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Preparation of α-alumina particles from alunite ores by using dry caustic assisted roasting

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Abstract: In this study, the production of α-alumina particles from alunite ore was carried out through roasting-leaching and precipitation tests. The effects of parameters such as temperature, stirring speed, solid ratio, and leaching time on Al yield% were investigated in leaching tests using the response surface methodology (RSM) and central composite design (CCD). As a result of the experiments, the highest Al yield ratio as 86.06% was achieved in water leaching under conditions of 100°C, 900 rpm, 15% as solid ratio, and 65 min. as time after roasting at 600°C with NaOH. The experimental results revealed that temperature and solid ratio% were more influential than other parameters. To precipitate $Al(OH)_3$ from the loaded solution, HCl was used to reduce the pH. The precipitate was roasted in a muffle furnace at 1100°C (±50°C) for 240 min. Then, α-alumina particles were obtained with an efficiency of 89.89% and purity of 91.24%. The morphological properties of the final product were confirmed by TGA-DTA, SEM/EDX and X-ray diffraction analyses.

Keywords: alunite, thermal activation, roasting, leaching

1. Introduction

Alumina (Al_2O_3) can be used in many industrial areas owing to its superior properties such as hardness, high melting point, resistance to corrosion, abrasion, and thermal and mechanical shocks. The bauxite reserve, which was 27.8 billion tons in 2016, decreased to 26.3 billion tons in 2020, whereas alumina production increased from 101.3 million tons to 134.5 million tons (USGS, 2023). Because of the limited bauxite reserves and the increase in alumina production costs, nonbauxite alumina production sources were investigated. Different resources such as clay, kaolinite, nepheline syenite, alunite, pyrophyllite, muscovite, and shales can be used as raw materials to prepare high-quality alumina. Among these sources, alunite is abundant in the Earth's crust (Al Aieel et al., 2006; Bazin et al., 2007; Numluk et al., 2012, Erdemoglu et al., 2018). From non-bauxite sources, Russia and Iran produce alumina from nepheline syenite on a pilot scale, while Canada produces alumina from kaolinite and fly ash (Aydoğmuş et al., 2023). Previous studies have reported that alunite does not dissolve in water, acid, or a base unless calcined (Scott et al., 2012). Thermal activation is pre- implemented to increase the alunite's ability to react with chemicals and ensure the degradation of the crystal structure, thereby increasing the dissolution efficiency by removing water from the aluminium silicate through thermal activation (Stroffregen et al., 1987; Peng et al., 2019; Parida et al., 2019). In the calcined state at 500 °C, the decomposition of the alunite structure facilitates its solubility through acidic and basic extractions.

The only component dissolved by hot water leaching after calcination is potassium sulphate. The insoluble residue contains alumina and silica. The residue is extracted with basic and acidic liquids to obtain aluminium sulphate (Zhao et al., 2015; Zhu et al., 2019). The first step in the production of alumina from alunite is leaching. Studies on the leaching of alunite ore have been conducted using various acids and bases, such as H_2SO_4, NaOH, KOH, NH_4OH, and $Ca(OH)_2$ (Mohammadi et al., 2013).

Özdemir and Çetişli (2004) conducted a sulfuric acid leaching study of alunite in a batch reactor with a KF/Al_2O_3 ratio of 0.15-0.90, achieving an aluminum recovery of 85%, while the hydrochloric acid leaching study resulted in an aluminum recovery of 83%. Zhao et al. (2015) performed a leaching

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experiment using 30% sulfuric acid with alunite in acid-concentrated water, obtaining an aluminum recovery of 87.2%. Luo et al. (2017) employed an alkaline ore ratio of 3/1 in a granular leaching process, resulting in an aluminum recovery of 84%. Li et al. (2018) achieved an aluminum recovery of 80% in their hydrometallurgical leaching study.

Al_2O_3 with different polymorphs can be prepared under different production conditions, including temperature and duration. Alumina forms according to roasting temperatures 600-800^oC (Das et al., 2007; Sarker et al., 2015; Guo et al., 2018; Mahinroosta et al., 2018), γ-alumina, 900^oC (Gangwar et al., 2014; Sarker et al., 2015;), η-alumina 1000^oC (Mirjalili et al., 2011; Matori at al., 2012; Pepper et al., 2018; Shi et al., 2021;), θ- alumina and 1000-1400^oC α-alumina (Mirjalili, 2011; Gangwar et al., 2014; Mahinroosta at al., 2018). θ- alumina form was determined at 900-1000^oC (Mirjalili et al., 2011; Pepper et al., 2018). α-alumina was obtained through roasting at 1200°C, whereas δ-alumina, γ-alumina, and θ-alumina were obtained through 700°C to 900°C roasting (Mirjalili et al., 2011; Pepper et al., 2018). Obtained mixed-phase alumina through 900°C roasting (Matori et al., 2012). Produced α-alumina via roasting at 1200°C for 270 min. Al_2O_3 synthesized from various alumina polymorphs (β, δ, γ, and κ) exists in metastable phases, making it unsuitable for high-temperature applications such as ceramics, refractory materials, microelectronic packaging, catalysis, coatings, and heavy metal purification. In contrast, α-Al_2O_3 particles, due to their phase stability, are well-suited for such high-temperature uses. Alunite finds contemporary use in fertilizer manufacturing. Its potential application in alumina production can establish a novel market, producing a high-value commodity (Said et al., 2020). Moreover, it presents a more environmentally sustainable and less impurity-laden process when compared to other methods.

This study aims to produce α-Al_2O_3 in a thermally stable phase from alunite obtained from the Eti Maden Enterprises site in Şaphane, Kütahya, Türkiye. Roasting temperature, leaching time, solid ratio (%), and NaOH concentration were analyzed using response surface methodology (RSM)–central composite design (CCD) to achieve the maximum yield of Al. The multivariate statistical model known as Response Surface Methodology-Central Composite Design (RSM-CCD) emphasizes the mathematical relationships among multiple dependent and independent variables at different levels. This design employs a spherical, rotatable (face-centered) configuration, which enhances the quality of the predictive variance at all points equidistant from the central point of the design. This study contributes to the accurate modelling of the process by taking into account the factors in Türkiye.

2. Materials and methods

2.1. Materials

The chemical composition of the sample, determined through XRF analysis, was presented in Table 1. The sulphur grade of the sample was found very high as a 14.4%. The experimental results were evaluated via Thermogravimetric (TGA)-differential thermal analysis (DTA), X-Ray diffraction analysis (XRD) and Scanning Electron Microscopy (SEM) (FEI Quanta 650 field-emission). The mineral composition of the sample was analysed using X-ray diffraction (XRD) with a Panalytical Empyrean system. The phase changes of alunite were investigated using XRD analysis on a Panalytical Empyrean instrument with Cu -Kα radiation in the 2θ range of 5° to 80°. The primary components identified were alunite ($KAl_3(SO_4)_2 \cdot (OH)_6$), kaolinite ($Al_2Si_2O_5(OH)_4$), and quartz ($SiO_2$), as illustrated in Fig. 1. The thermogravimetric method was used to show the change in the mass of alunite during roasting at different temperatures. TGA-DTA (Mettler Toledo) analysis was conducted on the alunite sample in a nitrogen atmosphere, at a heating rate of 20°C/min, in the range of 30°C to 1200°C. It showed that the mass change of alunite ore at different temperatures is presented in Fig. 2. Two large endothermic peaks were observed in the TGA-DTA curves as a weight loss. The first endothermic peak appears around 560°C, indicating the dehydration of alunite, resulting in the formation of alum and α-alumina particles. The second endothermic peak was observed around 810°C. The observed endothermic peak indicates the removal of hydroxyl groups from the structure through evaporation. The second peak indicates the decomposition of aluminium sulphate into alumina and sulphur dioxide. A weight loss of approximately 6.173% is observed in the first endothermic peak, and a weight loss of 13.673% is observed in the second peak, corresponding to a total sulphate loss of 23.509%.

Table 1. Chemical analysis of the alunite sample

Contents	SiO_2	Al_2O_3	K_2O	Fe_2O_3	CaO	MgO	MnO	Na_2O	P_2O_5	SO_3	TiO_2	LOI
Grade (%)	45.1	14.2	3.3	0.9	0.2	0.2	0.1	0.1	0.1	14.4	0.3	20.85

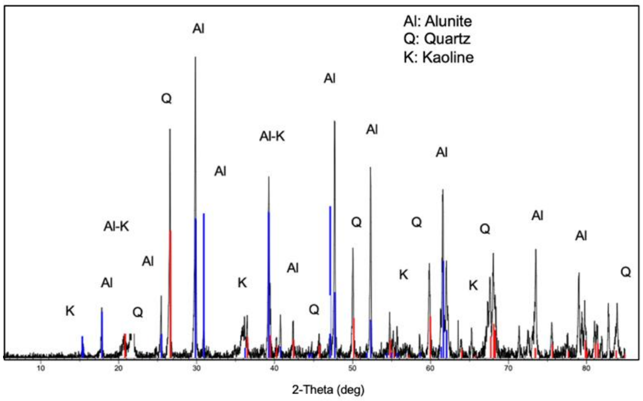

Fig. 1. XRD pattern of the alunite sample

Previous studies have reported that anhydrous alum transforms into amorphous Al_2O_3 because of an endothermic reaction at 560°C. As the temperature increases, $K_3Al(SO_4)_3$ decomposes into K_2SO_4 and amorphous $Al_2(SO_4)_3$. It then further decomposes with SO_3 emission and transforms into crystalline γ-Al_2O_3 upon reaching 800°C (Mohammadi et al., 2013). The transformation of alunite by decomposing crystalline water into $KAl(SO_4)_2$ and amorphous Al_2O_3, which creates endothermic peaks at approximately 560°C and 810°C, is shown in Equation 1. The reactions occurring at approximately 800°C to 810°C, where the second endothermic peak was observed in Equation 2 and Equation 3. During roasting of alunite, products such as K_2SO_4, Al_2O_3, and SiO_2, were processed, optimally.

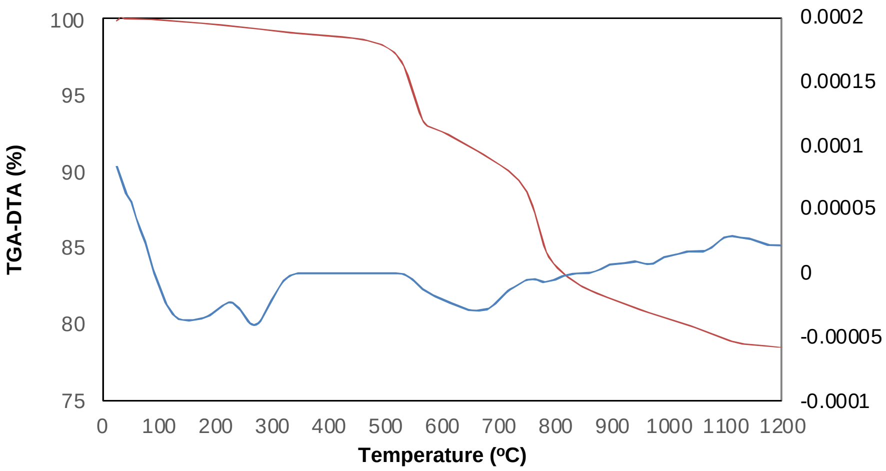

Fig. 2. DTA/TGA tests of the alunite sample

$$KAl_3(SO_4)_2(OH)_6 \rightarrow KAl(SO_4)_2 + Al_2O_3 + 3H_2O \quad (1)$$

$$2/3\ K_3Al(SO_4)_3 \rightarrow K_2SO_4 + 1/3Al_2(SO_4)_3 \quad (2)$$

$$2/3\ Al_2(SO_4)_3 \rightarrow 2/3\,Al_2O_3 + 2SO_2 + O_2 \quad (3)$$

2.2. Methods

The chemicals (NaOH, KOH, and HCl) used in this study were supplied by Merck and used without any purification. The experimental process, as shown in Fig. 3, is divided into six stages: sample preparation, roasting 1, roasting 2, leaching, precipitation, roasting 3. These stages are shown in Fig. 2.

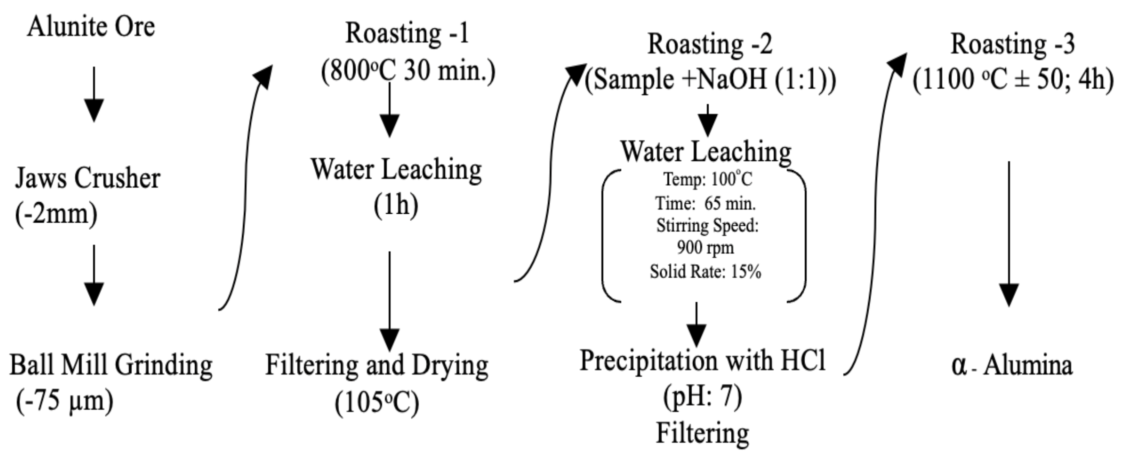

Fig. 3. The experimental steps of α-alumina processing methods

The sample was crushed to -2 mm using a lab-scale jaw crusher. Then, the crushed sample was ground to -75 µm using a lab-scale ball mill with a diameter of 17 cm and a critical speed of 72 rpm for 30 min. The water-soluble elements (K, Na, Cl) were removed before the NaOH roasting test through calcination at 800°C for 30 min, followed by water leaching for 60 min. A solid ratio of 15% and a stirring speed of 300 rpm were set. The slurry was filtered using filter paper and washed with distilled water. The residues obtained after washing were used as the initial samples for NaOH roasting tests. This sample was mixed with NaOH in a 1:1 ratio in porcelain crucibles and roasted in a muffle furnace at 600°C for 60 min. The chemical reaction in Equation 4 which represents the precipitation of Al ions from the leachate solution in the form of aluminium hydroxide.

$$2KAl\,(SO_4)_2.12H_2O + 6NaOH \rightarrow 2Al\,(OH)_3 + K_2SO_4 + 3Na_2SO_4 + 24H_2O \quad (4)$$

The Response Surface Methodology-Central Composite Design (RSM-CCD), known as a multivariate statistical model, emphasizes the mathematical relationships between multiple dependent and independent variables at different levels. This design includes the use of corner points to improve the quality of variance prediction at points equidistant from the central point and to estimate extreme responses. The four selected screening parameters are Solid Ratio (%), Leaching Time (min), Leaching Temperature (°C), and Stirring Speed (rpm). Additionally, the effects of these parameters on aluminum recovery efficiency have been analyzed.

In this study, RSM-CCD was calculated using the Design-Expert software (Ease-Stats version 13). The response variables were evaluated using statistical models incorporating polynomial terms. Equation 5 represents the cause-and-effect relationship, where Y denotes the response variable (metal recovery efficiency), Xi represents the levels of process variables, β_0 is the constant term, βi are the linear coefficients, βii are the quadratic coefficients, βij are the interaction coefficients, e represents the random error, and k denotes the total number of variables used to optimize Al recovery efficiency.

For model selection, the highest-degree polynomial model was determined based on the criteria of sequential p-value, as well as the maximization of adjusted R_2 and predicted R_2 values. A non-aliased model was preferred for analysis. The selected model was analyzed using Analysis of Variance (ANOVA), while the interaction effects of process parameters on the responses were interpreted using the regression equation and RSM contour plots (Kaynar et al., 2018; Sunny et al., 2020).

$$Y = \beta_0 + \sum_{i=1}^{4} \beta_i X_i + \sum_{i=1}^{4} \sum_{j=i+1}^{4} \beta_{ij} X_{ij} + \sum_{i=1}^{4} \beta_{ii} X_i^2 + e \quad (5)$$

ANOVA was conducted for all regressions at a 95% confidence interval to assess statistical significance (Table 4). The significance of each variable was determined by analyzing the p-value and F-value. Additional Table 2 shows the experimental parameters, and their levels used in the study. These tests were conducted in double-walled glass containers with a capacity of 250 mL. After the reaction was completed, the solid and liquid phases of the leachate solution were separated using Whatman filter paper. The amount of dissolved Al in the loaded solution was determined by atomic absorption spectrometry (AAS, 900 H; PerkinElmer).

Table 2. Experimental parameters and the levels of the variables

Variables	Code	-α	-1	0	+1	+α
Solid Ratio (%)	A	5	10	15	20	25
Temperature (°C)	B	20	40	60	80	100
Mixing Speed (rpm)	C	300	600	900	1200	1500
Time (m)	D	5	35	65	95	125

Precipitation and Roasting Tests; 2000 mL stock solution was prepared considering the optimal leaching conditions prior to the precipitation test. The Al ions in the leachate solution were precipitated as hydroxide by adding the required amount of HCl acid (15%) to decrease the solution pH from 13.5 to approximately 7. The solution was then stirred at 300 rpm for varying durations (1 hours). Once the reaction time was completed, the precipitate was recovered through solid-liquid separation. The aluminium ions in the leachate solution were precipitated in the form of aluminium hydroxide. The obtained precipitate was calcined at 700-1100°C for 240 min.

3. Results and discussion

The XRD patterns of alunite calcined at 600°C, 700°C, and 800°C were shown in Fig. 4. Quartz (SiO_2), potassium aluminium sulphate ($KAl(SO_4)_2$), and kaolin ($Al_2Si_2O_5(OH)_4$) were identified as the main phases of dehydrated alunite. The presence of potassium aluminium sulphate ($KAl(SO_4)_2$) was attributed to the decomposition of alunite ($KAl_3(SO_4)_2(OH)_6$). However, as the temperature increases, the quartz peaks become more prominent, and the sample gradually transitions to an amorphous state. In the first stage, the alunite sample was calcined at 800°C for 30 min in a muffle furnace Potassium was extracted into the solution with an efficiency of 85% through a water leaching experiment of the activated product. The aluminium content in the filtered solid part was 38.9%. In the second stage of the leaching experiments, NaOH was used as the leaching agent. The reaction induced by mixing the sample and NaOH in a 1:1 ratio in a muffle furnace was expressed in Equation 4. Obtained values had higher priority than some previous studies (Li et al., 2018). After water leaching following roasting with NaOH, the dissolution efficiencies of Al were 86.06%.

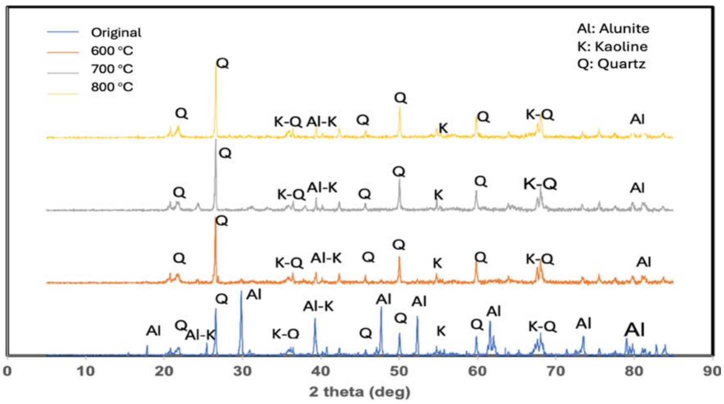

Fig. 4. XRD pattern of main and roasting samples

Leaching experiments based on the central composite design (CCD) using response surface methodology (RSM) were conducted to investigate the effects of four independent parameters solid ratio (A), temperature (B), stirring speed (C), and leaching time (D)—on the dissolution of Al from alunite, as well as the interactions between these parameters. A total of 27 tests were designed by varying these parameters. The experimental conditions and observed results were given in Table 3.

Using RSM-CCD modelling, the adequacy of the model was evaluated based on statistical parameters through analysis of variance (ANOVA) values. ANOVA was employed to assess the interactions between experimental parameters, the significance of model coefficients, and the lack of fit. Parameters such as mean squares, F-value, p-value, and sum of squares were calculated. An F-value greater than 1 and a p-value less than 0.05 indicate the statistical significance of the model. The ANOVA analysis results, which measure the variation explained by the model and determine its quality for aluminum dissolution yield during water leaching after roasting, along with the R^2, Radj, and standard deviation, are presented in Table 4. The fact that the R^2 and Radj values are close to 1 indicates the compatibility of the applied model. The low standard deviation value indicates that there is little variability in the experimental data. The adjusted R^2 value highlights the variations in the mean explained by the model (Behera et al., 2018). When evaluated alongside the ANOVA results, it suggests that the predicted values are close to the actual measurements.

Table 3. Response surface methods-Central compozit design with actual values of responses

No	Solid Ratio (%)	Temperature (°C)	Mixing Speed (rpm)	Time (min.)	Yield Al%
1	10	80	1200	35	81.61
2	10	40	1200	35	62.43
3	20	80	600	35	84.52
4	15	60	900	65	84.38
5	20	40	1200	95	74.23
6	15	60	900	5	64.82
7	5	60	900	65	83,21
8	15	100	900	65	86.06
9	15	20	900	65	70.47
10	20	80	1200	95	75.45
11	10	80	1200	95	80.12
12	20	80	1200	35	83.22
13	10	40	600	95	84.02
14	15	60	900	65	84.38
15	25	60	900	65	82.48
16	15	60	900	125	70.31
17	15	60	900	65	84.38
18	10	80	600	95	79.09
19	10	40	600	35	70.28
20	15	60	300	65	83.92
21	15	60	1500	65	76.63
22	20	40	600	35	74.68
23	10	40	1200	95	77.36
24	20	40	600	95	81.24
25	20	80	600	95	74.09
26	10	80	600	35	81.51
27	20	40	1200	35	65.87

After evaluating the results using the program, the interactions between each parameter were determined and presented in ANOVA (Analysis of Variance) charts. The Prob>F value in the table was less than 0.05, indicating that the variables had a significant effect on the experiments. According to the analysis, it was determined that the parameters represented by B, C, D, AB, AD, BC, BD, CD, A^2, B^2, C^2, D^2 were effective in the model. Based on these data, it was concluded that the concentration, temperature, stirring rate, and leaching time linearly affect the reactivity of leaching in the water leaching of 600°C Alunite NaOH mixture, which are important factors affecting the applicability of leaching. The Adequate Precision value was 48.2331, indicating that the model discrimination was good. The Probs>F value showed that the lack of fit was also important in the model, but the predicted quadratic model equations had a good degree of fit. The quadratic and linear model equations obtained from multiple regression analysis are presented in the equations.

$$\text{Al Yield\%} = +6{,}44362 + 1{,}59073*A + 0{,}855141*B - 0{,}005739*C + 1{,}21282*D - 0{,}004131*A*B - 0{,}000087*A*C - 0{,}011837*A*D + 0{,}000325*B*C - 0{,}006801*B*D + 0{,}000048*C*D - 0{,}017687*A^2 - 0{,}003968*B^2 - 0{,}000012*C^2 - 0{,}004797*D^2$$

The agreement between the results obtained according to the model and the predicted values generated by the model can be seen in the predicted and actual value graph. It is seen that the values obtained because of the studies do not deviate from line on the graph (Fig. 5).

Table 4. ANOVA analysis results for water leaching based on the RSM-CCD model after roasting the sample+NaOH mixture at 600°C

Source	Adj SS	DF	Adj MS	F-Value	P-Value
Model	1208.07	14	86.29	216.29	0.0001
A	0.7597	1	0.7597	1.90	0.1928
B	268.87	1	268.87	673.94	0.0001
C	81.07	1	81.07	203.21	0.0001
D	47.29	1	47.29	118.54	0.0001
AB	2.73	1	2.73	6.84	0.0225
AC	0.2730	1	0.2730	0.6843	0.4243
AD	50.45	1	50.45	126.45	0.0001
BC	60.80	1	60.80	152.40	0.0001
BD	266.42	1	266.42	667.81	0.0001
CD	2.98	1	2.98	7.48	0.0181
A^2	4.17	1	4.17	10.46	0.0072
B^2	53.74	1	53.74	134.71	0.0001
C^2	25.10	1	25.10	62.91	0.0001
D^2	397.61	1	397.61	996.64	0.0001
Residual	4.79	12	0.3990		
Lack of Fit	4.79	10	0.4787		
Cor Total	1212.85	26			
Model	**R^2 =99.61%**		**R_{adj} = 99.14%**		**Std=0.6316**

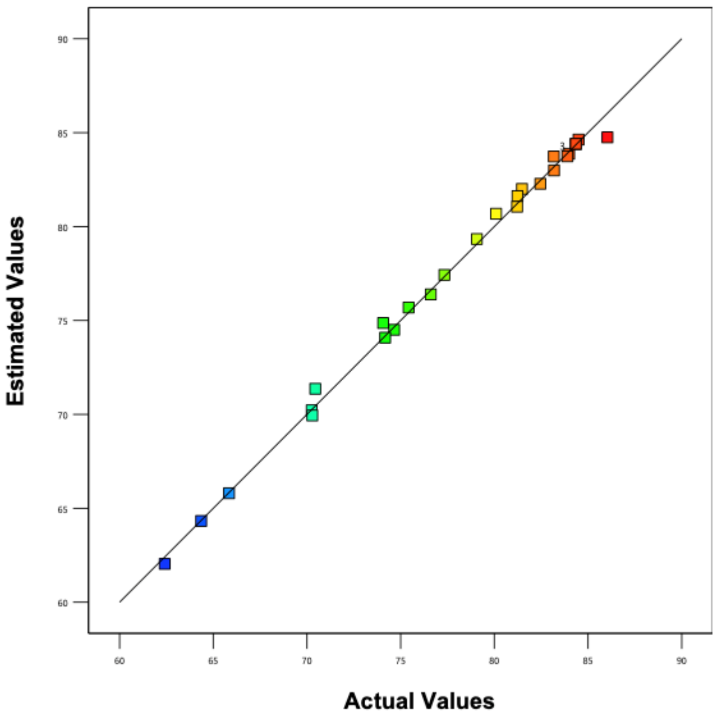

Fig. 5. Graph of predicted and calculated values for water leaching results based on the RSM-CCD model

Three-dimensional response surface plots were used to investigate the interactive effects of leaching factors on the Al leaching rate. As shown in Figure 6, the interaction effect of leaching temperature and solid ratio on Al leaching is significant compared to the other binary parameter plots. Graphs a, b, and c demonstrate that as the solid ratio decreases, the leaching efficiency increases. Graphs d and e indicate that an increase in temperature enhances the efficiency. In graph f, an increase in efficiency is observed at a leaching duration of 65 min. According to the graphs, the highest leaching efficiency was determined by evaluating lixiviant variables, including roasting at 600°C for 65 min, a stirring speed of 900 rpm, a reaction temperature of 100°C, and a solid concentration of 15%.

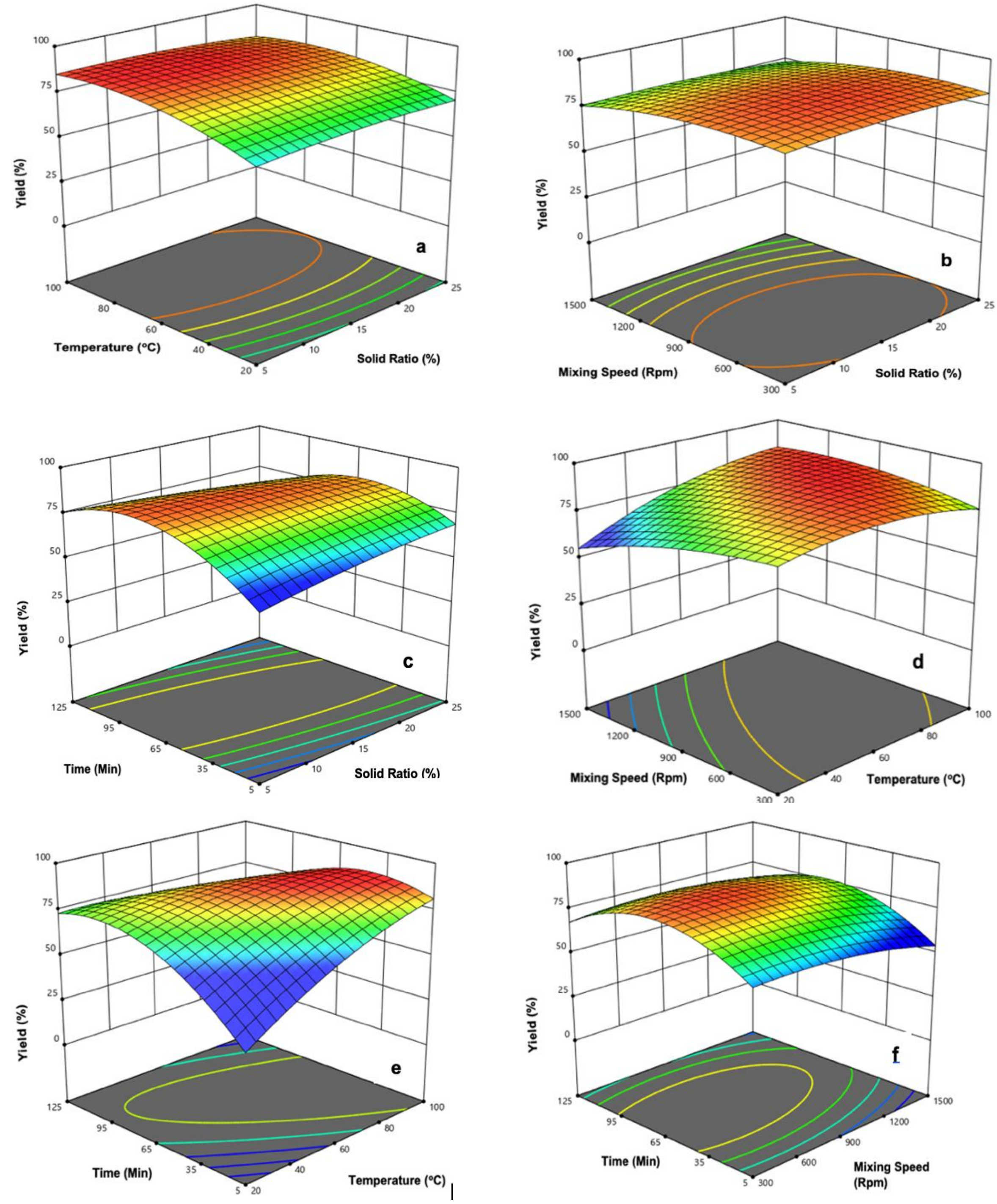

Fig. 7. 3D interaction effect ploth of solid ratio–temperature (a), solid ratio–stirring speed (b), solid ratio–leaching time (c), temperature–stirring speed (d), temperature–leaching time (e), and leaching time–stirring speed (f) parameters on aluminum recovery efficiency

The leachate solution with a volume of 2000-mL was prepared under optimal parameter conditions. The initial pH value was measured as 13.7 and subsequently reduced to 7 using 15% HCl, initiating precipitation. To ensure sufficient precipitate formation, the leachate was stirred at 300 rpm for 1 hour. The precipitate formed was filtered, and solid–liquid separation was performed. To obtain alumina from aluminium hydroxide, the recovered precipitate was washed with hot water, dried, and then roasted at temperatures of 700°C, 1000°C, and 1100°C for durations ranging from 60 to 240 min. The reaction (6) for the resulting.

$$2Al(OH)_3 \rightarrow Al_2O_3 + 3H_2O \quad (6)$$

According to the analyses, the obtained product by roasting with precipitation at 1100°C for 240 min. was identified as the α-Al_2O_3 polymorph based on its morphological properties, which were observed in the XRD patterns (Fig. 7).

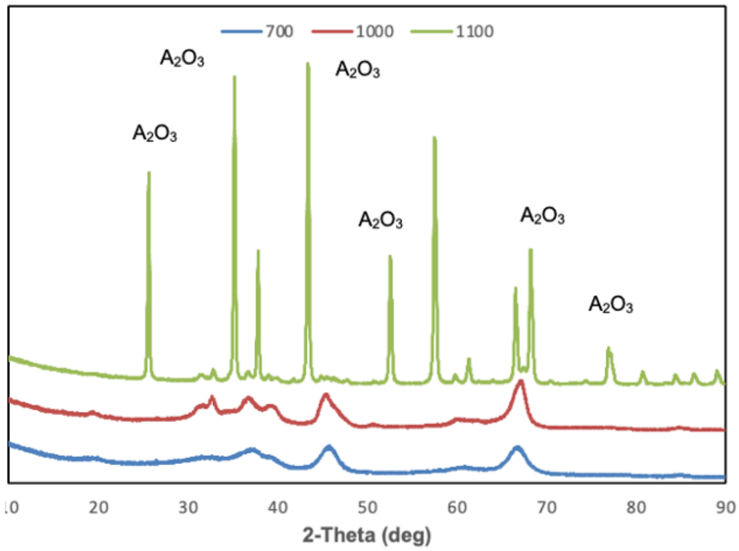

Fig. 7. XRD patterns with different roasting temperature

SEM and EDX spectra result of alunite ore and alumina samples are presented in Figure 8 and Figure 9. The findings show that the SEM examinations of the alumina sample obtained as a product are consistent with the XRD analysis. The observations of tests were revealed agglomerated α-alumina particles, polycrystalline crystals, and bubble structures with rounded corners. when compared with Turk et al. (2020) and Shi et al. (2021), the particle shapes of the samples were quite consistent and regular. The elemental distribution of the regions selected for EDX in the alunite sample; Si 47.21%, O 18.96%, Al 14.56%, C 2.34%, K 7.24%, S 9.69%. In the alumina sample; Al 39.24%, Si 26.57%, O 20.87%, C 13.32%. EDX spectra was showed that after the application of alumina production methods, Al level was increased and S and K levels were purified, respectively.

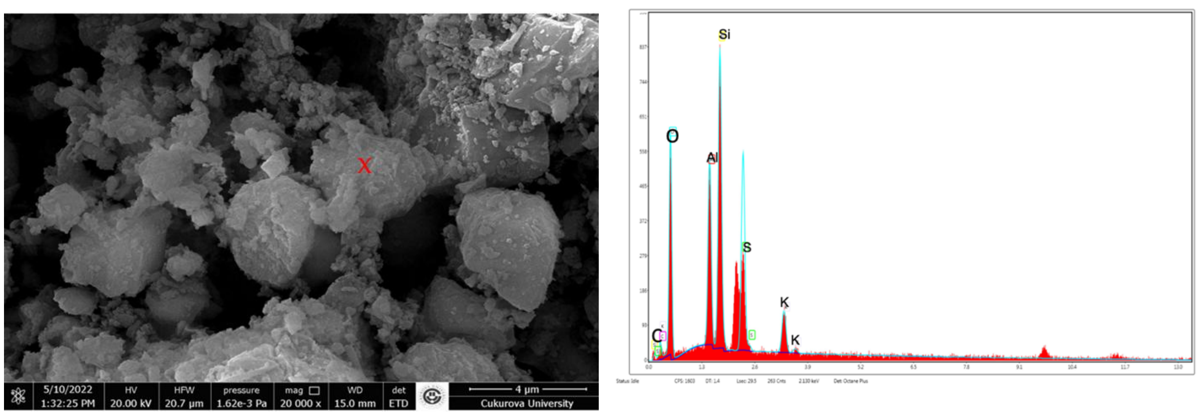

Fig. 8. SEM analysis and micro chemical analysis (EDX) of alunite samples (mag. 20000×), (mag. 5000×)

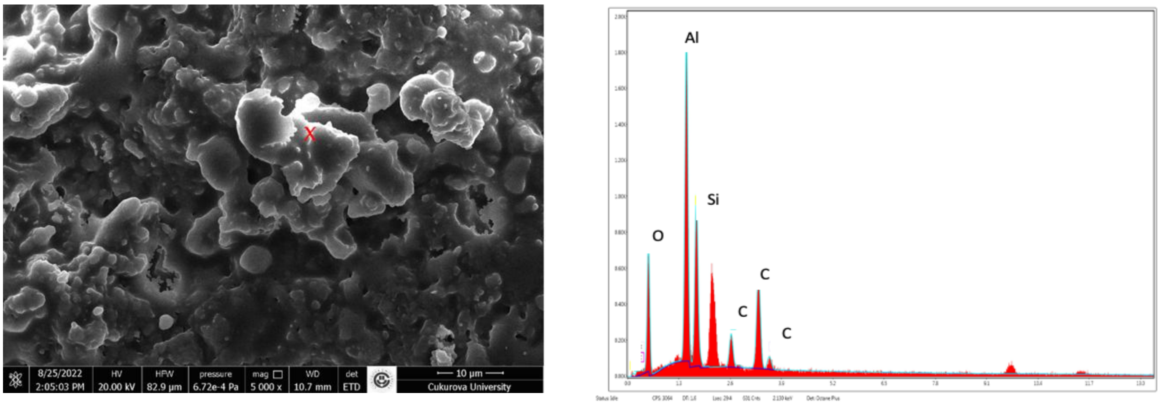

Fig. 9. SEM analysis and micro chemical analysis (EDX) of alumina samples (mag. 5000×)

4. Conclusions

The focus of this research was to analyse effects of thermal conditions with dry NaOH on aluminium purify from the alunite ore Kutahya/Türkiye, which could serve as a critical alumina production under high-efficiency conditions with water leaching. In the initial stage, pre-treatment studies were conducted, where after a thermal activation process at 800°C for 30 min, potassium, which could cause contamination in aluminium recovery, was removed by water leaching for 1 hour. In the second stage, the leaching residue was roasted with a 1:1 NaOH mixture at 600°C for 60 min, followed by tests based on the central composite design of response surface methodology, using parameters such as temperature, stirring speed, leaching time, and solid ratio. The optimal leaching conditions were found to be a temperature of 100°C, stirring speed of 900 rpm, solid ratio of 15%, and leaching time of 65 min. The results indicated that temperature, stirring speed, and duration had a significant effect on leaching efficiency, and there were interactive relationships between the parameters of solid ratio-temperature, solid ratio-duration, temperature-stirring speed, temperature-duration, and stirring speed-duration. To produce high-purity α-alumina, a neutral pH after roasting was sufficient. The precipitate was then sintered at 1100°C for 240 min, yielding a recovery rate of 89.89% and a purity grade of 91.24%. Different methods used for aluminium production from sources other than bauxite can contribute to improving the sustainability of the processes, both environmentally and economically. The combined methods for alumina production from alunite have shown promising results, and the development of more critically efficient methods in the future is crucial for the future of the industry.

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