شدند و هیدروکسید آلومینیوم [$Al(OH)_3$] رسوب داده شد. در نهایت آلومینای خالص (Al_2O_3) با خلوص ۹۸ درصد پس از کلسینه کردن در دمای ۱۲۰۰ درجه سانتی گراد به مدت ۲ ساعت به دست آمد.

کلمات کلیدی: بوکسیت، کائولینیت، آلومینا، فرآیند زینترینگ آهک- سود، معدن سمیرم.