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Extracting Alumina from a Low-grade (Shale) Bauxite Ore using a Sintering Process with Lime-soda followed by Alkali Leaching

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Abstract

The global growth of aluminum demand with the modernization of our society has led to the interest in developing alternative methods to produce aluminum from non-bauxite and low-grade resources such as shale bauxites. For such reserves, the conventional Bayer process is challenging and is not efficient to extract aluminum, and the sintering process is known to be effective. Thus, this study aimed to scrutinize the technical feasibility of alumina extraction from an Iranian low-grade (shale) bauxite ore containing 36.22% Al2O3, 22.11% SiO2, 20.42% Fe2O3, 3.33% TiO2, and 3.13% CaO. In this regard, the sintering process with lime-soda followed by alkaline leaching was adopted to extract alumina, and response surface modeling was employed to assess the important parameters such as the sintering temperature, Na2O(caustic) concentration, CaO/SiO2 molar ratio, and Na2O/Al2O3 molar ratio. The findings indicated that the extraction rate improved by increasing the sintering temperature and CaO/SiO2 ratio and decreasing the Na2O(caustic) dose and Na2O/Al2O3 ratio. It was also found that the Na2O(caustic) concentration, sintering temperature, and interactive effect of Na2O(caustic) concentration with Na2O/Al2O3 ratio had the greatest influence on the extraction efficiency. The process optimization was conducted applying the desirability function approach, and more than 71% of Al2O3 was extracted at 1150 °C sintering temperature, 2.1 CaO/SiO2 molar ratio, 0.9 Na2O/Al2O3 molar ratio and 30 g/L Na2O(caustic) dose. Ultimately, it was concluded that a lime-soda sintering process at 1150 °C followed by one-step alkaline leaching with Na2O(caustic) at 90 °C could be metallurgically efficient for treating the low-grade (shale) bauxites.

1. Introduction

Aluminum is one of the most significant and plentiful non-ferrous metals in the earth's crust and is mostly in the forms of oxide and hydroxide. Aluminum and its compounds are widely applied in various industrial fields (such as packaging, aerospace, automotive, mineral processing, power transmission, building, and marine) owing to the high electrical and thermal conductivity attributes, high strength and hardness, good corrosion resistance, lightweight, and mechanical particularities [1-4]. It is also accepted that aluminum occupies the highest amount after iron and steel in terms of production and consumption [5]. The primary resource for extracting aluminum is bauxite, and currently, above 90% of the world's aluminum is obtained from bauxite [6]. However, other materials such as nepheline syenite, alunite, shale, red mud, coal fly ash, and some clays (like kaolinite) have been recognized as alternative non-bauxite sources containing aluminum [7, 8]. It is estimated that the aforementioned compounds contain ~25-40% Al_2O_3 values [8, 9]. In metallurgy and mineral processing industries, aluminum production from bauxite is usually performed by a consecutive two-step process. In the first step, pressure leaching of the mined bauxite with a concentrated sodium hydroxide (caustic) solution is conducted to produce

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aluminum oxide or alumina (Bayer process). The operating conditions (like temperature, leaching time, leaching agent concentration) are also highly dependent on the type of aluminum-bearing minerals (i.e. gibbsite, boehmite, and diaspore) and impurities in bauxite. In the second step, the Hall–Héroult process (electrolysis) is utilized for the electrolytic reduction of alumina to aluminum [10-11].

In the recent years, the rapid development of the aluminum industry, the increase in global demand for aluminum and its compounds, the increase in price, and the limited high-grade resources have resulted in scrutinizing the extraction of aluminum from low-grade sources and wastes. On the other hand, the Bayer process, which is the main and conventional technique for treating bauxite deposits worldwide, does not respond well to low-grade ores, so it is essential to investigate other methods. According to Smith [7] and Azof *et al.* [12], Al_2O_3/SiO_2 mass ratio lower than 6.25 and or silica content more than 8 wt% is called a low-grade bauxite ore and is not suitable for the Bayer process. In this regard, several different methods were developed and proposed to extract alumina from low-grade reserves and residues such as second Bayer, Pederson, reductive alkali roasting, sintering (soda or lime-soda), calcification and carbonation, smelting followed by slag leaching [11-25]. For instance, Kaußen & Friedrich [11] examined the various techniques for the alkaline extraction of aluminum from bauxite residue (red mud) and reported the recovery of about 70% for the Bayer process, the extraction efficiency of 95% for the Bayer process with alumina-enriched slag at high NaOH doses and the recovery of above 90% for sodium carbonate sinter process with subsequent leaching with water at 50 °C under optimal conditions. Valeev *et al.* [13] studied the extraction of alumina from a high-silica bauxite sample (Severoonezhsk deposit) and expressed that the extraction rate of alumina could reached 89% using pre-calcination and HCl leaching. Le *et al.* [14, 15] suggested the microwave roasting-leaching approach as an efficient technique for recovering alumina from low-grade diasporic bauxite samples. Toama *et al.* [16] applied a lime-soda sintering process before leaching for extracting alumina from a colored kaolinite ore and reported the recovery of ~80% using this process at CaO/SiO_2 molar ratio of 2.2, Na_2O/Al_2O_3 molar ratio of 1.2, and the sintering temperature of 1213 °C for 90 min. Wang *et al.* [17] utilized the calcification–carbonization process for extracting alumina from a low-grade bauxite and the extraction magnitude of 75.82% was obtained using this process, which was comparable to the Bayer process (61.44%). ElDeeb *et al.* [18] utilized a combination of the lime sinter method and the leaching process with sodium carbonate solution for extracting the alumina from a kaolin sample (Irkutsk region). In this work, the sintering process was took place in the temperature range 800-1400 °C for 1 h, and nearly 87% of the alumina was extracted at 1360 °C, and briquetting pressure of 5 MPa. Azof *et al.* [12] described treating a low-grade bauxite sample using the Pedersen process (smelting-reduction of bauxite and then leaching) for alumina recovery. They obtained the recovery of 46.7%, at 75 °C, 1 atm, 60 g/L $Na_2O_{(carbonate)}$ liquor within 30 min. Mahecha-Rivas *et al.* [19] evaluated the Bayer process, HCl leaching, and isopropanol for extracting aluminum from a metallurgical industry sludge in Medellín, Colombia. Their findings indicated that HCl leaching had a higher extraction rate (about 99.3%) and the Bayer process had better selectivity. Tang *et al.* [20] extracted about 99% alumina using a two-stage process combined with the Bayer process from secondary aluminum dross. Zhou *et al.* [22] proposed a clean two-stage Bayer process for recovering alumina from a high-iron gibbsitic bauxite. Generally, the literature demonstrates that among the different methods, the sintering operation before the leaching process can be very effective in the alumina production from hard-to-digest bauxites (like high silica bauxites). According to Kar *et al.* [24], the first commercial alumina production from bauxite was performed using a soda (Na_2CO_3) sintering process developed by Louis Le Chatelier in 1855. In the soda sintering method, bauxite ore is sintered with sodium carbonate at ~1200 °C to form $NaAlO_2$. Then the sintered compound is leached in an aqueous liquor to dissolve $NaAlO_2$. After that, it was found that the lime addition could increase the extraction rate of alumina and reduce the consumption of soda by forming complexes [24]. Indeed, in the lime-soda sintering operations, the goal is to make the Al_2O_3, Fe_2O_3, SiO_2, and TiO_2 content of the bauxite, consequently producing $Na_2O.Al_2O_3$, $Na_2O.Fe_2O_3$, $2CaO.SiO_2$, and $CaO.TiO_2$ at an appropriate sintering temperature [21]. Ghaemmaghami *et al.* [21] employed the lime-soda sinter process for recovering alumina from low-grade bauxite of the Semirom mine and gained an extraction efficiency of 88% under conditions: Na_2O/Al_2O_3 molar ratio of 0.9, CaO/SiO_2 molar ratio of 1.2, and sintering temperature of 1250 °C for 80 min. Sun *et al.* [23] suggested the sintering operation with Na_2CO_3

followed by a two-step leaching process with water and sulfuric acid for the extraction of Al_2O_3 and SiO_2 from the high-silica bauxites. The optimal sintering conditions were found to be the mole ratio of $Na_2O/(Al_2O_3 + SiO_2)$ of 1, the sintering temperature of 950 °C, and the sintering time of 30 min. Meanwhile, under these conditions, about 97% of Al_2O_3 and ~86% of SiO_2 were extracted for the production of alumina. Following this, Xiao *et al*. [25] developed a three-stage process based on alkali lime sintering, leaching, and magnetic separation to extract aluminum and iron by the simultaneous treatment of red mud (RM) and phosphogypsum (PG). Their findings exhibited that the optimal sintering conditions were the C/S ratio of 2, N/A ratio of 1.3, sintering temperature of 1100 °C, sintering atmosphere of N_2, and sintering time of 30 min. Under these conditions, the aluminum extraction rate, iron recovery, and iron grade were found to be 69, 78, and 83.8%, respectively.

Despite a great deal of studies on the extraction of alumina from bauxite resources, there are scant reports on extracting alumina from low-grade and non-bauxite resources, especially shale bauxites. On the other hand, although several research works have been developed in recent years to treat these deposits using the sintering process followed by the leaching process, there are still few progresses in this field, especially on the process optimization and the possible interactions between the influential operating factors. Additionally, each resource has its own individual mineralogical and chemical compositions, and the optimal operating conditions for the extraction of alumina are different from one type of aluminum-bearing resource to another. Thus, we pay special attention to characterizing and optimizing the extraction process of alumina from a low-grade (shale) bauxite using the sintering operation followed by the leaching process in detail.

2. Experimental Section

2.1. Materials

The low-grade shale bauxite samples required in the current study were supplied from the Jajarm alumina complex, in Iran. To provide representative samples, bauxite samples were firstly crushed by a laboratory jaw crusher and then pulverized by a pneumatic ring mill so that nearly 80% of the particles were finer than 90 microns in diameter (d_{80} = 90 μm). After that, the samples were well-mixed and homogenized utilizing a riffle. The obtained representative sample was analyzed by XRF SPECTRO iQ II (Ametek, Germany) to measure the chemical composition, as shown in Table 1. Additionally, the mineralogical compositions of the shale bauxite ore studied were characterized using a Siemens D-5000 X-ray diffractometer (Figure 1).

Table 1. XRF analysis of low-grad (shale) bauxite and limestone samples studied.

Composition	Al_2O_3	SiO_2	Fe_2O_3	TiO_2	CaO	MgO	LOI
Content (wt. %) for bauxite sample	36.22	22.11	20.42	3.33	3.13	0.62	13.65
Content (wt. %) for limestone	0.13	0.35	0.17	-	54.8	0.91	43.57

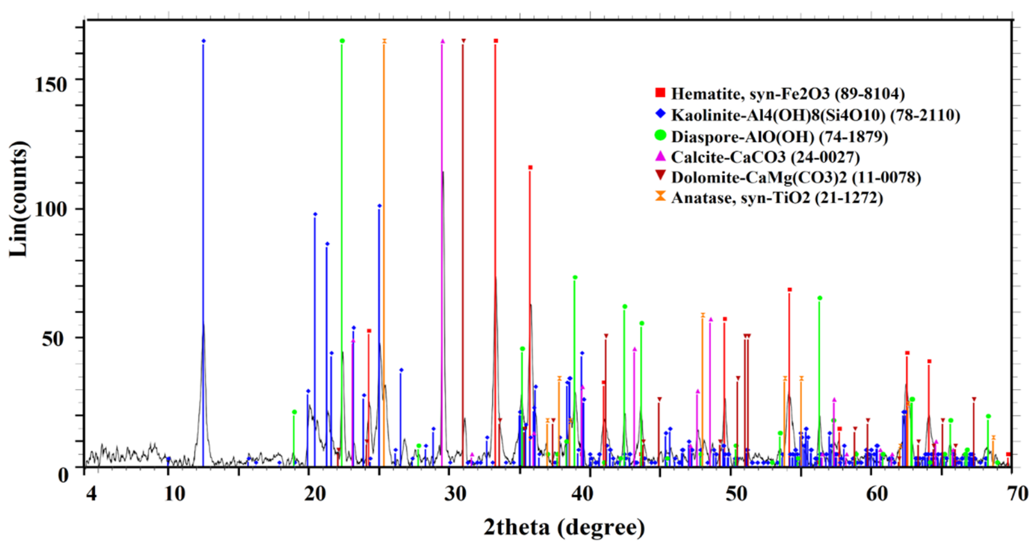


Figure 1. XRD pattern for low-grade (shale) bauxite sample studied.

It is clear from Table 1 that the Al_2O_3 to SiO_2 (A/S) ratio is about 1.64 and very low compared to the target modulus in the Jajarm bauxite mine complex (i.e. A/S ratio of about 4). The limestone samples were also provided from the Jajarm alumina complex, and its characterization is presented in Table 1. It can be observed from X-ray diffraction patterns (XRD) (Figure 1) that the main phases identified were hematite, kaolinite, diaspore, calcite, dolomite, and anatase.

Sodium carbonate (Na_2CO_3) with a purity level of 99% and NaOH supplied from Merck were also used for the sintering and leaching processes, respectively.

2.2. Sintering procedure

To conduct the experiments, firstly, a certain value of representative sample (low-grade shale bauxite) was accurately weighed and mixed with the additives consisting of lime and sodium carbonate based on predetermined ratios. A binder was also used to shape the mixture, which was 5% of the total weight of the materials including shale bauxite sample, lime, and sodium carbonate. The mixture was molded and pressed into a cylinder and afterward sintered in a furnace at various temperatures (1000-1200 °C) for 1 h. Figure 2 shows the molded (cylindrical) mixture (Figure 2a) before the sintering process and furnace output in the form of clinker after the sintering operation (Figure 2b).

Meanwhile, regarding the main compositions available in bauxite ore, the most important reactions that may occur during sintering operations are as follows [21, 24, 26]:

$Al_2O_3 + Na_2CO_3 \rightarrow Na_2O.Al_2O_3 + CO_2 \uparrow$	Forming sodium aluminate	(1)
$SiO_2 + 2CaCO_3 \rightarrow 2CaO.SiO_2 + 2CO_2 \uparrow$	Producing dicalcium silicate	(2)
$Fe_2O_3 + Na_2CO_3 \rightarrow Na_2O.Fe_2O_3 + CO_2 \uparrow$	Forming sodium ferrite	(3)
$Fe_2O_3 + CaCO_3 \rightarrow CaO.Fe_2O_3 + CO_2 \uparrow$ or $Fe_2O_3 + 2CaCO_3 \rightarrow 2CaO.Fe_2O_3 + 2CO_2 \uparrow$	Forming calcium ferrite or dicalcium ferrite	(4)
$TiO_2 + Na_2CO_3 \rightarrow CaO.TiO_2 + CO_2 \uparrow$	Producing calcium titanate	(5)

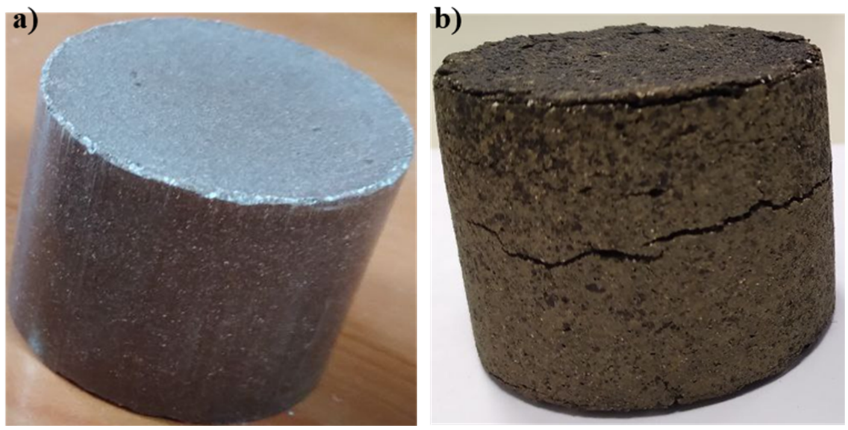


Figure 2. Image of the clinker before (a) and after (b) the sintering process of shale bauxite sample.

2.3. Sinter leaching process

After the sintering operation, the sintered cylindrical clinker was cooled and then ground to achieve a d_{80} of about 90 µm. Thereafter, the obtained sinter was leached using an alkali solution ($Na_2O_{(caustic)}$) with a certain concentration (15-75 g/L) at 90 °C and 400 rpm stirring rate within 30 min. All experiments were performed inside a 1000 mL covered glass beaker heated on a hot plate and equipped with a temperature thermometer and digitally controlled magnetic stirrer under atmospheric pressure. It is also noteworthy that the volume of the solution for all experiments was 100 ml. After finishing the leaching, the pulp was filtered and the liquid and solid phases were separated from each other and were analyzed separately. Figure 3 implies a schematic flow sheet of alumina treatment in the system investigated. Ultimately, two indices based on the following equations were used to determine the dissolution efficiency of alumina from the clinker [23, 27].

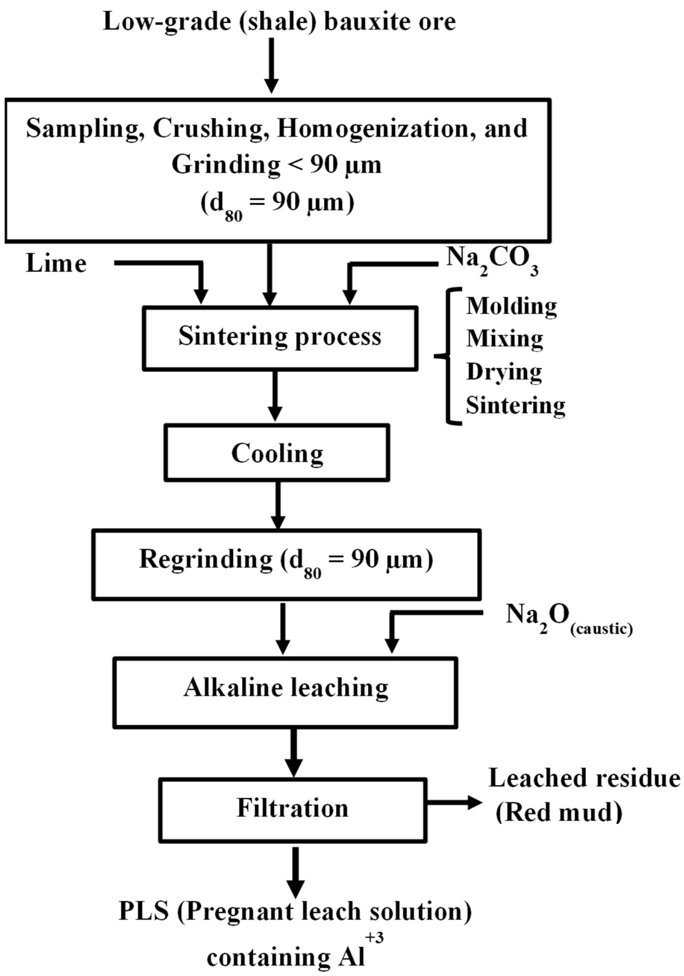


Figure 3. A schematic flow sheet of the alumina extraction process from an Iranian low-grade (shale) bauxite sample.

$$R_{Al_2O_3} = \frac{m_1 A_C - m_2 A_R}{m_1 A_C} \times 100 \tag{6}$$

$$R_{A/S} = \frac{m_1 \frac{A_C}{S_C} - m_2 \frac{A_R}{S_R}}{m_1 \frac{A_C}{S_C}} \times 100 \tag{7}$$

in whic, R_{Al2O3} represents the leaching rate of alumina (Al_2O_3) from the clinker, $R_{A/S}$ depicts the dissolution yield of Al_2O_3/SiO_2, A_c and A_R imply the content of Al_2O_3 and S_c, and S_R exhibit the content of SiO_2 in the sintered clinker and leaching residue (%), and m_1 and m_2 are the weight of the sintered clinker and residue (g), respectively.

3. Results and Discussion

3.1. Design of experiments

To obtain a deeper and better understanding of the sintering process and its effect on the extraction performance of alumina from low-grade shale bauxites, a suitable strategy was first chosen to conduct the experiments. To achieve this goal, the design of experiments (DOEs) technique with response surface methodology (RSM), which is a multipurpose tool in different situations for analyzing and interpreting data [28-30], was employed. Eventually, according to RSM, a rotatable central composite design (CCD) including four important operating parameters affecting the sintering process with a set of 27 runs (16 full factorial experiments, 8 axial experiments (α = 2), and 3 repeated experiments at central point) was selected and the experiments conducted. Table 2 represents the selected parameters based on their coded and actual amounts. The operational conditions of the experiments are presented in Table 3.

Table 2. Key parameters influencing the extraction process of alumina (Al_2O_3) and the selected range of factors based on the coded and real values.

Parameters	Symbol	Low axial level	Low factorial level	Central level	High factorial level	High axial level
		α = -2	-1	0	+1	α = +2
Sintering temperature (˚C)	*A*	1000	1050	1100	1150	1200
CaO/SiO_2 (C/S) molar ratio	*B*	1.8	1.9	2	2.1	2.2
Na_2O/Al_2O_3 (N/A) molar ratio	*C*	0.8	0.9	1	1.1	1.2
$Na_2O_{(caustic)}$ concentration (g/L)	*D*	15	30	45	60	75

Table 3. Experimental conditions for conducting experiments based on RSM-CCD strategy and the measured values of alumina extraction yield.

Run	Sintering temperature (˚C)	C/S molar ratio	N/A molar ratio	$Na_2O_{(caustic)}$ dose (g/L)	R_{Al2O3} (%)	$R_{A/S}$ (%)
1	1100	2.2	1	45	55.59	45.38
2	1100	1.8	1	45	58.25	52.74
3	1150	1.9	1.1	60	70.22	45.5
4	1200	2	1	45	40.09	35.66
5	1100	2	1	15	64.86	59.37
6	1150	2.1	1.1	30	61.1	53.93
7	1100	2	1.2	45	41.7	28.83
8	1050	2.1	1.1	30	36.01	24.1
9	1050	1.9	1.1	60	54.86	49.64
10	1000	2	1	45	16.26	15.16
11	1050	1.9	1.1	30	37.94	12.1
12	1050	2.1	1.1	60	20.86	9.64
13	1100	2	1	45	41.38	33.15
14	1050	2.1	0.9	60	19.79	13.07
15	1150	1.9	0.9	30	69.95	66.36
16	1150	2.1	0.9	30	64.22	59.95
17	1050	1.9	0.9	30	45.53	41.02
18	1100	2	1	45	44.88	36.33
19	1150	1.9	1.1	30	55.82	48.92
20	1150	2.1	0.9	60	29.76	21.41
21	1150	2.1	1.1	60	34.6	22.16
22	1100	2	1	45	41.21	31.81
23	1150	1.9	0.9	60	39.54	31.27
24	1100	2	0.8	45	35.75	31.2
25	1050	2.1	0.9	30	48.12	44.32
26	1050	1.9	0.9	60	13.38	9.45
27	1100	2	1	75	14.72	9.94

3.2. Modeling and statistical analysis

To investigate the behavior of the effective parameters consisting of sintering temperature, $Na_2O_{(caustic)}$ concentration as leaching lixiviant,, the molar ratio of CaO/SiO_2 (C/S), and the molar ratio of NaO_2/Al_2O_3 (N/A) on the dissolution of alumina from shale bauxite sample, it is first necessary to select an appropriate model for the relationship between the influential operating parameters and the process responses (here the extraction efficiency of alumina). In this regard, according to Equation 8 [28-30], the traditional models of linear, two-factorial interaction (2FI), and quadratic were utilized in the Design Expert (DX) software environment (version 13) to model and optimize the parameters influencing the extraction efficiency of alumina. The statistical analysis of the fitted models is summarized in Table 4.

$$Y = \alpha_0 + \sum_{i=1}^{n} \alpha_i x_i + \sum_{i=1}^{n} \alpha_{ii} x_i^2 + \sum_{1 \le i \le j}^{n} \alpha_{ij} x_i x_j + \varepsilon \quad (8)$$

in which Y denotes the process response (R_{Al2O3} or $R_{A/S}$), n is the number of factors, α_0 exhibits a constant term, α_i denotes the coefficients for the linear part of the model, x_i and x_j are the parameters, α_{ii} depicts the coefficients for the quadratic terms, α_{ij} implies the coefficients for the interaction variables, and ε is the residual error (the difference between the real and the approximate values) [28].

According to the results presented in Table 4, quadratic and two-factorial interaction (2FI) models are suggested, respectively, for the relationship between the operating parameters with the leaching yield of alumina (R_{Al2O3} and $R_{A/S}$). The adequacy of the models was checked using analysis of variance (ANOVA) and diagnostics nodes [29]. When the p-value (probability value) is less than 0.05, the F-value is large enough, and the R-square is larger than 0.8, the model is statistically significant [31, 32]. As observed, the proposed models for each output had a p-value smaller than 0.05 at the 95% confidence level and also a high R^2 magnitude (0.9039 for R_{Al2O3} and 0.8909 for $R_{A/S}$). Also the p-value of lack of fit is larger than 0.05 and is not significant relative to the pure error, showing that the selected model was well-matched to the experimental data.

Table 4. Summary of statistical analysis of the fitted models on the extraction process of alumina (R_{Al2O3} or $R_{A/S}$).

	Statistical analysis of R Al_2O_3		R^2	Adjusted R^2	Std. Dev.	
Source	**Sequential p-value**	**Lack of fit p-value**				
Linear	0.0004	0.0284	0.5907	0.5163	11.64	
2FI	0.0702	0.0382	0.7871	0.6541	9.84	
Quadratic	0.0364	0.0599	0.9039	0.7918	7.63	Suggested
Cubic	0.053	0.1556	0.9924	0.9507	3.71	Aliased
	Statistical analysis of R A/S					
Source	**Sequential p-value**	**Lack of fit p-value**	**R^2**	**Adjusted R^2**	**Std. Dev.**	
Linear	0.0007	0.0327	0.5666	0.4879	12.15	
2FI	0.0258	0.051	0.8067	0.6859	9.51	Suggested
Quadratic	0.1166	0.0643	0.8909	0.7637	8.25	
Cubic	0.0437	0.1857	0.9922	0.9496	3.81	Aliased

Ultimately, the selected models were fitted to the data and developed after removing insignificant terms (p-value > 0.05). Indeed, the coefficients with P-values greater than 0.05 were excluded from the model [33]. Equations 9 and 10 indicate the final developed model in terms of coded parameters for R_{Al2O3} with R^2 of 0.8972 and $R_{A/S}$ with R^2 of 0.7538, respectively. The coded amounts were utilized to simplify the calculations and more easily compare the operational factors.

$$R_{Al_2O_3} = +41.65 + 8.18 \times A - 3.25 \times B + 2.21 \times C - 9.83 \times D - 3.73 \times B \times C - 4.58 \times B \times D + 7.19 \times C \times D - 2.93 \times A^2 + 4.26 \times B^2 \quad (9)$$

$$R_{A/S} = +34.53 + 7.8 \times A - 2.93 \times B - 1.07 \times C - 10.31 \times D - 5.22 \times B \times D + 7.77 \times C \times D \quad (10)$$

It is noteworthy that the relationship between the coded and real amounts of the parameters was obtained based on Equation 11.

$$\theta_i = \frac{\theta_i - \theta_0}{\Delta\theta}, i = 1,2,3,..., n \quad (11)$$

In the above equation, θ_i is the codified extent of the *i*th parameter, θ_i is the real amount of the parameter, θ_0 is the amount of θ_i at the central point, and $\Delta\theta$ is the step change value [30, 31].

The statistical assessment of operating parameters based on the suggested models for each response (R_{Al2O3} and $R_{A/S}$) was carried out and the findings are displayed in Tables 5 and 6.

Table 5. ANOVA results of the obtained quadratic model to approximate the values of R_{Al2O3}.

Source	Sum of Squares	Degree of freedom	Mean square	F-value	p-value	Remarkable
Model	6528.1	9	725.34	16.48	< 0.0001	significant
A-Sintering temperature (˚C)	1606.88	1	1606.88	36.51	< 0.0001	
B-C/S ratio	254.15	1	254.15	5.77	0.028	
C-N/A ratio	117.13	1	117.13	2.66	0.1212	
D-$Na2O_{(caustic)}$ (g/L)	2319.88	1	2319.88	52.71	< 0.0001	
BC	223.2	1	223.2	5.07	0.0378	
BD	334.89	1	334.89	7.61	0.0134	
CD	826.85	1	826.85	18.79	0.0005	
A^2	219.55	1	219.55	4.99	0.0392	
B^2	464.08	1	464.08	10.54	0.0047	
Residual	748.19	17	44.01			
Lack of fit	739.6	15	49.31	11.49	0.0829	not significant
Pure error	8.58	2	4.29			
Cor total	7276.29	26				
Std. Dev.	6.63					
R^2	0.8972					
Adjusted R^2	0.8427					
C.V. %	15.49					
Adeq Precision	14.7511					

Table 6. ANOVA results of the obtained quadratic model to approximate the values of $R_{A/S}$.

Source	Sum of squares	Degree of freedom	Mean square	F-value	p-value	Remarkable
Model	5645.88	6	940.98	10.21	< 0.0001	significant
A-Sintering temperature (˚C)	1459.54	1	1459.54	15.83	0.0007	
B-C/S ratio	206.51	1	206.51	2.24	0.1501	
C-N/A ratio	27.31	1	27.31	0.2962	0.5923	
D-$Na2O_{(caustic)}$ (g/L)	2550.69	1	2550.69	27.67	< 0.0001	
BD	435.56	1	435.56	4.72	0.0419	
CD	966.28	1	966.28	10.48	0.0041	
Residual	1843.83	20	92.19			
Lack of fit	1833.05	18	101.84	18.89	0.0514	not significant
Pure error	10.78	2	5.39			
Cor total	7489.71	26				
Std. Dev.	9.6					
R^2	0.7538					
Adjusted R^2	0.68					
C.V. %	27.8					
Adeq Precision	12.7211					

As can be observed from ANOVA (analysis of variance) tables, the values of R^2 for R_{Al2O3} and $R_{A/S}$ are determined as 0.8972 and 0.7538, respectively, and also the difference between the value of R^2 and adjusted R^2 for R_{Al2O3} (0.0545) and $R_{A/S}$ (0.0738) is small, demonstrating that the proposed models have a high correlation and good validity for predicting the dissolution yield of alumina. Meanwhile, the adequate precision, which measures the signal-to-noise ratio, should be a ratio greater than 4 [30, 31], and it was found to be about 14.75 and 12.72 for R_{Al2O3} and $R_{A/S}$, showing that the proposed models have a proper signal to navigate through the design space of the models [29]. It is also found from ANOVA tables that the concentration of $Na_2O_{(caustic)}$, the sintering temperature, and the interactive effect of N/A ratio with the concentration of $Na_2O_{(caustic)}$ respectively have the greatest impact on the leaching yield of alumina. Meanwhile, the dissolution efficiency is strongly dependent on the interaction effects between the parameters.

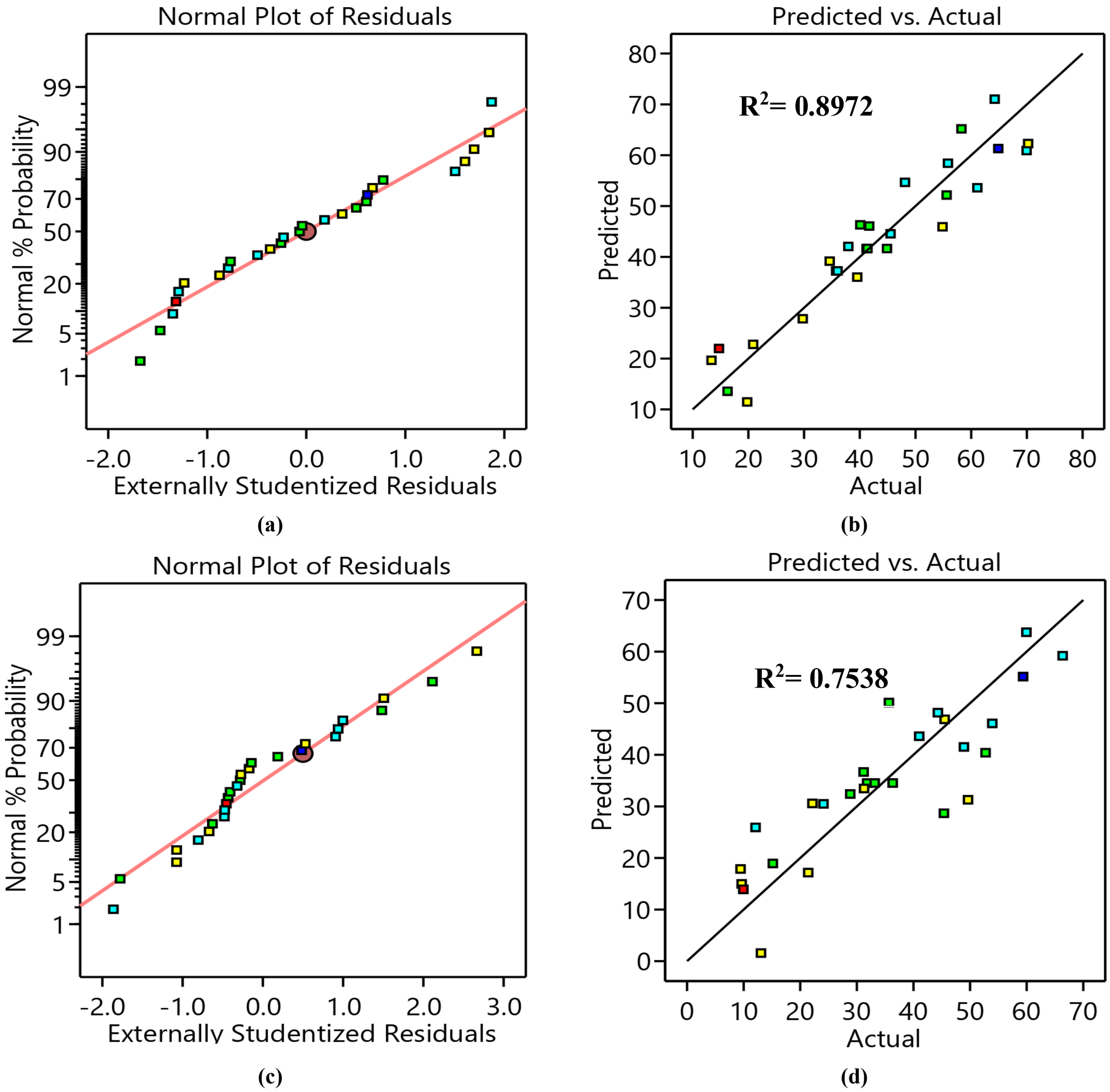
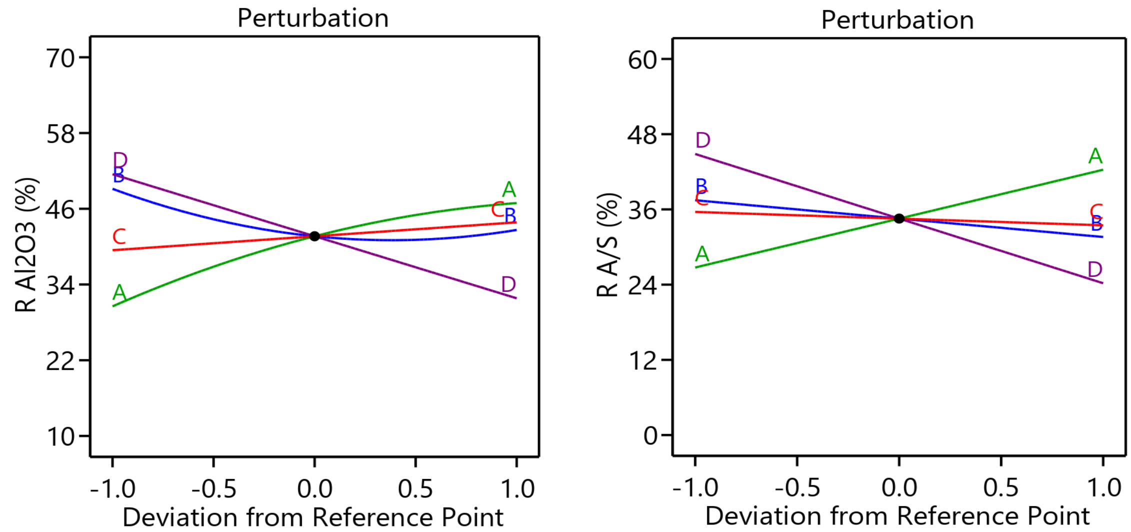


Figure 4. Normal probability plot of residuals and the measured values against predicted for R_{Al2O3} (a,b) and $R_{A/S}$ (c,d).

Additionally, the normal probability plot of residuals and the predicted versus actual graph displayed in Figure 4 affirm the accuracy and reliability of these models for estimating the extraction efficiency of alumina (R_{Al2O3} or $R_{A/S}$).

3.3. Effect of key parameters

After modeling and ANOVA analysis, the perturbation plot (Figure 5) was used to identify and evaluate the impact of all parameters at a specific point within a similar design space. According to Figure 5, a steep slope or curvature in each parameter shows the sensitivity of the alumina leaching rate to that parameter. As can be seen, the leaching reagent concentration ($Na_2O_{(caustic)}$) and the sintering temperature had the highest effect on the extraction efficiency of alumina, confirming the ANOVA results. In general, the influence degree of the parameters was: $Na_2O_{(caustic)}$ concentration > sintering temperature > C/S ratio > N/A ratio.



(a) (b)

Figure 5. Perturbation graph showing the relative importance of all parameters (*A*: sintering temperature, *B*: CaO/SiO$_2$ ratio, *C*: Na$_2$O/Al$_2$O$_3$ ratio, and *D*: Na$_2$O$_{(caustic)}$ dose) on the extraction process of alumina from shale bauxite sample studied.

In addition, to scrutinize and perceive a better understanding of the behavior of the parameters, 3D response surface diagrams were utilized and studied. Figures 6 and 7 indicate the combined impacts of two parameters when other parameters were fixed at their central point. As can be seen from 3D surface graphs, the dissolution yield of alumina (R_{Al2O3} and $R_{A/S}$) increased with a steep slope by increasing the sintering temperature ranging from 1050-1150 °C (Figures 6a and 7a). However, the changes were quadratic for the response of R_{Al2O3}, and the intensity of the increase decreased a little before the temperature of 1150 °C. Meanwhile, considering Table 2 and comparing run 4 with runs 13, 18, and 22 (central points), it can be seen that after 1150 °C, the dissolution efficiency decreased. This behavior may be owing to form insoluble complex phases at high temperatures. Kaußen & Friedrich [34] reported that the aluminum recovery increases in the leaching stage by enhancing the sintering temperature, but complete melting should be avoided. They recommended the sintering temperature between 1000-1100 °C. The increased dissolution efficiency with the sintering temperature rising (here from 1050 to 1150 °C) can be ascribed to the entrapment of the part of aluminum due to the addition of sodium carbonate and lime [35]. In addition, when the sintering temperature reaches above 1150 °C (here), the material enters the molten phase and the clinker strength increases, consequently leading to a low leaching of alumina [21, 24, 36]. It is accepted that at the roasting temperature more than 1100 °C, the extraction rate of alumina starts to diminish owing to the formation of insoluble complex phases [24]. Xiao *et al*. [25] brought up that the extraction rate of aluminum reduces at the sintering temperatures higher than 1100 °C and attributed it to the fact that the materials become hard and difficult to grind at great temperatures, which subsequently led to a decrease in the leaching rate. Xie *et al*. [37] also expressed that the high roasting temperature may cause to formation of more liquid phases into the product and accordingly a poor leaching efficiency.

The combined effect of N/A and C/S ratios (Figures 6b and 7b) indicates that at the low and high values of C/S (1.9 and 2.1), there are very few changes in the extraction degree of alumina with increasing or decreasing the ratio of N/A, and it is almost unchanged for $R_{A/S}$, indicating the less interactive effects between these two parameters. In contrast, it was observed a very strong synergistic (interaction) effects of Na$_2$O$_{(caustic)}$ concentration with C/S and N/A ratios on the extraction yield of alumina (R_{Al2O3} and $R_{A/S}$). The interaction graphs in Figure 8 provide a better representation of this issue. As can be seen in Figures 6-8, at the high and low C/S ratios the extraction efficiency promoted with decreasing Na$_2$O$_{(caustic)}$ concentration from 30 to 60 g/L, and this increase was highly greater at high C/S ratios (2.1) (Figures 6c, 7c, and 8). A similar trend was also observed at a low N/A ratio (0.9) and the dissolution yield greatly increased with a decrease in the concentration of Na$_2$O$_{(caustic)}$ (Figures 6d, 7d, and 8). However, the presence of a small value of $Na_2O_{(caustic)}$ in the leachate improves the stability of the leachate and helps to enhance the aluminum leaching efficiency. The decrease in the extraction efficiency of alumina with an increment in the concentration of $Na_2O_{(caustic)}$ can be because when $Na_2O_{(caustic)}$ concentration rises, $Na_2O_{(carbonate)}$ available in the liquor diminishes, whereas the accessibility of $Na_2O_{(carbonate)}$ is essential for the dissolution process. Meanwhile, the great dose of $Na_2O_{(caustic)}$ is led to generate a grey residue containing an aluminum hydrated phase and consequently the loss of aluminum in the leach liquor [12]. According to reactions (1) and (3), a low alkali ratio is not enough to react Na_2O with Al_2O_3 and Fe2O3 to form the soluble $Na_2O.Al_2O_3$ and $Na_2O.Fe_2O_3$ [21, 24, 26]. In addition, Ghaemmaghami *et al*. [21] reported that the insoluble materials are formed at a high alkali ratio, which can lead to the losses of Na_2O and Al_2O_3.

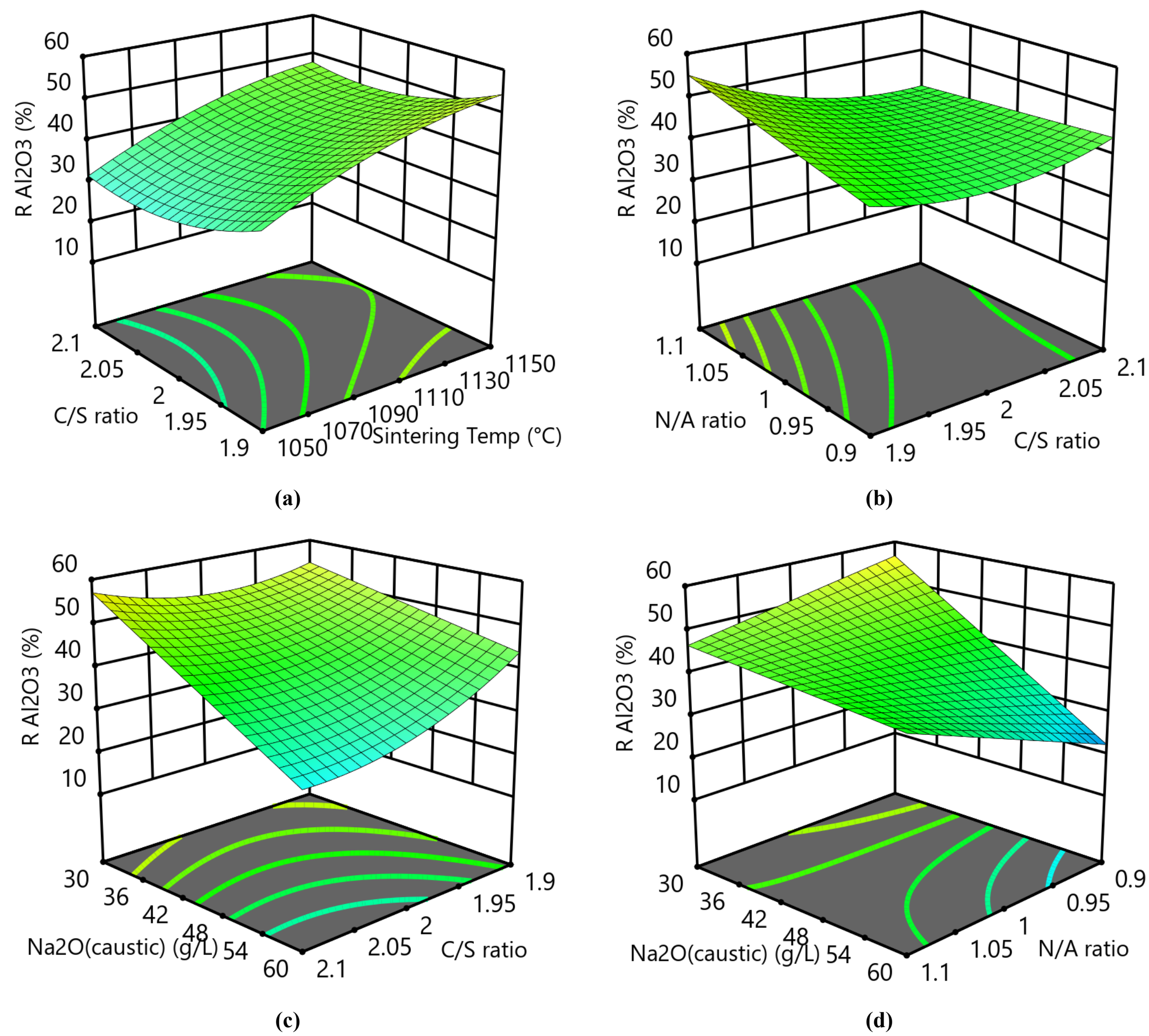


Figure 6. 3D response surface graphs describing the combined effects of two parameters on the dissolution rate of Al_2O_3 (R_{Al2O3}) when other parameters are fixed at the centre level.

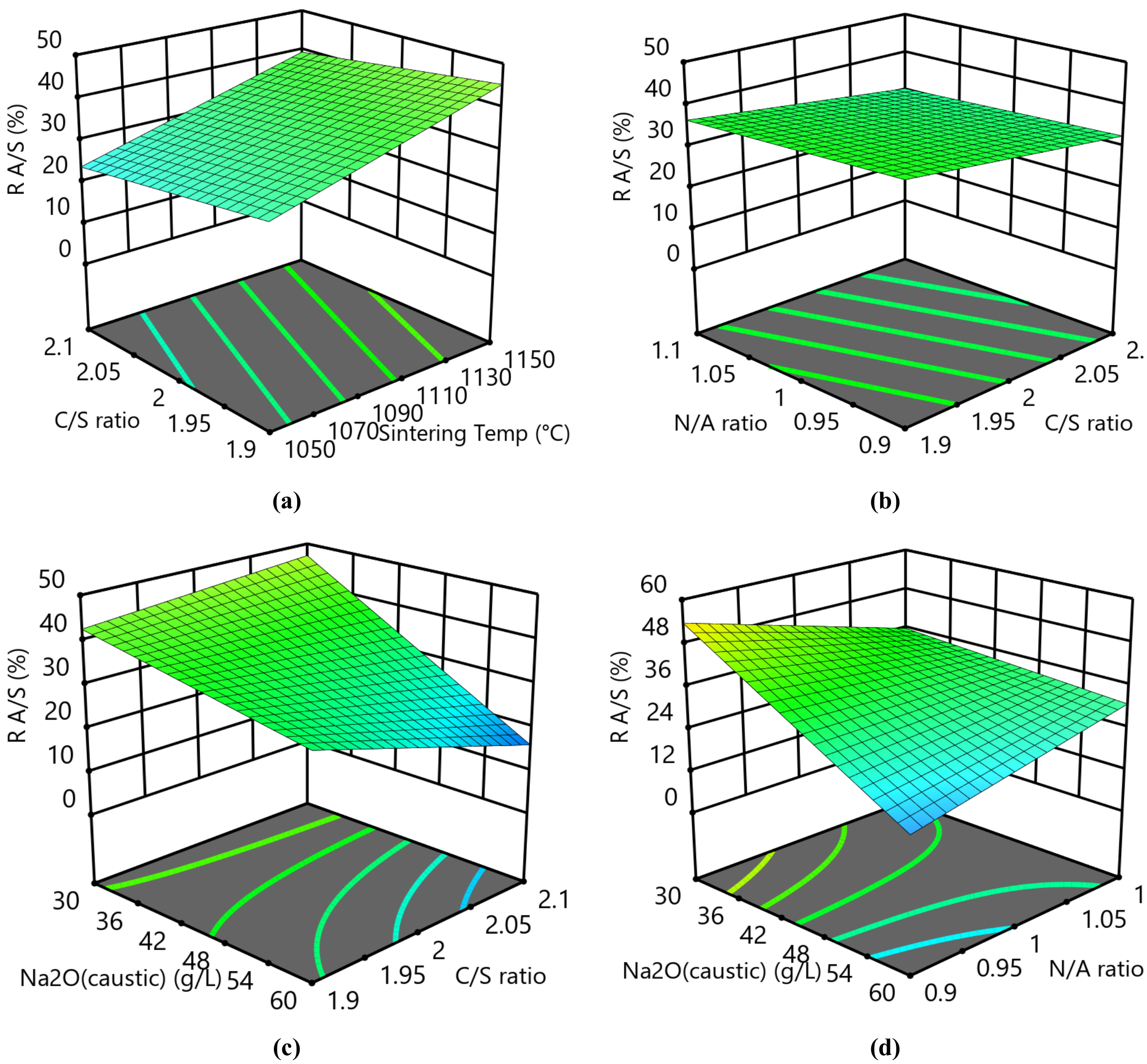


Figure 7. 3D response surface graphs describing the combined effects of two parameters on the dissolution rate of Al_2O_3/SiO_2 ($R_{A/S}$) when other parameters are fixed at the centre level.

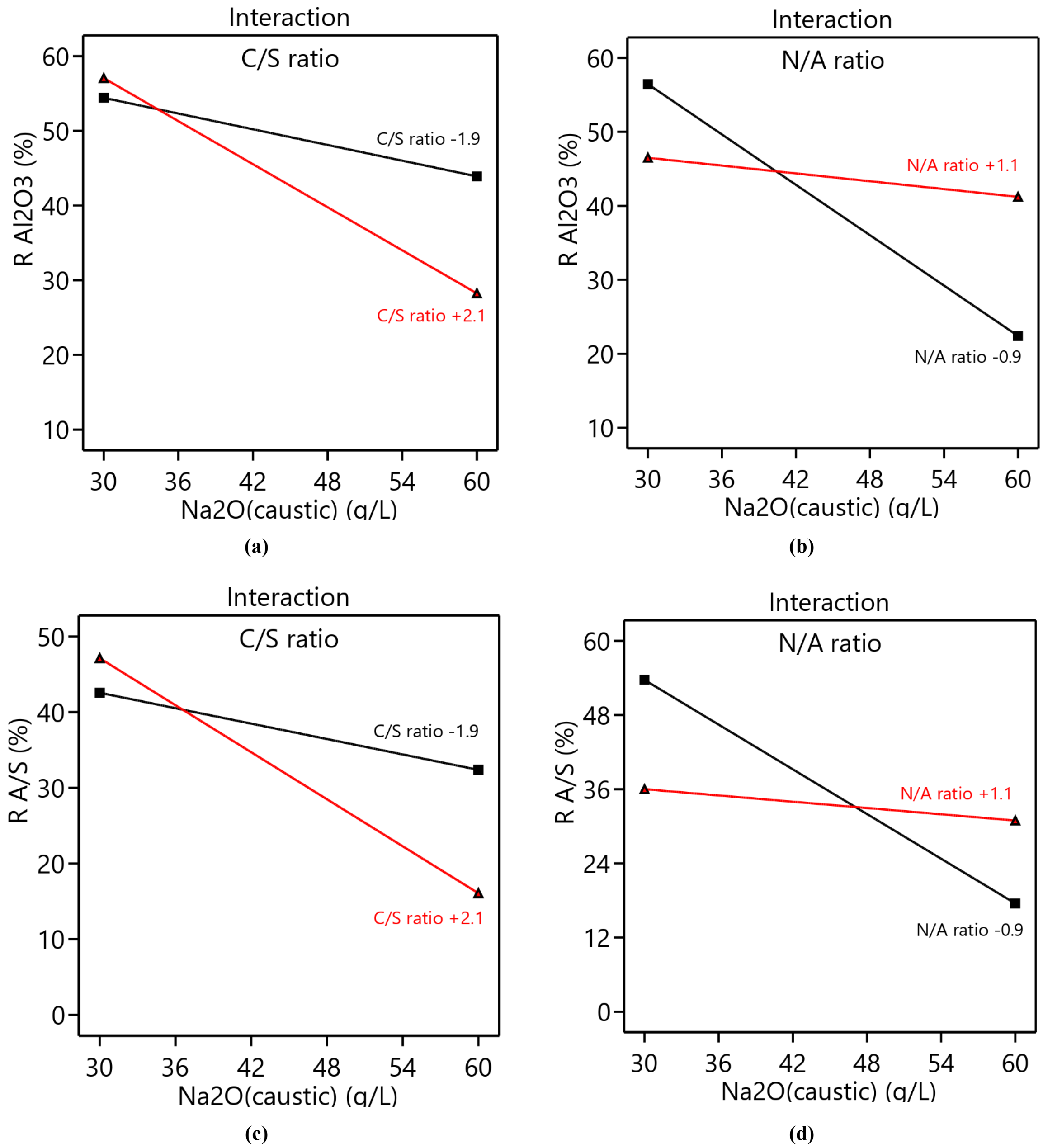


Figure 8. Plot of interactive (synergistic) effects of the concentration of Na_2O $_{(caustic)}$ with CaO/SiO_2 (C/S) ratio and Na_2O/Al_2O_3 (N/A) ratio for R_{Al2O3} (a,b) and $R_{A/S}$ (c,d).

In general, it was distinguished that the highest values of the extraction were achieved at high sintering temperature and C/S ratio and low values of $Na_2O_{(caustic)}$ and N/A. It is also clear from the graphs that in addition to the sintering temperature and $Na_2O_{(caustic)}$ concentration, the molar ratios of C/S and N/A are the critical parameters to increase the extraction efficiency. It can be also observed from Figure 6 that at the low concentration of $Na_2O_{(caustic)}$ (30 g/L), the extraction rate of alumina increases with increasing N/A (Na_2O/Al_2O_3) molar ratio. Whereas the dissolution yield reduces at the high level of $Na_2O_{(caustic)}$ concentration (60 g/L) with an increment in the N/A molar ratio. Meanwhile, an opposite trend was observed for the A/S ratio. He *et al.* [38] reported that the side reactions of alumina with silica and CaO at the low N/A ratios led to generating $Na_2Al_2SiO_4$ and $Ca_2Al_2O_5$ and hindered the generation of $NaAlO_2$ and accordingly reduced the extraction rate of alumina. They presented an optimum N/A molar ratio to extract the alumina. On the other hand, Meher and Padhi [39] expressed that a high N/A molar ratio (greater than 1.3) is harmful to the dissolution process of alumina owing to the formation of insoluble compounds between soda and alumina and so there is an optimum range for alumina extraction. As seen in Figures 6-8, a molar ratio of 0.9 is realized to be suitable to achieve a high extraction efficiency of alumina. Additionally, it is also found from the 3D surface curves that raising the C/S ratio (adding lime) improves the extraction performance and the C/S molar ratio of 2.1 is favorable to gain a high dissolution yield of alumina. The findings were in good agreement with the results obtained by Xiao *et al*. [25], in which a CaO/SiO_2 (C/S) molar ratio of 2 was reported for the recovery of alumina from red mud. Alp & Selim Goral [39] expressed that the excess lime reacts with a small value of alumina and leads to the form of insoluble calcium aluminum silicate hydrates and accordingly the dissolution efficiency reduces. In addition, Bai *et al*. [40] reported that both high and low values of CaO (Ca^{2+} ions) lead to the loss of Al_2O_3 and Na_2O with the formation of insoluble $Na_2O.Al_2O_3.2SiO_2$ at low calcium values and insoluble $4CaO.Al_2O_3.Fe_2O_3$ at high low calcium ratios.

3.4. Process optimiziation

Regarding the application and advantage of the desirability function method in simultaneous and multi-objective optimization, this technique was utilized to compute the optimum values of the process parameters to attain the maximum extraction efficiency of alumina (R_{Al2O3} and $R_{A/S}$). Figure 9 illustrates the results obtained from numerical optimization. As can be observed, the optimized values of operating parameters were found to be 1150 °C sintering temperature, 2.1 CaO/SiO_2 molar ratio, 0.9 Na_2O/Al_2O_3 molar ratio, and 30 g/L $Na_2O_{(caustic)}$. Under these conditions, the highest values of R_{Al2O3} and $R_{A/S}$ were measured at about 71 and 63.76% with a desirability of 0.977, respectively. Three verification tests were performed in the same optimal conditions and the average amounts of R_{Al2O3} and $R_{A/S}$ were calculated to be approximately 70.89 and 63.46%, showing a superior agreement with the predicted values.

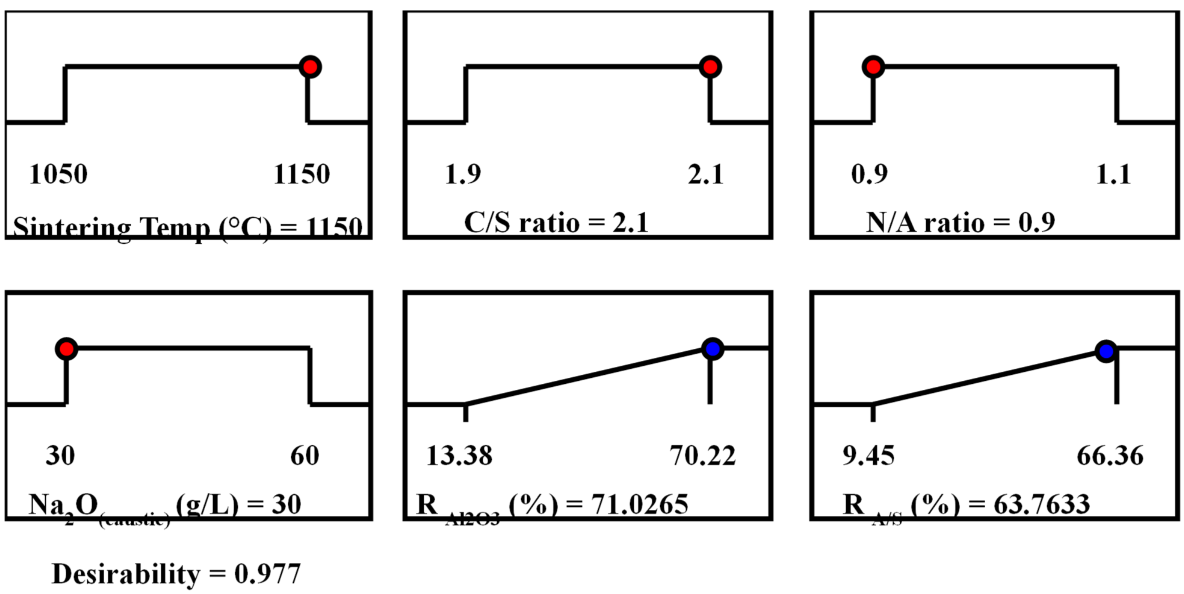


Figure 9. The optimized values of key parameters influencing alumina extraction performance (R_{Al2O3} and $R_{A/S}$) based on the desirability function method.

4. Conclusions

This research work investigated the feasibility of the alumina extraction process from an Iranian low-grade (shale) bauxite ore using the sintering method and then its alkaline dissolution. To achieve this goal, two indices including the leaching rate of Al_2O_3 (R_{Al2O3}) and Al_2O_3/SiO_2 ($R_{A/S}$) were considered as the response variables in the alumina extraction process. Then the behavior of important operating parameters including the sintering temperature, CaO/SiO_2 (C/S) molar ratio, Na_2O/Al_2O_3 (N/A) molar ratio, and Na_2O $_{(caustic)}$ concentration was examined on the dissolution yield of alumina applying response surface modeling based on central composite design. The results of this study can be summarized as follows:

1) A quadratic polynomial mathematical model with R^2 of 0.8972 and a two-factorial interaction (2FI) model with R^2 of 0.7538 was matched with the experimental data for predicting R_{Al2O3} and $R_{A/S}$, respectively.

2) ANOVA analysis and the perturbation graphs proved that $Na_2O_{(caustic)}$ concentration, sintering temperature and interactive effect between Na_2O/Al_2O_3 ratio and the concentration of $Na_2O_{(caustic)}$ had the highest impact on the extraction performance.

3) The extraction efficiency was enhanced strongly by raising the sintering temperature and reducing $Na_2O_{(caustic)}$ concentration. The effect of N/A ratio and C/S also showed that the high levels of C/S (2.1) and the low values of N/A (0.9) had a positive effect on the extraction efficiency of alumina.

4) The process optimization was carried out utilizing the desirability function procedure in the Design Expert 13 software environment, and the optimum conditions were distinguished at 1150 °C sintering temperature, 2.1 CaO/SiO_2 molar ratio, 0.9 Na_2O/Al_2O_3 molar ratio and 30 g/L $Na_2O_{(caustic)}$. In the optimized values of parameters, the maximum extraction efficiency was nearly 71.03% for R_{Al2O3} and 63.76% for $R_{A/S}$.

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استخراج آلومینا از سنگ معدن بوکسیت کم عیار (شیلی) با استفاده از یک فرآیند سینترینگ با آهک- سودا و بدنبال آن فروشویی قلیایی

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چکیده:

رشد جهانی تقاضای آلومینیوم با مدرن شدن جامعه ما منجر به علاقه‌مندی به توسعه روش‌های جایگزین برای تولید آلومینیوم از منابع غیر بوکسیتی و کم عیار مانند بوکسیت‌های شیلی شده است. برای چنین ذخایری، فرآیند مرسوم بایر چالش برانگیز است و برای استخراج آلومینیوم کارآمد نیست و فرآیند سینترینگ موثر شناخته شده است. بنابراین، این مطالعه بر بررسی دقیق امکان فنی استخراج آلومینا از یک سنگ معدن بوکسیت کم عیار (شیلی) ایران حاوی ۳۶/۲۲٪ Al_2O_3، ۲۲/۱۱٪ SiO_2، ۲۰/۴۲٪ Fe_2O_3، ۳/۳۳٪ TiO_2 و ۳/۱۳٪ CaO انجام شد. در این راستا، فرآیند سینترینگ با آهک- سودا و سپس لیچینگ قلیایی برای استخراج آلومینا اتخاذ شد و مدل‌سازی سطح پاسخ برای ارزیابی پارامترهای مهم مانند دمای سینترینگ، غلظت Na_2O(سوداور)، نسبت مولی CaO/SiO_2، و نسبت مولی Na_2O/Al_2O_3 به کار گرفته شد. یافته‌ها نشان داد که میزان استخراج با افزایش دمای سینترینگ و نسبت CaO/SiO_2 و کاهش مقدار Na_2O(سوداور) و نسبت Na_2O/Al_2O_3 بهبود یافت. همچنین مشخص شد که غلظت Na_2O(سوداور)، دمای سینترینگ و اثر متقابل غلظت Na_2O(سوداور) با نسبت Na_2O/Al_2O_3 بیشترین تأثیر را بر راندمان استخراج داشتند. بهینه‌سازی فرآیند با استفاده از رویکرد تابع مطلوبیت انجام شد و بیش از ۷۱ درصد Al_2O_3 در دمای سینترینگ ۱۱۵۰ درجه سانتی‌گراد، نسبت مولی CaO/SiO_2 ۲/۱، نسبت مولی Na_2O/Al_2O_3 ۰/۹ و میزان Na_2O(سوداور) ۳۰ گرم بر لیتر استخراج شد. در نهایت، نتیجه گرفته شد که فرآیند سینترینگ آهک- سودا در دمای ۱۱۵۰ درجه سانتی گراد و به دنبال آن فروشویی قلیایی یک مرحله‌ای با Na_2O(سوداور) در دمای ۹۰ درجه سانتی‌گراد می‌تواند از نظر متالورژیکی برای عمل‌آوری بوکسیت‌های کم عیار (شیلی) کارآمد باشد.

کلمات کلیدی: بوکسیت‌های کم‌عیار، لیچینگ آلومینیوم، سینترینگ، راندمان انحلال، بهینه‌سازی.