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Investigation of parameters affecting desilication of diasporic bauxite in Jajarm mine by thermo-chemical treatment

M. Rezaee Rad[1]*, S. Shahhoseini[2], M. Janfada[1], H.A. Mirzaee[3] and P. Kelidari[1]

1. Jajarm Alumina Complex, Jajarm, Iran
2. School of Chemical Engineering, Iran University of Science and Technology, Tehran, Iran
3. School of Mining Engineering, College of Engineering, University of Tehran, Tehran, Iran

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**Corresponding author: mohammadrad.r@gmail.com (M. Rezaee Rad).*

Abstract

Low grade diasporic bauxite in the Jajarm mine with an A/S (Al_2O_3/SiO_2) ratio of 2.3 is not usable in the Bayer process at Jajarm Alumina Complex. Due to the severe interlocking effect between the diaspore and aluminosilicate minerals (Chamosite and Kaolinite) and iron-containing minerals in a microcrystal matrix, the thermo-chemical treatment, which is independent from micro-mineralogy, was chosen for bauxite desilication. Five parameters affecting the process and their interactions were investigated using the Taguchi experimental design method. The results obtained showed that there was an interaction between the furnace temperature and the leaching time. Moreover, the optimum values for the parameters involved in the thermo-chemical treatment were determined to be a furnace temperature of 950 °C, a furnace residence time of 90 min, a leaching agent (soda) concentration of 150 g L^{-1}, and a leaching time of 120 min, where the solid content (in leaching) had no effect. Moreover, a model was proposed using the Dx7 software to predict the A/S ratio. The ratio was predicted to be 7.52 at the optimum conditions, whereas in the experiments carried out under the same conditions, it was obtained to be 6.96 ± 0.2, which means a 59% decrease in silica and an increase in the A/S ratio of up to 3 times with 80% weight recovery.

Keywords: *Interaction Effect, Bayer Process, Desilication, Bauxite, Thermo-Chemical.*

1. Introduction

Increasing the demand for bauxite as the main resource for aluminum production has led to a considerable growth of its global production capacity in the recent years [1]. The most important and problematic minerals in the bauxite deposit are those that contain active silica, which have very undesirable effects on the Bayer process efficiency so that for 1 ton of silica in the composition of clay minerals, 1 ton of soda has to be consumed [2]. Bauxites with an A/S ratio of less than 6.25 and a silica content higher than 8% Wt. are known as high silica bauxites, which are non-economical for the Bayer process [3]. The Bayer process is simple at a glance, although, in practice, bauxite imposes very difficult chemical conditions in the process owing to its heterogeneous and complicated nature. Bauxite impurities such as organic carbons and silica, and their reactions and precipitation in the solution reduce the efficiency of bauxite refinery. There are many ways to reduce the damages on the process efficiency resulting from impurities. However, these methods are expensive and increase the complexity of the plant flow sheet [4]. Bauxite upgrading methods such as the screening/washing, floatation, gravity separation, magnetic separation, and thermo-chemical method depend on the bauxite recognition from a microscopic viewpoint (texture, liberation degree, and mineralogical association type) [3]. In high silica bauxites, whose alumina minerals have a low liberation degree due to severe interlocking between the minerals, the thermo-chemical method has a considerable effect on the silica removal as a pre-treatment procedure [5]. This method has been tested by Saint Petersburg's VAMI Complex in a pilot scale [6]. The aim of bauxite heating is the formation of maximum amorphous silica, while formation of the mullite mineral is kept at its minimum value. Mullite does not dissolve in soda, and causes lots of difficulties for the Bayer process [7]. Kaolinite is an important alumosilicate material since it is the active silica in bauxite. Several studies have been conducted to investigate the thermal transformation of kaolinite. Based on these investigations, the kaolinite chemical transformation has been identified as follows [8]:

1. Dehydroxylation at a temperature of about 420-660 °C results in the formation of metakaolinite ($Al_2O_3.2SiO_2$):

$$Al_2O_3.2SiO_2.nH_2O \rightarrow Al_2O_3.2SiO_2 + nH_2O$$

2. Decomposition of metakaolinite at a temperature above 980 °C results in the formation of amorphous SiO_2 and γ-Al_2O_3:

$$Al_2O_3.2SiO_2 \rightarrow \gamma Al_2O_3 + 2SiO_2 \text{(Amorphous)}$$

3. Recrystallization of the amorphous substances forms mullite at above 1100 °C:

$$\gamma Al_2O_3 + 2SiO_2 \text{(Amorphous)} \rightarrow 3Al_2O_3.2SiO_2$$

The formation of mullite through the alumina-silica reaction at a temperature range of 1200-1400 °C, and then transformation of mullite to a glassy phase at a temperature around 1900 °C has also been reported [9]. Amorphous SiO_2 is easily soluble in soda (NaOH). Increasing the soda concentration not only improves the amount of silica dissolution but also extracts a little bit of Al_2O_3 [10, 11].

This study aims to investigate the parameters affecting the thermo-chemical process with the purpose of reduction of silica in the Jajarm bauxite mine using an experimental design method. The Taguchi model is suitable for the processes that are in early stages of designing, with high number of parameters in different levels [12, 13]. This method inspects the whole space of the parameters with a few numbers of the experiments using a particular design of orthogonal arrays, and determines the effective parameters and their optimum values causing considerable reduction in the optimization cost and time [14, 15]. Therefore, in this research work, the Taguchi method was chosen to design the experiments for considering several parameters and their levels.

2. Materials and method

2.1. Mineralogy of low-grade Jajarm bauxite

Low-grade diasporic bauxite is not a usable material in the Jajarm mine since it has 38.09% Al_2O_3 and16.61% SiO_2, resulting in an A/S (Al_2O_3/SiO_2) ratio of 2.29 (shown in Table 1).

Hematite, which contains 14.1% iron, is the main iron-containing mineral of bauxite. Aluminosilicate-containing minerals are mainly chamosite (13.4%) and kaolinite (18.4%), as shown in Table 2.

A huge mass of the Jajarm mine consists of high silica bauxite, in which diaspore and aluminosilicates (chamosite, kaolinite) are finely interlocked so that they are surrounded by iron-containing minerals, especially hematite. These blocks often have oolitic-concretion texture, and, in some cases, nodular texture is also found (Figure1) [16].

Table 1. Chemical composition of Jajarm low-grade bauxite (analyzed by XRF).

Al_2O_3	SiO_2	Fe_2O_3	TiO_2	CaO	MgO	K_2O	Na_2O	L.O.I
38.09	16.61	22.75	4.6	1.1	0.43	0.35	1.24	14.83

Table 2. Minerals of Jajarm low-grade bauxite (analyzed by XRD).

Diaspore	Hematite	Goethite	Anatase	Calcite	Cancrinite	Iilite	Chamosite	Kaolinite
33.2	14.1	3.6	4.6	1.8	6.8	4.1	13.4	18.4

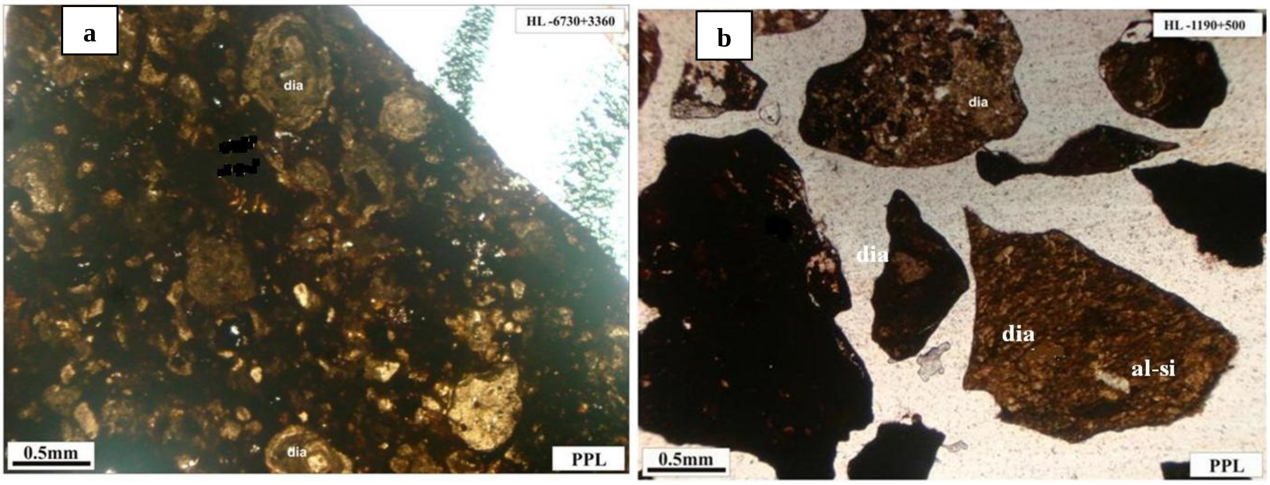

Figure 1. a) Diaspore (dia) oolitic texture b) Interlocking of diaspora (dia) with aluminosilicates (Al-Si).

2.2. Procedure in laboratory scale

Samples were taken from different places of a low-grade bauxite dump. They were then crushed until below 20 mm, and divided to several smaller samples. Calcinations were conducted on the smaller samples in an electrical furnace. Afterwards the cooled samples were ground to nearly 90 microns in a laboratory ball mill in dry mode, and prepared for leaching tests at 90 ^{0}C. Industrial-grade soda was used for all of the experiments as the leaching agent. Some parameters that could be more effective on the bauxite upgrading (decreasing silica-containing bauxite) by the thermo-chemical method were furnace temperature (A), furnace residence time for bauxite with 20 mm diameter (B), soda concentration (C), solid content in leaching (D), and leaching time (E), which were examined in three different levels corresponding to the L_{27} orthogonal array pattern requested by the Taguchi experimental design method (Table 3). In addition, the amount of bauxite SiO_2 content was chosen as the response for the DX7 software. The response with 95% confidence was analyzed to find the optimum value for each parameter.

Table 3. Parameters and their values corresponding to their levels.

Parameters		Levels		
		1	2	3
A	Furnace temperature (°C)	950	1000	1050
B	Furnace residence time (min)	30	60	90
C	Soda concentration (g L^{-1})	100	150	200
D	Solid content in leaching (g L^{-1})	300	350	400
E	Leaching time (min)	60	120	180

3. Results and discussion

The main purpose of conducting this study was to determine the parameters affecting the silica removal and their optimum value, in which the maximum yield for silica removal could be obtained. Table 4 shows the percentage of the bauxite SiO_2 content and the A/S (Al_2O_3/SiO_2) ratio in each experiment according to the L_{27} pattern. In Table 5, the results of the analysis of variance (ANOVA) using the Dx7 software are shown, where SiO_2% is the response with 95% confidence. Therefore, the P-value of each parameter had to be less than 0.05. The results obtained from the analysis indicated that the solid content (D) with a P-value of 0.5 could not be considered as a principle and influential parameter. In addition, only the interaction between the furnace temperature (A) and leaching time (E) had an impact on the response. As Figure 2 shows, two parameters showed interactions in a slight span.

Table 4. Experiments and responses (Bauxite SiO_2 content) according to Taguchi's L_{27} orthogonal array.

No	A	B	C	D	E	SiO_2	Module
1	950	30	100	300	60	10.99	3.53
2	950	30	100	300	120	9.86	4.15
3	950	30	100	300	180	12.11	3.39
4	950	60	150	350	60	7.84	5.28
5	950	60	150	350	120	7.1	6.18
6	950	60	150	350	180	7.73	5.54
7	950	90	200	400	60	7.5	6.1
8	950	90	200	400	120	6.24	6.79
9	950	90	200	400	180	7.71	5.92
10	1000	30	150	400	60	10.72	4.21
11	1000	30	150	400	120	9.97	4.26
12	1000	30	150	400	180	8.97	4.76
13	1000	60	200	300	60	9.3	4.78
14	1000	60	200	300	120	8.86	4.73
15	1000	60	200	300	180	9.46	4.28
16	1000	90	100	350	60	10.53	4.30
17	1000	90	100	350	120	9.74	4.69
18	1000	90	100	350	180	8.19	5.27
19	1050	30	200	350	60	11.69	3.57
20	1050	30	200	350	120	9.06	4.48
21	1050	30	200	350	180	9.95	3.99
22	1050	60	100	400	60	13.02	3.38
23	1050	60	100	400	120	12.33	3.46
24	1050	60	100	400	180	9.77	4.55
25	1050	90	150	300	60	10.28	4.75
26	1050	90	150	300	120	7.58	6.86
27	1050	90	150	300	180	7.09	6.84

Table 5. ANOVA results.

Source	Sum of Squares	Mean Square	F-value	P-value Prob>F
Model	64.33	6.43	6.84	0.0004
A- Furnace temperature	10.66	5.33	5.67	0.0138
B- Furnace residence time	19.06	9.53	10.13	0.0014
C- Soda concentration	24.38	12.19	12.97	0.0004
D- Solid content in leaching	1.24	0.62	0.66	0.5301
E- Leaching time	8.98	4.49	4.78	0.0236
AE	8.93	2.23	4.25	0.0186

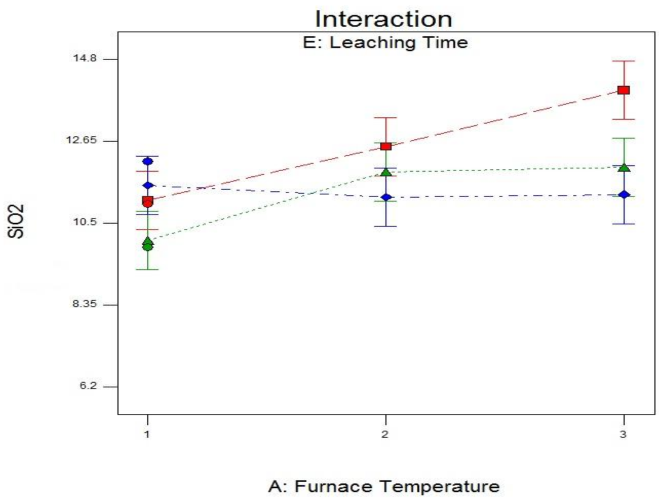

Figure 2. Interaction between furnace temperature (A) and leaching time (E) for SiO_2 response.

3.1. Validation of final equation of model

After selecting the effective parameters, the software recommended an equation, which could be used to calculate the bauxite SiO_2 content in a variety of the values for the parameters.

$$SiO_2 = 9.39 - 0.83A[1] + 0.13A[2] + 0.98B[1] + 0.098B[2] + 1.33C[1] - 0.81C[2] + 0.82E[1] - 0.42E[2]$$

Letters show the codes used for the parameters.

[ ] shows the number of levels.

At first, the software determines the statistical parameters (shown in Table 6) to assess the validation of the SiO_2 calculation equation that comes from a regression model.

Table 6. Parameters for validation of model.

Adequate precision	10.549
Pre-R squared	0.5383
Adj-R squared	0.7036
P-value	<0.0001

The model is valid under the conditions where the P-value is less than 0.05, the adequate precision value is more than 4, and the difference between adjusted R squared (Adj-R squared) and predicted R squared (Pre-R squared) is less than 0.2. The results obtained indicated that the proposed model was suitable under these conditions. The Dx7 software also considers three graphs called normal plot of residuals, residuals *vs.* predicted, and box cox in order to make sure that the chosen model is valid. As shown in Figure 3, the plots in a normal plot of residuals graphs are on a line but in residuals *vs.* predicted graph are scattered, and lambda location is also in the allowed district defined at box cox graph. Therefore, these results confirm the adequate precision of the model.

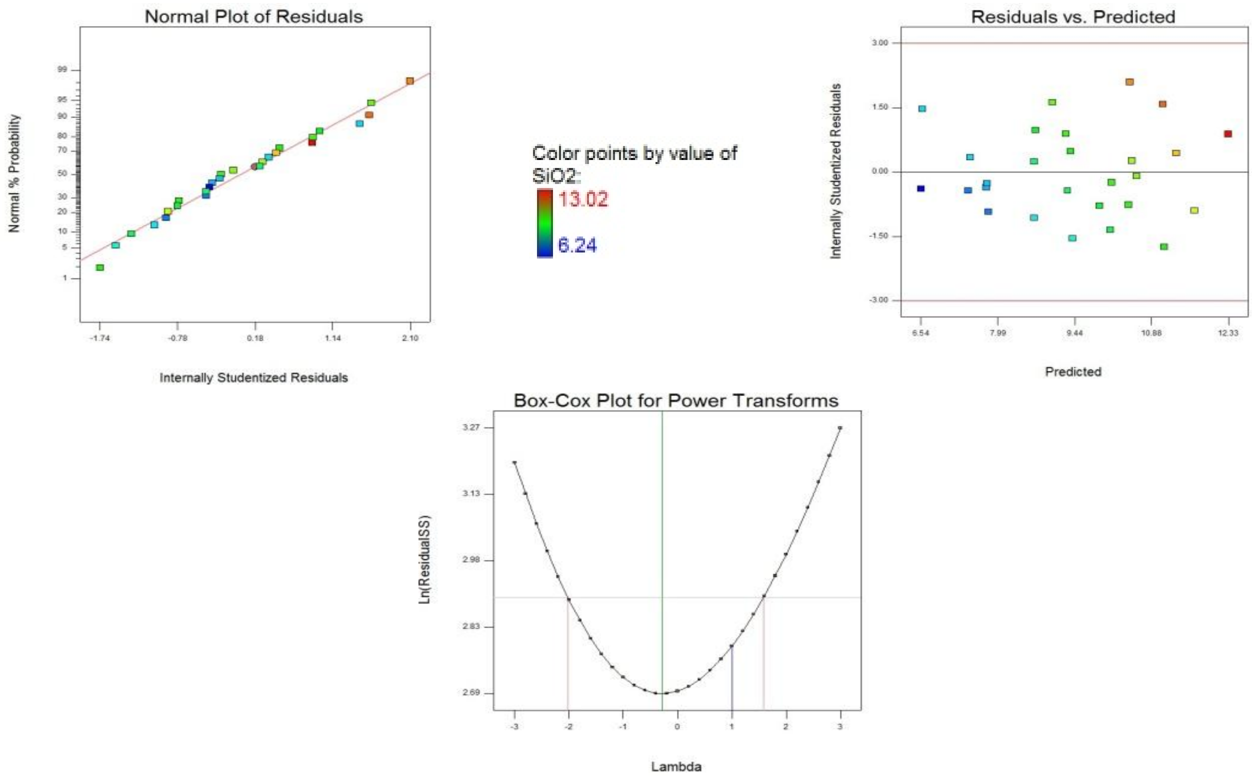

Figure 3. Graphs for validation of model.

3.2. Determination of optimum values for effective parameters

Based on the proposed equation, the graphs in Figure 4 show the trend of changing the SiO_2 content in bauxite. Clearly, increasing the furnace temperature causes the formation of mullite (aluminosilicate mineral), and SiO_2 remains in force without dissolution in soda (Figure 4-a). On the other hand, increasing the furnace residence time decreases the bauxite SiO_2 content, and improves dissolution (see Figure 4-b). Increasing the soda concentration and leaching time up to the second level (150 g L^{-1} and 120 min, respectively) results in desilication. However, more increase in the level has no effect (see Figures 4-c and 4-d).

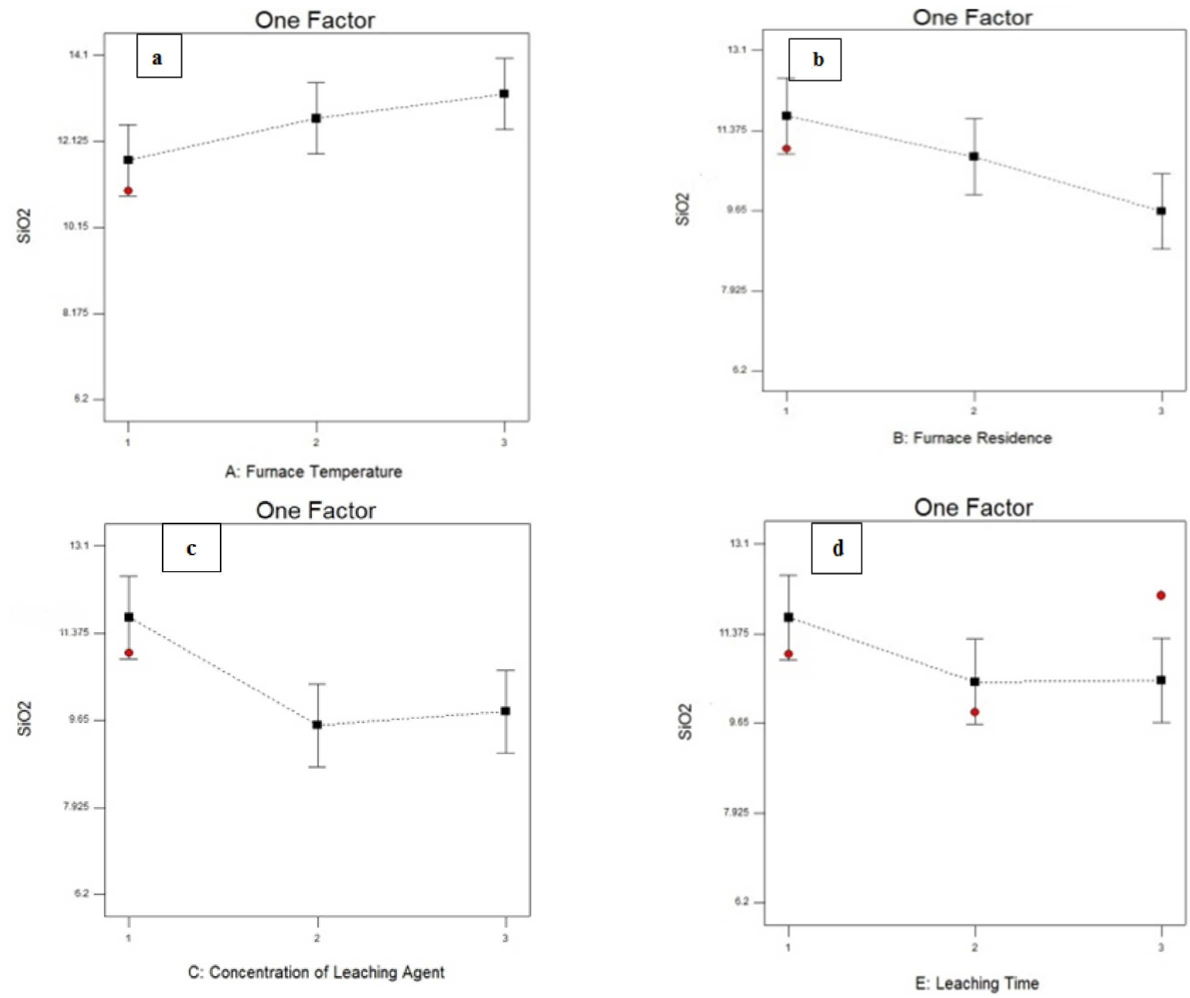

Figure 4. Trend of SiO_2 content in bauxite *vs* a) Furnace temperature b) Bauxite residence time in furnace temperature c) Soda concentration d) Leaching time.

With the purpose of upgrading the process, the optimal values for the parameters were determined using the software, where the SiO_2 content in bauxite was chosen to be the response. Table 7 shows the optimal values for the parameters to achieve the maximum desilication.

The optimization performed using the Dx7 software suggested that the A/S ratio for concentrated bauxite was 7.52. However, when these optimal conditions were applied in the Jajarm Alumina Complex laboratory, an A/S ratio of 6.96 ± 0.2 with around 6.8% of SiO_2 was obtained, i.e. the Jajarm low-grade bauxite was upgraded by the thermo-chemical method about 3 times, resulting in 80% weight recovery of usable bauxite, as shown in Table 8.

Table 7. Optimum amount of effective parameters for maximum desilication.

Parameters	(A) Furnace Temperature (°C)	(B) Furnace residence time (min)	(C) Soda concentration (g L^{-1})	(E) Leaching Time (min)
Level	1	3	2	2
Amount	950	90	150	120

Table 8. Beneficial optimization result by applying thermo-chemical process on Jajarm low-grade bauxite.

Component	Al_2O_3%	SiO_2%	A/S ratio
Low-grade bauxite	38.09	16.61	**2.29**
Prediction of A/S ratio	47.05	6.26	**7.52**
Actual A/S ratio after upgrading	47.32	6.8	**6.96**

4. Conclusions

Low-grade diasporic bauxite in the Jajarm mine was experimentally upgraded using a thermo-chemical method and applying the Taguchi's experimental design technique. The results obtained indicated that the solid content in leaching had a minimum effect on the thermo-chemical method. Surprisingly, there was an interaction between the furnace temperature and leaching time parameters. On the other hand, the optimum conditions were obtained as a furnace temperature of 950 °C, a furnace residence time of 90 min for bauxite (with a dimension below 20 mm), a soda concentration of 150 g L^{-1}, and a leaching time of 120 min. The optimal A/S ratio for bauxite was predicted to be from 2.29 to 7.52. The optimal conditions were then applied in practice, resulting in usable bauxite with an A/S ratio of 6.96 ± 0.15, which meant 59% SiO_2 removal and a 3 times upgrading with 80% Wt. recovery.

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بررسی پارامترهای مؤثر بر سیلیس زدایی بوکسیت دیاسپوری معدن جاجرم به روش ترموشیمی

محمد رضایی راد[۱*]، شاهرخ شاه‌حسینی[۲]، مهدی جانفدا[۱]، حسین‌علی میرزایی[۳] و پرویز کلیدری[۱]

۱- مجتمع آلومینای جاجرم، ایران

۲- دانشکده مهندسی شیمی، دانشگاه علم و صنعت، ایران

۳- دانشکده فنی و مهندسی، دانشکده معدن، دانشگاه تهران، ایران

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* نویسنده مسئول مکاتبات: mohammadrad.r@gmail.com

چکیده:

بوکسیت دیاسپوری کم عیار معدن جاجرم با مدول (Al_2O_3/SiO_2) ۲٫۳ غیر قابل استفاده در فرآیند بایر مجتمع آلومینای جاجرم است. به علت قفل‌شدگی شدید کانی دیاسپور با کانی‌های آلوموسیلیکات (شاموزیت و کائولینیت) و کانی آهن‌دار (هماتیت) در یک بستر ماتریکس ریز بلور، روش ترموشیمی که مستقل از میکرومینرالوژی سنگ است به منظور سیلیس زدایی از بوکسیت انتخاب شد. پنج پارامتر مؤثر و تأثیر متقابل بین آن‌ها توسط طراحی آزمایش تاگوچی و نرم‌افزار DX7 بررسی و ضمن تشخیص پدیده تأثیر متقابل بین دمای کوره و زمان لیچینگ، مقدار بهینه دمای کوره ۹۵۰ درجه سانتی‌گراد، زمان ماند بوکسیت با ابعاد کمتر از ۲۰ میلی‌متر در کوره ۹۰ دقیقه، غلظت عامل لیچینگ ۱۵۰ گرم بر لیتر، غلظت جامد ۳۰۰ گرم بر لیتر و زمان لیچینگ ۱۲۰ دقیقه در فرآیند ترموشیمی با هدف دستیابی به بیشترین راندمان کاهش سیلیس، تعیین شد. مدل پیشنهاد شده، مدول (Al_2O_3/SiO_2) بوکسیت پر عیار شده در شرایط بهینه را ۷٫۵۲ پیش‌بینی کرد، در حالی که در آزمایش‌های عملی با شرایط مشابه ۰٫۱۵ ± ۶٫۹۶ به دست آمد که بیانگر ۵۹٪ کاهش سیلیس و افزایش مدول تا ۳ برابر با بازیابی وزنی ۸۰٪ است.

کلمات کلیدی: تأثیر متقابل، فرآیند بایر، سیلیس زدایی، بوکسیت، ترموشیمی.