

Research Article

Extraction of Aluminum Oxide from Local Kaolin Clay Deposits in Ethiopia

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Abstract

The need for aluminum is growing worldwide, which has sparked interest in finding alternate ways to make alumina from materials other than bauxite, particularly clays. This article examines the use of sodium carbonate as a leaching agent in the lime sintering process to recover alumina from kaolin. Excavated from Tarmaber, Ethiopia, kaolin clay contains a content of 32.88%, which has a relatively good composition. Collecting and grounding raw kaolin to micrometer-level particle size is the first task. The recovery of kaolin alumina was studied at sintering temperatures of ($T_1=800$ °C, $T_2=900$ °C & $T_3=1000$ °C) at different sintering times of ($t_1=1$ hr, $t_2=2$ hrs & $t_3=3$ hrs). After the raw material was burned at the given temperatures and times, it was cooled for the night in the furnace and leached with different concentrations of sodium carbonate ($M_1=50$ g/l, $M_2=60$ g/l & $M_3=70$ g/l). The response surface methodology (RSM) in combination with the central composite design was used to optimize the operating parameters. The optimization result shows that the optimal conditions were a calcination temperature of 953.84 °C, a sintering time of 2.99 h, and a leaching agent concentration of 70 g/l. At this optimal condition, the yield of Alumina was 1.05 g of 20 g of Kaolin clay. The resulting Alumina was crystalline in structure (from XRD analysis), contains 89.05% Al_2O_3 (from silicate analysis) and a large broad band between 400-1000 cm^{-1} is attributed to Al-O-Al stretching of Alumina (from FT-IR analysis). So, it is possible to conclude that alumina production from no bauxite ores is possible.

Keywords

Alumina, Kaolin, Sintering, Leaching, Carbonization, Calcination

1. Introduction

Aluminum is known to be the third most abundant element and the most abundant metallic element that covers 8 % by weight of the earth's crust and is found in oxide form [1]. Alumina has been widely used in advanced technological applications, including its use for the processing of high-quality insulators, semiconductors, high explosive charges, and microelectronic materials [2]. It is also used for abrasive [3], refractories [4], glass [5], and ceramic [6],

medical industries [6], military and protective Equipment's [1] and gem industry [7].

Of course, alumina comes from a variety of sources. Among these, the majority of ores of alumina include feldspar, kaolin clay, and bauxite. Bauxite, or $Al_2O_3 \cdot 2H_2O$, is the primary raw material used in the Bayer process to produce alumina. It contains between 40 and 60 percent Al_2O_3 [6]. However, as an alternative to bauxite, kaolinitic clay has been

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Received: 10 August 2024; Accepted: 5 September 2024; Published: 18 October 2024



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processed for the production of alumina due to the fact that bauxite is already on the verge of disappearing from the earth's crust. In many tropical and sub-tropical soils, kaolin is a typical weathering product [8]. It is made up of the mineral kaolinite ($\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$) as well as numerous other minerals such as feldspar, hematite, and carbonates [9]. Researchers are more interested in finding alternate methods to produce alumina from low-grade minerals than from bauxite sources, which is the primary source of aluminum oxide, due to the growing global demand for alumina. Kaolin is the primary low-grade mineral that produces alumina among these minerals [10].

One of the most extensively dispersed gifts from nature is kaolin, and its products are crucial to the progress of a nation's growth. Aluminum oxide, sometimes known as alumina, is one of the primary ingredients of kaolin [11]. Tetrahedral silica and octahedral alumina single sheets joined by hydrogen bonding make up the structural unit of kaolinite [11]. Alumina may be extracted from kaolin using a variety of techniques. These include the lime sintering process, the leaching of kaolin by bases [13], the leaching of kaolin by acids [12], and various techniques for the extraction of alumina [14]. Using nitric acid, hydrochloric acid, or sulfuric acid, the acid leaching procedure dissolves alumina alone. Acid leaching is a widely employed method in extractive metallurgy because of its high extraction efficiency, environmentally acceptable process, low energy consumption, and capacity to handle low-grade ores [15]. From an industrial perspective, the leaching of calcined kaolin clay in hydrochloric acid offers a number of noteworthy benefits, such as

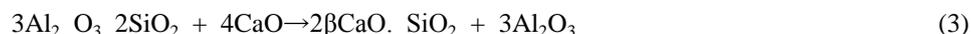
Limestone Calcinations



The binding of silica to the β form of calcium orthosilicate



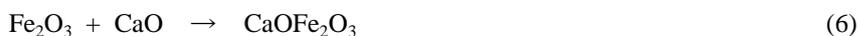
Destruction of Mullite



The Transition of Alumina to the Extracted Form



Binding of Iron Oxide

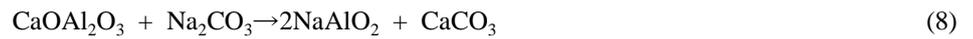


The Leaching Process

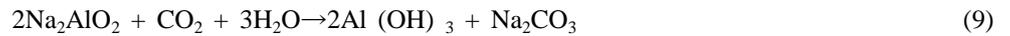


the low solubility of silica, the potential for $\text{AlCl}_3 - 6\text{H}_2\text{O}$ to crystallize selectively, and the ability to recover hydrochloric acid from waste products for further use in the process [16]. However, in comparison, the cost is higher. Although leaching by bases is a rather cheap method, it is ineffective for extracting materials. This indicates that it produces alumina of low quality and little amount. The sintering technique can be used to extract alumina by sintering kaolin, lime, or soda. After that, silica and alumina are extracted using a heated, diluted sodium carbonate solution. It yields a product of a qualifying sort, or one that is comparable to that obtained using the acid-leaching extraction technique, and is competitively priced [17]. This research project's main objective was to extract aluminum oxide from Ethiopia's kaolin clay deposits by employing a sodium carbonate solution as a leaching agent and the lime sintering process. The lime sintering process may extract alumina from kaolin by the following reactions [17].

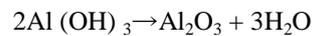
In general, a number of factors, such as the calcination duration, calcination temperature, and concentration of the leaching agent sodium carbonate, affect the extraction of alumina from calcined kaolin by the lime sintering process [17]. Limestone was acquired at the Merhabite, Jemma River site, while kaolin was gathered from several locations around Ethiopia, especially the Amhara area (Tarmaber, Mehalemeda, and Shewa Robet). Over 90% of the 45 million tons of aluminum oxide produced worldwide each year are utilized in the manufacturing of aluminum metal. Ethiopia imports 200 tons or more of aluminum oxide annually on average from outside [18].



Carbonation



Calcination



2. Materials and Methods

2.1. Materials

A 20 g sample of kaolin was taken from the Tarmaber mine in Ethiopia, while a limestone sample was taken from the Merhabeti Jemma river mineral site. As a leaching agent, chemically pure sodium carbonate was employed.

2.1.1. Description of the Study Area

Among many samples, the one that has a high aluminum oxide composition (Tarmaber sample specifically *Kosober's*) was taken as a sample for the extraction of aluminum oxide. Limestone is found in large amounts in different parts of the country, for example, in the Amhara region, Merhabeti around the Jemma river mineral site which was taken as a sample for this research work.

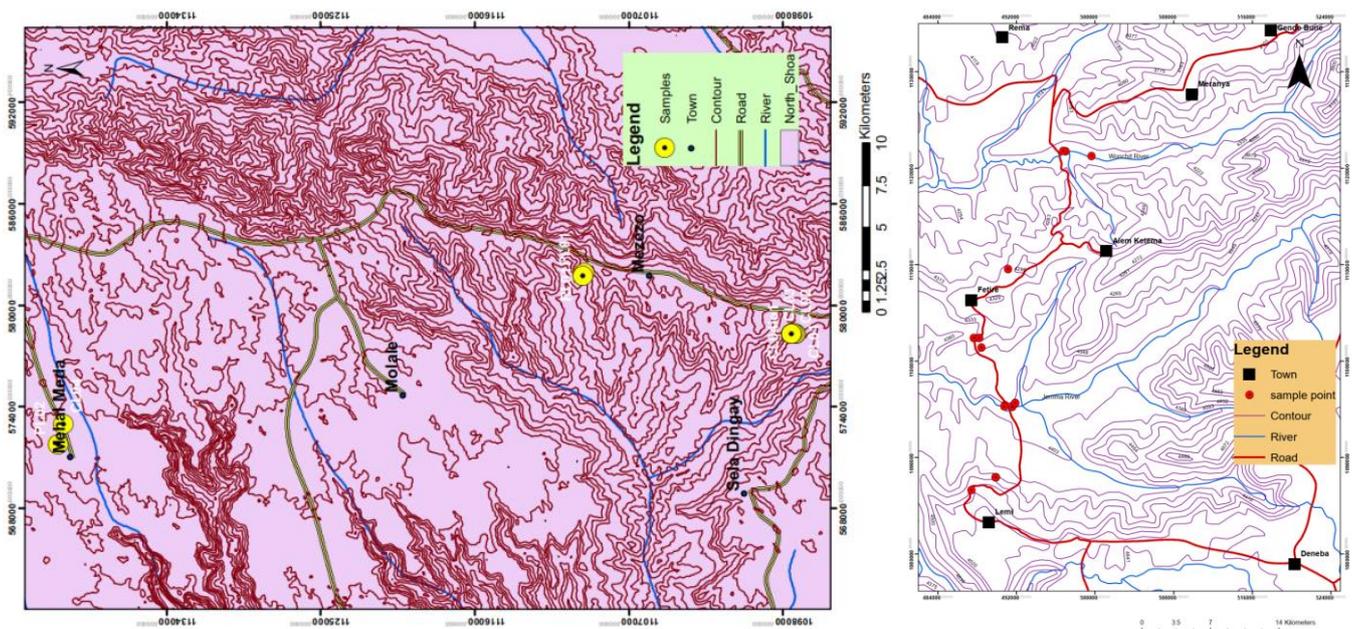


Figure 1. Kaolin and limestone location map in north Shewa.

2.1.2. Sample Characterization

The oxide composition of kaolin and limestone was investigated using the silicate analysis method at the Ethiopian Ministry of Mining and Petroleum.

2.2. Methods

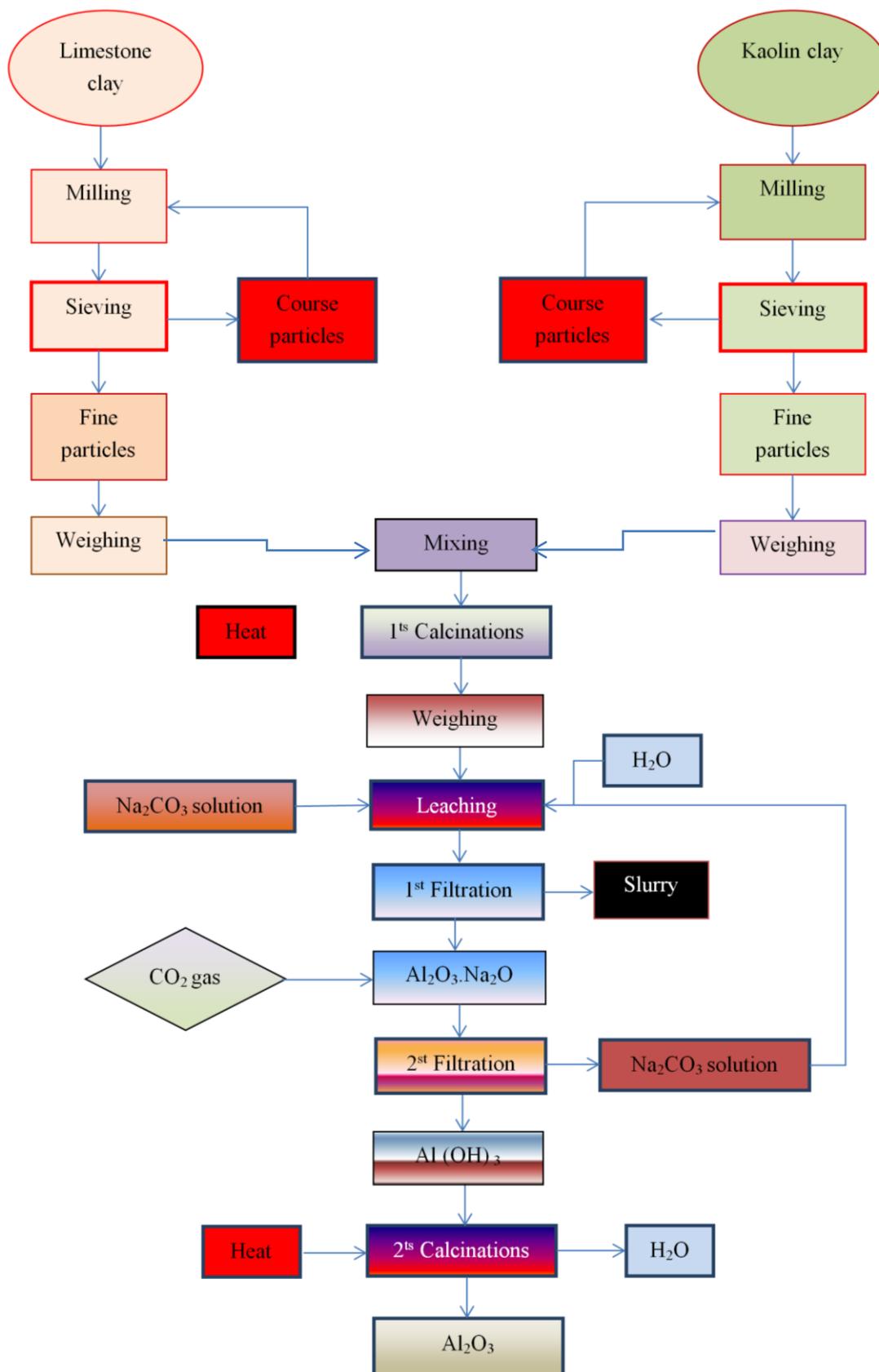


Figure 2. Process flow diagram for the production of alumina from kaolin by the lime sintering process.

2.2.1. Sample Collection and Preparation



Figure 3. Raw Kaolin (A), Kaolin Powder (B), Raw Limestone (C) and Fine Limestone Powder (D).

Figure 3 above shows the raw minerals and their ground powder. Kaolin from Tarmaber Ethiopia and limestone from



Figure 4. Kaolin powder plus limestone powder gives the normal raw material mixture.

2.2.3. First Phase Calibrations



Figure 5. (A) Mixture of Geo Minerals and (B) Calcined Geo Mineral.

After the raw minerals were mixed, the calcination process began. Figure 5. A and B show the mixed raw material and the calcined one in the muffle furnace. Blended raw materials were calcined at three different temperatures (800 °C, 900 °C, and

Merhabete, Jemma River, Ethiopia, were taken as the working geomineral sample. Unnecessary impurities from the georaw materials were removed by sieving and drying unit operations. The raw materials were then crushed and milled to the required particle size distributions using the universal crusher miller and then separated into the required size. The Kaolin Clay ($Al_4(OH)_8Si_4O_{10}$) is transformed into Meta Kaolin Clay ($Al_4(OH)_{8-2x}OSi_4O_{10}$) and Lime stone clay $CaCO_3$ was changed into CaO by heating the clay mixture to about a temperature range of 800 °C to 1000 °C [9].

2.2.2. Grinding, Sieving, and Mixing of Raw Materials

Following raw material collection and characterization, both kaolin and limestone were ground separately and sieved through a 63- μ m size sieve. As Figure 4 below shows, the sieved raw materials, with a size of 63 μ m, were mixed and mixed with limestone kaolin clay. To achieve uniform proportion, this procedure should be carried out with caution.

1000 °C) to remove moisture content and some unnecessary heat sensitive impurities before proceeding to the chemical reaction.

2.2.4. Cooling

As mentioned above, calcination takes place at different temperatures and also at different times. Once the calcination time was completed, the furnace was cooled down for annealing of the calcined material to achieve self-disintegration. It may take a while.

2.2.5. Leaching

The leaching process began after the raw material was cooled and reached room temperature in the furnace. At 70 °C, the calcined material and leaching agent were mixed with a proportionate amount of distilled water. Once the distilled water has reached 70 °C, various amounts of leaching agent (sodium carbonate) are added to the heated water and thoroughly mixed. After sodium carbonate has been dissolved in heated water, the solution is added to the main raw material

and thoroughly stirred with a stirrer. Finally, for 30 min, gently shake the mixture using a shaker that has a shaking capacity of around 600 rpm [9].

2.2.6. First-Phase Filtration

Once the leaching process was completed, the first filtration process was processed using a vacuum filters. Separates the filtrate from the slurry. Sparging/washing of the slurry with around 100 ml of distilled water was performed to minimize the Aluminum oxide wastage.

2.2.7. Carbonization

This was the process of incorporating carbon dioxide into the filtrate solution. This process was carried out after the filtrate had reached 80 °C [19]. Sodium aluminates are present in the filtrate solution obtained from the first filtration process. Sodium Aluminates solution was converted into an Aluminum hydroxide precipitate and sodium carbonate solution during carbon dioxide sparging.

2.2.8. Second Phase Filtration

In the filtrate solution, precipitation developed following the completion of the carbonization procedure. A vacuum pump filter was used to extract the desired product, or precipitate, from the solution.

2.2.9. Second-Phase Calcinations

After adding aluminum hydroxide to the crucibles, the second calcinations were carried out in a furnace at 1000 °C for one hour. During this process, the water in the aluminum hydroxide was removed, yielding pure aluminum oxide.

2.3. Experimental Design

A fully randomized design was used to conduct the experiment. A central composite design was used to identify the ideal aluminum oxide manufacturing conditions (CCD). An analysis

was conducted on the key variables (sintering temperature, sintering time, and leaching solution concentration) across 20 tests. For the purpose of designing experiments, analyzing data, and creating models, the software design expert (version 7.0) was utilized. Analysis of variance (ANOVA) was used to establish the significance of the outcome.



Figure 6. Leaching (A), 1st Filtration (B), Carbonation (C), 2nd Filtration (D), Aluminum Hydroxide (E) and Aluminum Oxide (F).

3. Result and Discussion

3.1. Characterization of the Raw Materials

The chemical composition of kaolin and limestone clay was determined using the complete silicate analysis method. Table 1; summarizes the results of the analysis. Since kaolin clay and limestone contain enough composition of alumina and calcium oxide, respectively, it is possible to produce alumina from those raw materials [20].

Table 1. Silicate analysis results of different kaolin clays.

Oxide	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	Na ₂ O	K ₂ O	MnO	P ₂ O ₅	TiO ₂	H ₂ O	LOI
Kaolin, %	46.3	32.88	3.26	1.12	0.1	0.14	<0.01	<0.01	0.34	0.09	5.04	10.7
Limestone, %	5.6	<0.01	<0.01	52.02	0.42	<0.01	<0.01	0.12	0.2	<0.01	0.22	41.38

3.2. Product Characterization Results

3.2.1. X-Ray Diffraction Analysis

The intense and sharp diffraction peaks reveal the crystal-

line nature of aluminum oxide (Al₂O₃). The Al₂O₃ XRD pattern found at 900 °C for 19.2 min with 70 g/l leaching agent concentration is given in Figure 7. The XRD pattern of the aluminum oxide product exhibits peaks at 25.6, 35.2, 37.84, 43.4, 46.44°, 52.6°, 57.56°, 61.3°, 66.64°, and 68.24°. The patterns indicate the presence of Aluminum oxide in the

sample in phases of Al_2O_3 , known calcite having a crystal structure of trigonal (hexagonal axes) with cell parameters $a = 19.91370$ and $c = 19.54700$ Å parameters. The result obtained

is in agreement with other results and ensures that the crystalline structure of Alumina is revealed using any deposition methods at high temperatures [17].

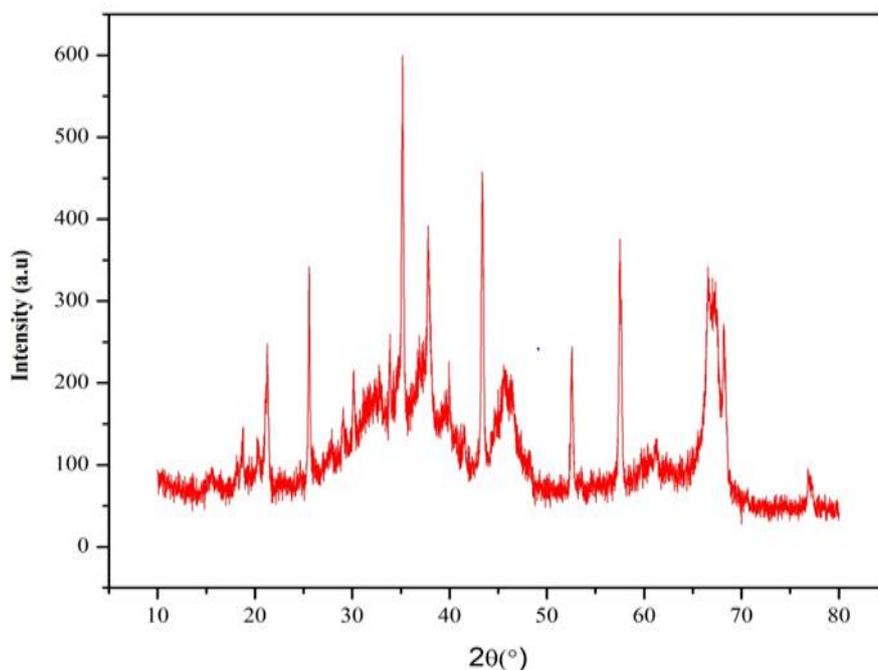


Figure 7. XRD pattern of produced Aluminum oxide.

3.2.2. Silicate Analysis Results of Produced Aluminum Oxide

The chemical analysis of the final calcined aluminum oxide has been studied by using the silicate analysis method. This test shows the chemical composition of any material in oxide form. Table 2. Shows the chemical composition of the aluminum oxide produced. The result shows that the amount of aluminum oxide (89.05 wt %) was enough. The comparable result found in the literature is ranged from (88- 97) wt% [21].

Table 2. Chemical composition of produced Alumina.

Oxides	Oxide wt (%)
Na_2O	1.31
MgO	1.11
Al_2O_3	89.05
SiO_2	1.1
P_2O_5	0.89

Oxides	Oxide wt (%)
SO_3	0.95
Cl_2	0.96
CaO	2.31
FeO	1.24
ZnO	1.08

3.3. The Influence of Parameters on Aluminum Oxide Amount

3.3.1. Time Influence on Aluminum Oxide Production

The main goal of this experiment was to determine the time at which the maximum aluminum oxide value occurs. As the figure below shows, when the time increases from 1 hour to 3 hours, the product decreases from 0.98 to 0.86 grams. This shows that as the time of calcination increases, the produced product, alumina, decreases constantly.

Design-Expert® Software

aluminium oxide

● Design Points

X1 = A: time

Actual Factors

B: temperature = 900.00

C: leaching agent = 60.00

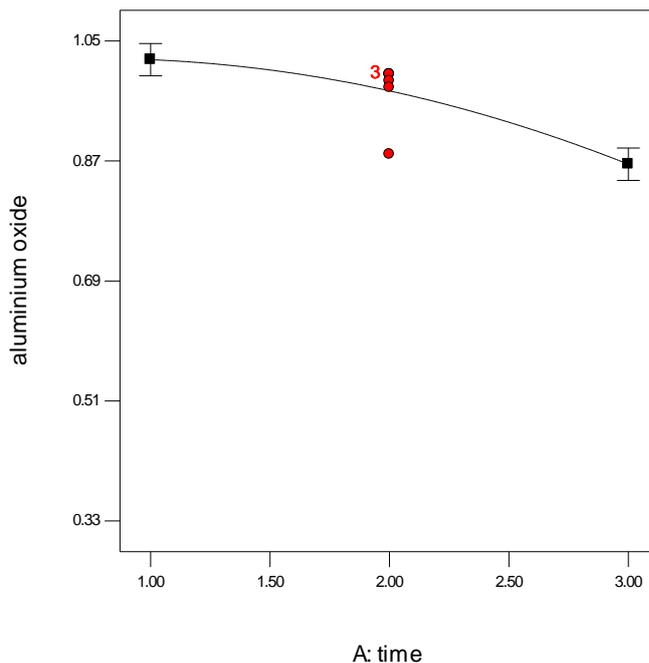


Figure 8. Effect of time on alumina product.

3.3.2. Influence of Temperature on Aluminum Oxide Production

Aluminum oxide production was also significantly affected by the sintering temperature. The obtained result is shown in

Figure 9 below. It shows that as the sintering temperature increases from 800 to 900 °C, the obtained result also increases from 0.77 g to 1.01 g, but beyond 900 °C, the Alumina product becomes decreased slowly.

Design-Expert® Software

aluminium oxide

● Design Points

X1 = B: temperature

Actual Factors

A: time = 2.00

C: leaching agent = 60.00

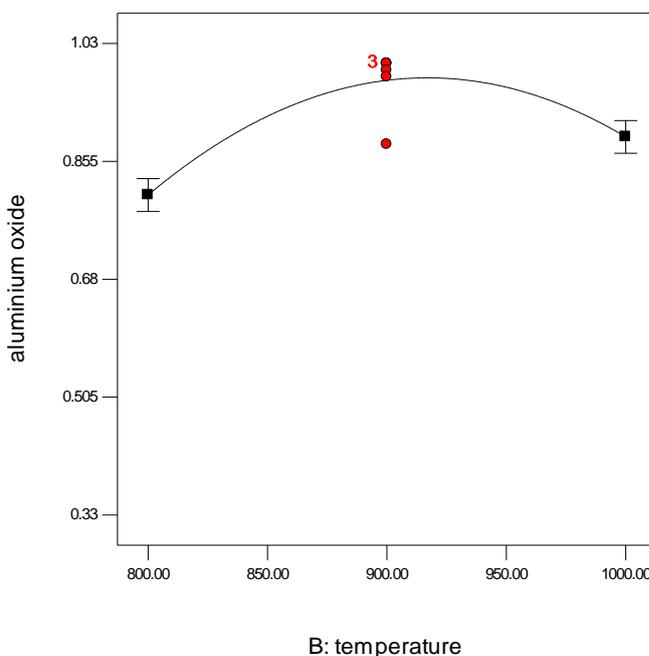


Figure 9. Effect of temperature on alumina product.

3.3.3. Leaching Agent Concentration Effect

One of the main factors that should be considered during the extraction of aluminum oxide from local Kaolin clay was the concentration of the leaching agent (the amount of sodium car-

bonate) in a given amount of kaolin clay and locally obtained Limestone clay. As Figure 10. Shows below, increasing the concentration of sodium carbonate from 50 g/l to 60 g/l increases the extraction rate of alumina from 0.77 g to 1.00 g. But beyond 60 g/l, the extraction rate of alumina slowly decreases.

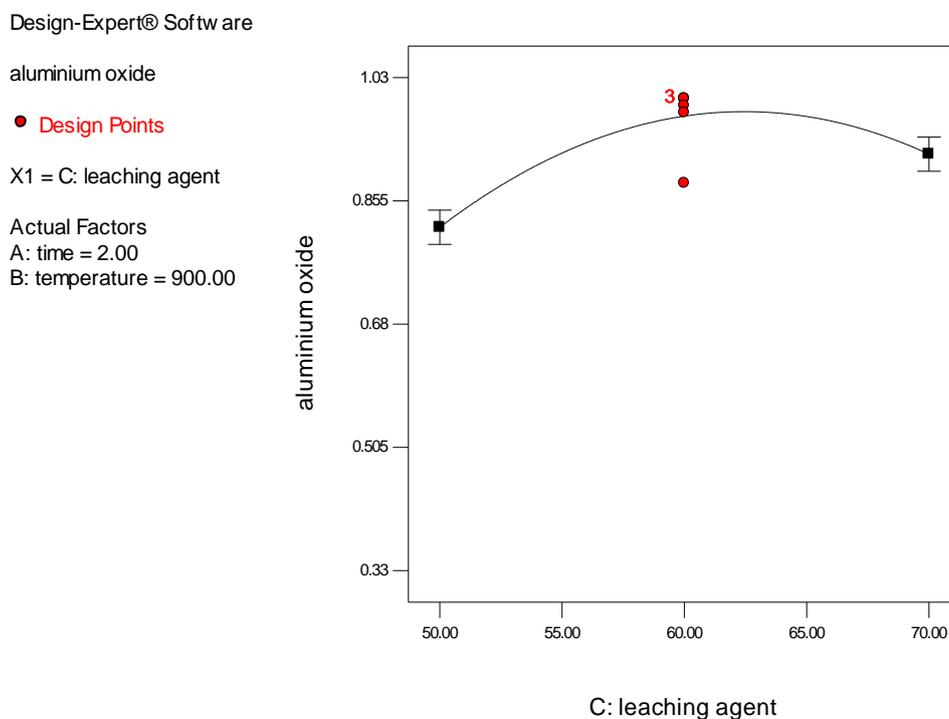


Figure 10. Effect of leaching agent concentration on alumina product.

3.4. Response Surface Method of Analysis

In this research design, an expert (response surface method of analysis with central composite) was used for modeling statistical analysis and optimization of significant parameters (sintering time, sintering temperature, and leaching agent concentration) for the production of aluminum oxide. The investigation of the optimal level and the influence of every parameter on the quality and quantity of aluminum oxide were carried out by the calcination process using a muffle furnace. However, 17 run experiments were performed using central composite design (CCD) to deter-

mine the maximum or minimum amount of aluminum oxide, and optimization of those parameters was conducted with design expert 7.0 (trial version); the results generated from CCD are listed in Table 3 below. Hence, in this paper, the design expert analysis was performed using the Aluminum oxide response value.

3.4.1. Analysis of Variance

In this research, the result of the analysis experiment was obtained using variance analysis with the analysis of the significant model term from the quadratic regression model.

Table 3. ANOVA for response surface quadratic model.

Source	Sum of Squares	Df	Mean Square	F-Value	P-value Prob > F	
Model	0.92	9	0.1	83.95	< 0.0001	Significant
A- sintering temperatures	0.025	1	0.025	20.94	0.001	
B- sintering time	0.084	1	0.084	69.2	< 0.0001	

Source	Sum of Squares	Df	Mean Square	F-Value	P-value Prob > F	
C-leaching agent concen	0.037	1	0.037	30.34	0.0003	
AB	5×10^{-005}	1	5×10^{-005}	0.041	0.8432	
AC	0.048	1	0.048	39.6	< 0.0001	
BC	0.36	1	0.36	297.71	< 0.0001	
A ²	0.23	1	0.23	191.93	< 0.0001	
B ²	0.014	1	0.014	11.91	0.0062	
C ²	0.16	1	0.16	133.22	< 0.0001	
Residual	0.012	10	1.21×10^{-3}			
Lack of Fit	9.844×10^{-004}	5	1.964×10^{-004}	0.088	0.9907	not significant
Pure Error	0.011	5	2.23×10^{-003}			
Cor Total	0.93	19				

3.4.2. Model Equation Development

The model equation that correlates the response process variables, in terms of coded factors, is given in Equation 4.1. The quadratic model was selected to predict aluminum oxide production. Design-expert software has been suggested to select a quadratic model in the analysis because the highest values of R², adjusted R², and predicted R² were obtained and the model is not aliased which is shown below in Table 4. The final equation in terms of coded factors,

$$\begin{aligned} \text{Aluminum oxide (g)} = & +0.98 + 0.04 * A - 0.078 * B + 0.052 * \\ & C - 2.500E-003 * A * B + 0.078 * A * C + 0.21 * B * C - 0.13 * \\ & A^2 - 0.032 * B^2 - 0.11 * C^2 \end{aligned} \quad (10)$$

where A- calcination temperature, B- sintering time, and C- leaching agent concentration. From this equation, the negative coefficient value indicated that negatively affects the Al₂O₃ product, while the positive coefficient values represent the positive relationship of that factor with the Al₂O₃ product.

Table 4. Fit summary of the response.

Source	Std. dev.	R ²	Adjusted R ²	Predicted R ²	PRESS	
Linear	0.22	0.1574	-0.0006	-0.4020	1.3	
2FI	0.17	0.5981	0.4126	0.0272	0.9	
Quadratic	0.035	0.9869	0.9775	0.9743	0.024	Suggested
Cubic	0.043	0.9878	0.9614	0.9385	0.057	Aliased

3.4.3. Model Adequacy Checking

The regression model developed is used to find the predicted value of the aluminum oxide product and has to be examined for its consistency. The quality of the model could be evaluated from their coefficients of correlation. The value of R-squared for the developed correlation is 0.9869 revealing that the model agrees well with the experimental data and can be used to predict the response accurately. If the R-squared value is closer to 1.0 it indicates that the regression line perfectly fits the data.

Similar to this investigation, the obtained R-squared was 0.9869, which was close to 1. The "Pred R-Squared" of 0.9743 is in reasonable agreement with the "Adj R-Squared" of 0.9752. The signal-to-noise ratio is measured by the statistical parameter called adequate precision. Adequate precision greater than four is desirable, indicating that the model gives as much as appropriate performance in predicting the Aluminum oxide product. In this study, adequate precision is found to be 27.879. A smaller value of the coefficient of variation (CV = 4.38%) that measures the variability of the

residuals as a percentage of the mean of the response variable shows the great precision and quite high reliability of the experiments carried out, as summarized in Table 5.

Std. Dev.	0.035	R-Squared	0.9869
PRESS	0.024	Adeq Precision	27.879

Table 5. Measurements of model adequacy.

Std. Dev.	0.035	R-Squared	0.9869
Mean	0.79	Adj R-Squared	0.9752
C.V. %	4.38	Pred R-Squared	0.9743

Analysis of the diagnostic tool was used to evaluate the adequacy of the model. The accuracy of the model was verified by examining the residual plots. Residuals are the deviations between the actual and predicted values of the aluminum oxide product. A straight line has coincided with the normal plot of residual and has witnessed its excellent adequacy to represent the aluminum oxide product as observed in Figure 11 below.

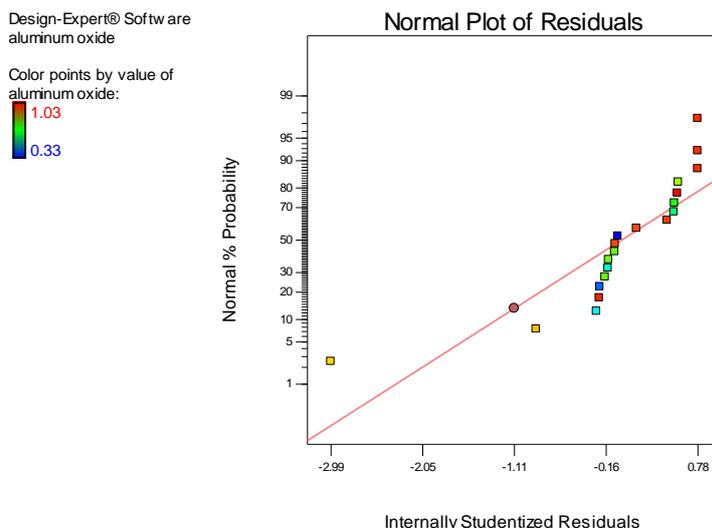


Figure 11. Normal probability plot of residuals.

A plot of the residuals versus the rising predicted response value tests the assumption of constant variance. Figure 12 show that random scatter justifies no need for any alteration to minimize personal error. The residual plots of the model are

randomly distributed without any trends. This result indicates good predictions of the maximum response along with constant variance and the adequacy of the quadratic models.

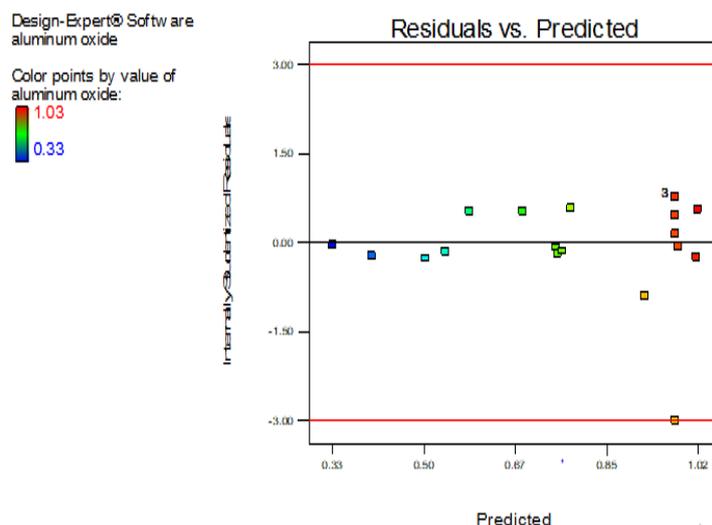


Figure 12. Residual versus predicted values plot.

Residuals may be plotted based on run order as shown in Figure 13 which indicates a plot of internally studentized residuals versus the run number that conducts a t-test for each residual. Points that drop outside the red limits are considered

outliers. Therefore, all the points in Figure are within the range of the model assumptions and there are no outliers observed.

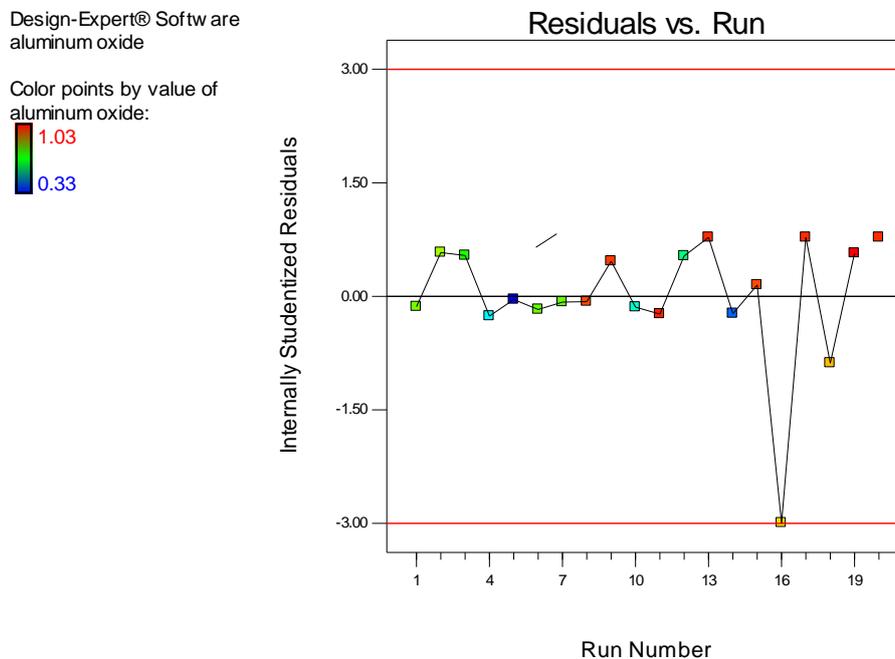


Figure 13. Plot of residuals versus the run number.

Figure 14, Shows the actual values versus the predicted values for aluminum oxide products using the developed model equation. The graph of predicted versus experimental

values indicates that the model was successful in capturing the correlation between the reaction parameters and the response with correlation coefficients $R^2 = 0.9869$.

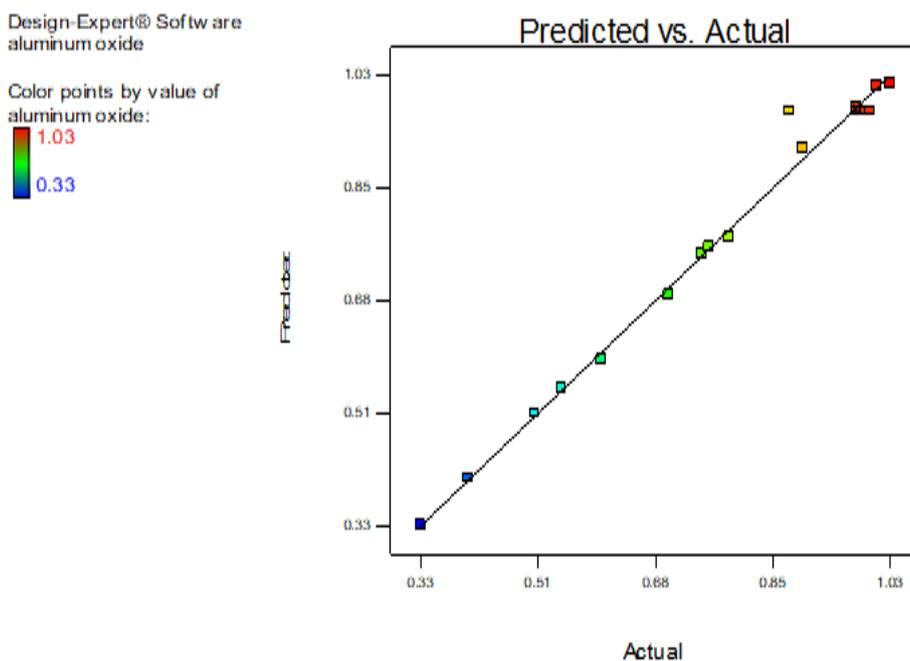


Figure 14. Plot of predicted versus actual value.

4. Conclusion

In this study, the production of Aluminum oxide from locally available raw materials, such as kaolin clay and limestone, was investigated based on the batch-type calcination method. The aluminum oxide value was influenced by three main process parameters, such as the sintering time, the calcination temperature and the concentration of the leaching agent. When the sintering time was varied from 1 to 3 hours, the amount of aluminum oxide decreases from 0.98 to 0.86 gram. This shows that time and alumina products are inversely related to each other. When the temperature increased from 800 °C to 900 °C the amount of Aluminum oxide become increased from 0.77 g to 1.01 g. But it becomes decreased beyond 900 °C. The concentration of the leaching agent and the amount of sodium carbonate in the given extraction process also have a high effect on the amount of aluminum oxide produced. As the amount of sodium carbonate increased from 50 g/l to 60 g/l, the amount of product also increased while the Alumina product becomes constant, then decreased beyond 60 g/l. The mathematical modeling developed for the production of aluminum oxide using design experiments appears to be an effective tool for the prediction and understanding of the interaction effect between factors. From the developed model, it is better to conclude that the three factors (sintering time, sintering temperature, and leaching agent concentration) have an interaction effect. The optimal aluminum oxide product (1.05 g) was obtained at 2.99 h of sintering time, 953.84 °C calcination temperature, and 70 g/l sodium carbonate concentration. The Aluminum oxide produced is crystalline and contains 89.05% alumina in it. The value of R-squared for the developed correlation is 0.9869 revealing that the model agrees well with the experimental data and can be used to predict the response accurately.

Abbreviations

T	Temperature
t	Time
FTIR	Fourier Transform Infrared Spectroscopy
XRD	X-ray Diffraction
RSM	Response Surface Methodology

Author Contributions

Workie Tsegaye Belay: Conceptualization, Data curation, Resources, Writing – original draft

Mengisst Kassaw Melesse: Investigation, Methodology, Visualization, Writing – review & editing

Amha Betemariam Gizmu: Formal Analysis, Software, Supervision, Validation

Funding

This research received no external funding.

Data Availability Statement

The data presented in this study are available on request from the corresponding author. The data are not publicly available due to the value of further research.

Conflicts of Interest

The authors declare no conflicts of interest.

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