






Kinetics Study of Alumina Leaching from Ogbunike Clay Using Hydrochloric Acid

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ABSTRACT

The kinetics study of alumina leaching from ogbunike clay was carried out using a solution of hydrochloric acid (HCl) for alumina dissolution. Variations in process parameters such as calcination temperature, leaching temperature, acid concentration, particle size, and solid-to-liquid ratio were considered for the leaching of alumina. The shrinking core model was used to analyze the experimental data. Alumina removal is feasible with 24.035% extractable alumina, and this can improve the economic value of ogbunike clay, which can be an alternative raw material in Nigeria's aluminum industries. According to the data the dissolution rate increases with increasing calcination temperature, leaching temperature and acid concentration and decreasing particle size and solid-to-liquid ratio. The statistical and graphical analysis revealed that the dissolution followed the product layer diffusion-controlled semi-empirical kinetic model. The activation energy was calculated to be 18.36 kJ/mol for HCl. The optimum conditions for the dissolution of the alumina in HCl include activation temperature of 700 °C for 60 minutes, leaching temperature of 90 °C, HCl concentration of 3 M, particle size of 75 µm, and the solid-liquid ratio of 0.2 g/ml yielding the alumina dissolution rate of 84.72%. The kinetic data can be used to construct large-scale manufacturing equipment.

Keywords: alumina, dissolution, kinetics, ogbunike clay, shrinking core

INTRODUCTION

Clay is a fine earthy powder produced by the weathering and disintegrating of granite and feldspathic rocks. They are anhydrous complex compounds of alumina (Al_2O_3) and silica (SiO_2) complexes with different quantities of iron, organic matter materials, and residual minerals. Clay materials are suitable substances as a source of metals and adsorbents because of their inexpensive cost, abundance on most continents, high absorption properties, high solubility in acidic solutions, and potential for ion exchange. The high demand for heavy metals and the recurrent development of alternative sources such as clays have stimulated studies into several metal recovery techniques from such sources (Shemi et al., 2015). Some fundamental needs of man, ranging from agricultural, medical, energy, metallurgy, transportation, fashion, etc., have their solution anchored on the availability of some heavy metals, including aluminum.

The low density of aluminum and its capacity to withstand corrosion through the process of passivation make it notable. The primary raw material for aluminum production is bauxite, and the Bayer process is usually employed. However, several

countries with severely depleted bauxite deposits have turned to alternative local methods of alumina mining (Siddique and Kurny, 2010). Clay is one of the many different raw materials for alumina production that is widely available in Nigeria.

Many of the clay have up to 25-40% alumina content that can be recovered for use in industry (Ohale et al., 2017). In order to improve the local production of alumina, multiple studies have been conducted on the leaching of alumina from clay using different acids, but none have used hydrochloric acid (HCl) on Ogbunike clay. Therefore, this research aims to cover this gap. The literature lists several benefits of using HCl over other acids for leaching alumina, including how easy it is to filter slurries, how insoluble titanium dioxide is, and how simple it is to remove iron, which may be found in many forms of clay (Stein et al., 2022).

The problem most frequently connected with the use of HCl is severe corrosion. But the creation of corrosion-resistant polymers and rubbers has partially solved this issue, making corrosion less of a barrier (Peters, 1962; Schoenborn and Hofmann, 1979). In their investigation of the kinetics of alumina leaching from calcined clay using hydrochloric, nitric, and sulphuric acids, Hulbert and Huff (1970) came to the conclusion that the removal of alumina using the acids could

Table 1. Composition of ogbunike clay

Al ₂ O ₃	MgO	Na ₂ O	SiO ₂	Cl	SO ₃	P ₂ O ₅	K ₂ O	TiO ₂	Mn ₂ O	Fe ₂ O ₃	ZnO	SrO	ZnO	CaO	Cr ₂ O ₃
24.035	0.472	0.000	67.84	0.014	1.095	1.096	0.662	2.075	0.016	2.194	0.005	0.021	0.005	0.460	0.019

be described by a “nucleation rate equation”, and that under the conditions used, the rate of alumina leaching was sped up by hydrochloric acid, slowed down by sulphuric acid, then followed by nitric acid.

This study aims to see how HCl solution affects the kinetics of alumina leaching from Ogbunike clay. For the leaching of alumina, process variables including calcination temperature, acid concentration, leaching temperature, particle size, and solid-to-liquid ratio were considered. The experimental results were analyzed using the shrinking core model (SCM). This investigation will also demonstrate the presence of leachable alumina in this clay.

MATERIALS AND METHODS

Materials Collection and Preparation

The clay sample was collected in Ogbunike (N: 6° 11' 5"; E: 6° 50' 39"; A: 113 m) in Anambra State, Southeast region of Nigeria. To eliminate light floating particles accumulated with the clay, it was rinsed with water and sundried for 72 hours. After being crushed using a pestle and mortar and sieved with a 75-micron sieve, the sample was then heated in a furnace to 350-1,000 °C for calcination. The calcination period was also extended from 30 to 180 minutes. The ideal calcination temperature and duration were then determined by dissolving the calcined samples in a solution of hydrochloric acid. All of the next dissolution processes used the ideal calcination temperature and duration that had been established.

Leaching Experiments

Leaching experiment was carried out in a bottom flask using a magnetic stirrer and a reflux condenser. The flask was put on a heating mantle with a thermostat. A thermometer was used to keep track of the temperature of the solution, which was kept at a specific level. A calculated volume of the acid, a 2 g sample of calcinated clay was added and heated constantly while stirring. The slurry was filtered once the reaction was completed, and the filtrate was collected for aluminum analysis using atomic absorption spectroscopy (AAS) “agilent technologies 200 series AA 240FSAA”. The proportion of alumina removal (x) was computed by Eq. 1, as follows (Arslan, 2022):

$$x = \frac{\text{Amount of Al}^{3+}}{\text{Total theoretical Al}^{3+} \text{ content of the calcined clay sample at time, } t} \quad (1)$$

To further examine the effects of the process parameters, the experiment was repeated with the following changes: leaching temperature (30-90°C), acid concentration (1-3 M), particle size (75-800 µm), and solid-to-liquid ratio (0.2-0.6 g/ml).

Dissolution Kinetics Using Shrinking Core Model

The experimental results were analyzed using the SCM to construct the kinetic model and rate-controlling step for the dissolving of the alumina in HCl solution. The SCM proposes that one of the following steps may be used to regulate a

heterogeneous process’ response rate (Eq. 2, Eq. 3, and Eq. 4) (Ismael et al., 2022; Levenspiel, 1999):

Film diffusion control:

$$X = \frac{3bdC_A}{\rho_B R_0} = K_1 t. \quad (2)$$

Surface chemical reactions control:

$$1 - (1 - X)^{1/3} = \left(\frac{bdC_A}{\rho_B R_0} \right) t = K_2 t. \quad (3)$$

Product layer diffusion control

$$1 + 2(1 - X) - 3(1 - X)^{2/3} = \left(\frac{6bdC_A}{\rho_B R_0^2} \right) t = K_3 t, \quad (4)$$

where X is the fraction reacted, ρ_B is the molar density of solid reactant (mol/m³), C_A is the concentration of A in the bulk solution (mol/m³), b is the stoichiometric coefficient of the solid, t is time in seconds, d is the effective diffusion coefficient (m²/s), and K_1 , K_2 , and K_3 are the rate constants.

RESULTS AND DISCUSSIONS

Analysis of the Ogbunike Clay Composition

Table 1 shows the clay’s composition as measured by x -ray fluorescence spectroscopy (XRF). **Table 1** demonstrates that ogbunike clay has 24.03% extractable alumina, indicating that the alumina leaching procedure is feasible. Most of the clay mineral compositions can be carried out using either the XRF or XRD analytical method of testing. In this case, in order to ascertain the degree or compositions of these compounds, present in our clay sample, XRF was used to investigate and give inference on the compositions.

Leaching Studies

Effect of calcination temperature

The significance of clays being heated to a certain temperature has been emphasized by the diverse results that were produced while using these distinct compositions. In fact, without heat treatment, the material is consolidated by coating it with various types of clay, which yields a material with a poor mechanical strength. The mechanical characteristics improve, and the pores are broken during calcination, which increases the clay ore’s susceptibility to leaching.

Figure 1 illustrates how the calcination temperature affects the percentage yield of alumina at 3 M of HCl, 0.2 g/ml solid-to-liquid ratio, and 75 µm particle size. The graph shows that the percentage of alumina dissolution in HCl solution is low below 500 °C and over 700 °C temperature. Researchers from earlier studies (Ajemba and Onukwuli, 2012b; Al-Zahrani, 2009; Ozdemir and Cetisli, 2005), that suggested 600 °C and 650 °C for the calcination process of clays noted similar behavior. After more than two hours of heating, the maximum dissolving percentage yield by the acids was obtained at 700 °C. Additionally, it shows that when calcinations progress over time, the rate of dissolution also accelerates (Ajemba and

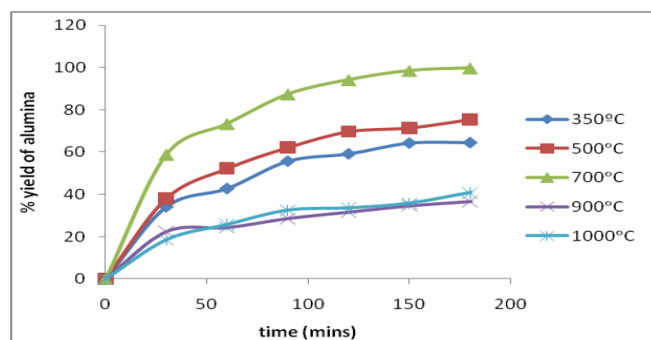


Figure 1. Effect of calcination temperature on %yield of alumina (3 M of HCl, 90 °C leaching temperature, 0.2 g/ml solid-to-liquid ratio and 75 μ m particle size)

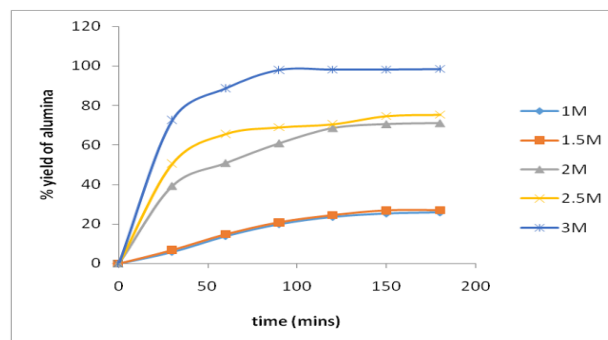


Figure 3. Effect of acid concentration on the leaching of alumina using HCl (90 °C leaching temperature, 0.2 g/ml solid-to-liquid ratio and 75 μ m particle size)

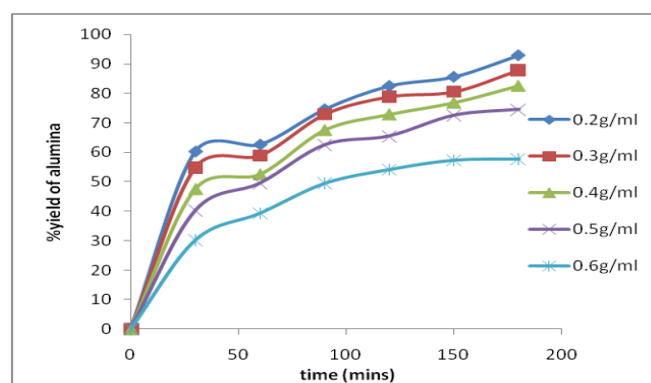


Figure 2. Effect of solid-to-liquid ratio on the leaching of alumina using HCl acid (3 M of HCl, 90 °C leaching temperature and 75 μ m particle size)

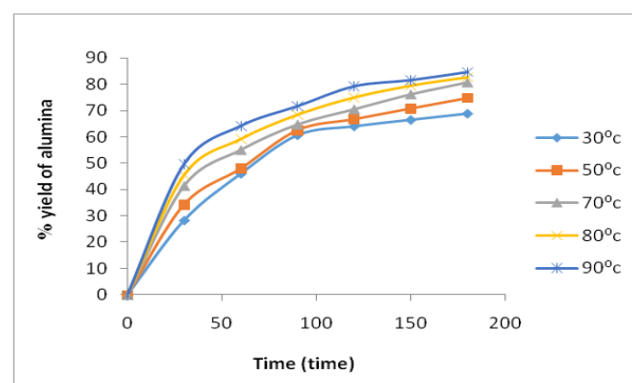


Figure 4. Effect of leaching temperature on the leaching of alumina using HCl (3 M of HCl, 0.2 g/ml solid-to-liquid ratio and 75 μ m particle size)

Onukwuli, 2012a; Nnanwube and Onukwuli, 2020a, 2020b). Thus, it can be deduced that there exists a direct variation between time and dissolution rate of the ore sample when dissolved into the residual acid.

Effect of solid-to-liquid ratio

The effect of the solid-to-liquid ratio on alumina dissolution was studied across a range of 0.2-0.6 g/ml dosage ratio. From **Figure 2**, it could be seen that the percentage dissolution of alumina increases with decreasing solid-to-liquid ratio. It has been found that reducing the size of the particles that make up a certain weight would result in an increase in the number of particles that the weight represents.

Because the surface area of the reactant has increased as a result of the smaller particles, the rate of reaction is increased. This is due to the larger effective surface area that the smaller particles allow for contact with the acid molecules, and this agrees with the result of (Ajemba and Onukwuli, 2012a; Nnanwube et al., 2022). In lieu of this, the optimal ratio of 0.2 g/ml of solute to solvent was chosen as best fit for this study, it gives an inverse relationship between the acid-clay ratio and percentage yield. The figure below illustrates how the percentage yield increases as the ratio decreases.

Effect of acid concentration

The effect of aid concentration on the dissolution of alumina was studied at five interval concentration points between 1-3 M of HCl at a temperature of 90 °C, the particle

size of 75 μ m, and the ratio of 0.2 g/ml which has been chosen as optimum conditions for this study.

From the graphical results presented in **Figure 3**, it could be seen that concentration has a significant effect on the leaching of alumina because the percentage removal increased as the concentration of the leachant increased. Thus, it is observed that concentration and percentage yield have a direct relationship, in that a high concentration increases the strength of the reacting molecules, which in turns increases dissolution rate and yielding high products respectively (Al-Zahrani, 2009; Arslan, 2022; Ohale et al., 2017).

Effect of leaching temperature

The effect of leaching temperature on alumina leaching from Ogbunike clay was studied at varying temperatures (30 °C, 50 °C, 70 °C, 80 °C, and 90 °C). Temperature is crucial for leaching to occur efficiently, as the temperature of the reacting molecules rises, they tend to collide with one another and the walls of the vessels in which they are contained, creating a perfect elastic collision.

From **Figure 4**, it was observed that as temperature increased, the dissolution rate of alumina increased; higher temperatures increase the kinetic energy of the reacting particles for more collisions to take place directly, resulting in increased alumina yield. This is in line with (Udeigwe et al., 2015) who found that the reason for the significant improvement in the leaching rate is likely due to the acceleration of the molecule's thermal motion caused by a rise

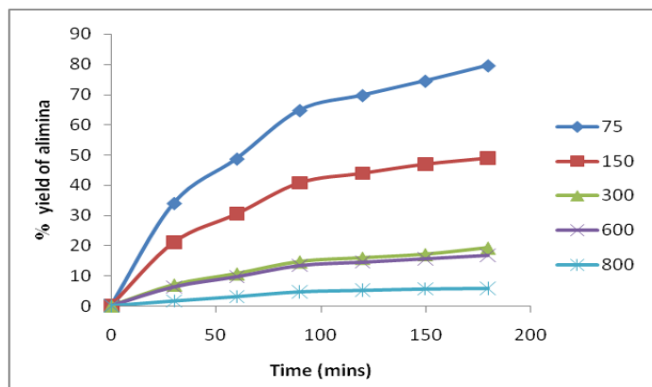


Figure 5. Effect of particle size on the leaching of alumina using HCl (3 M of HCl, 90 °C leaching temperature and 0.2 g/ml solid-to-liquid ratio)

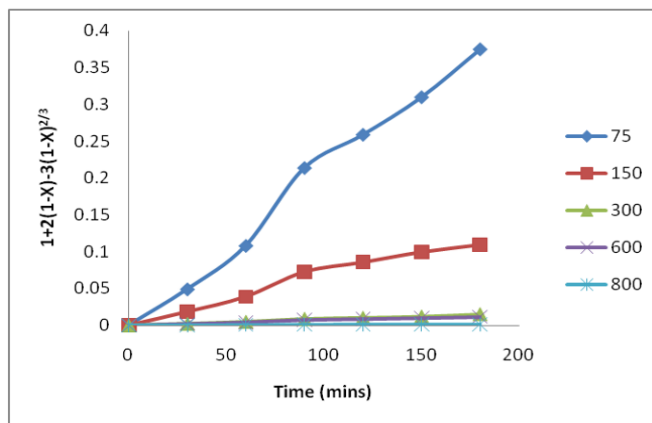


Figure 8. Plot of $1 + 2(1 - x) - 3(1 - x)^{2/3}$ against time at different particle sizes for alumina leaching using HCl

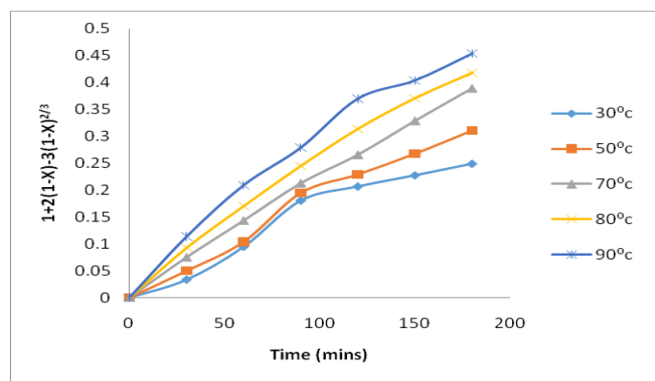


Figure 6. Plot of $1 + 2(1 - x) - 3(1 - x)^{2/3}$ against time at different temperatures for alumina leaching using HCl

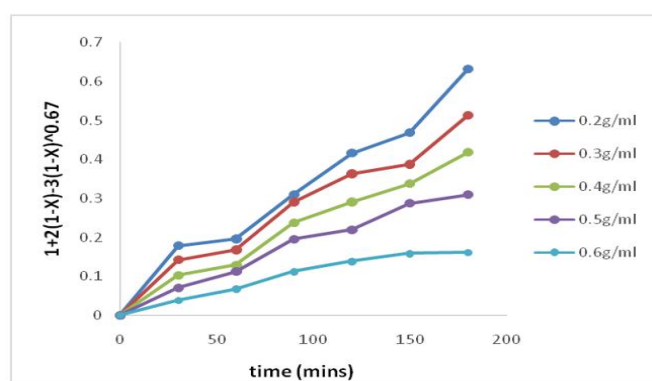


Figure 9. Plot of $1 + 2(1 - x) - 3(1 - x)^{2/3}$ against time at different solid-to-liquid ratio for alumina leaching using HCl

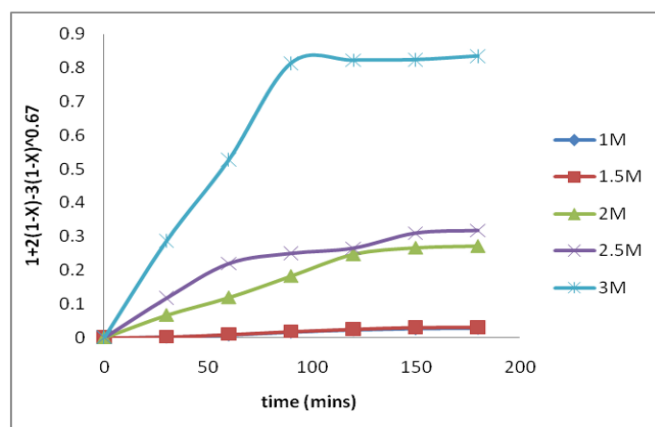


Figure 7. Plot of $1 + 2(1 - x) - 3(1 - x)^{2/3}$ against time at different concentrations for alumina leaching using HCl

in temperature and an increase in the contact surface between a single particle and the leaching agent, as well as the thinner barrier of products layer and an increase in diffusion flux with a rise in temperature. Optimum yield of alumina using HCl is 84.72% at a temperature of 90 °C as can be seen from **Figure 4**.

Effect of particle size

Clay particles with diameters ranging from 75 μm to 800 μm were dissolved in an HCl solution (**Figure 5**). Because the smaller particles had a greater specific surface area for

interaction with the acid molecules, it was found that the dissolving rate increased with decreasing particle size. Thus, particle size has a negative effect on the dissolution rate of a leaching process. The smaller particle screen size helps to improve the surface area and enhances proper interaction between the solutes and the solvent for proper dissolution (Ajemba and Onukwuli, 2012a).

Dissolution of Alumina in HCl

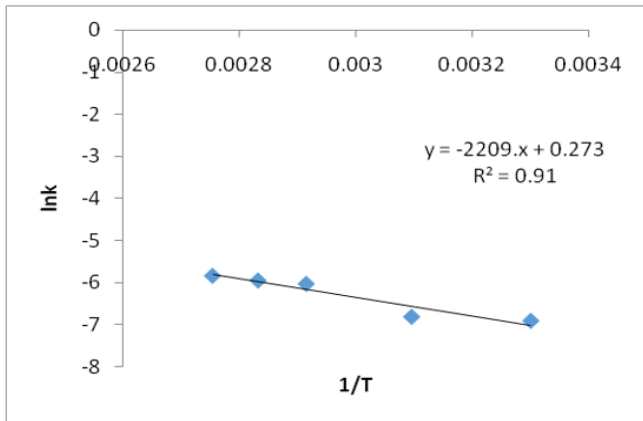
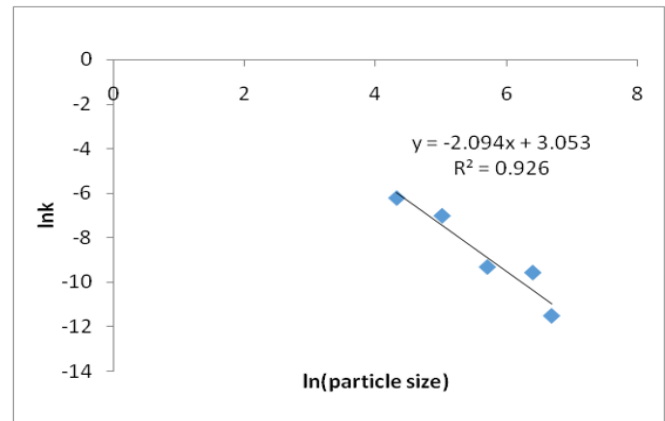
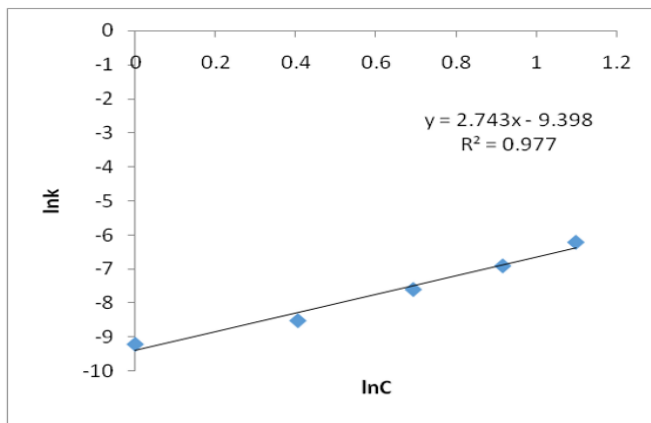
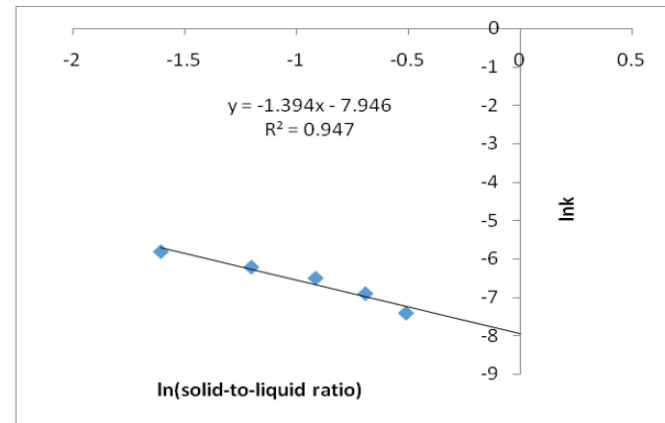
There exist different postulations by different authors for comparative analysis. For the effective dissolution of the calcined clay particle, an assumption was made that related the clay particle to be spherical in nature, and as such shrinks when in contact with a suitable acid. In order to effectively study the mechanism of dissolution of this spherical material (clay), SCM as postulated by different authors was invoked.

In contrast to earlier postulations, the investigation of the experimental data using the SCM revealed that the product layer diffusion step governs the dissolution of alumina in HCl solution. **Figure 6**, **Figure 7**, **Figure 8**, and **Figure 9** depict the graphs that illustrate the mechanism of alumina leaching in hydrochloric acid, and they reveal a very strong link between temperature, acid concentration, particle size, and solid-to-liquid ratio.

As a result, **Table 2** displays the values of the rate constants and regression correlation coefficients.

Table 2. Kinetic energy parameters

E_a/R	$\ln k_0$	R^2	R	E_a	k_0
2,209.2	0.2738	0.9100	8.3145	18.368	1.3139

**Figure 10.** A plot of $\ln k$ versus $1/T$ **Figure 12.** A plot of $\ln k$ versus \ln (particle size)**Figure 11.** A plot of $\ln k$ versus $\ln C_{HCL}$ **Figure 13.** A plot of $\ln k$ versus \ln (solid-to-liquid ratio)

The activation energy was calculated from Arrhenius equation or Eq. 5, as follows (Gooch, 2011; Onyechi and Igwegbe, 2018, 2019):

$$k = k_0 e^{(-E_a/RT)} \quad (5a)$$

$$\ln(k/k_0) = -E_a/RT \quad (5b)$$

$$\ln k - \ln k_0 = -E_a/R \times T^{-1} \quad (5c)$$

$$\ln k = \ln k_0 - E_a/R \times T^{-1} \quad (5d)$$

It can be seen from Eq. 5d, that plotting a graph of $\ln k$ against $1/T$, E_a/R could be estimated as slope and $\ln k_0$ as the intercept as shown in **Figure 10**, where E_a is the activation energy in kJ/mol, k is the rate constant, k_0 is the exponential factor in s^{-1} , R is the universal gas constant in J/molK, and T is the temperature in degree Kelvin, are shown in **Table 2**. Since, the value of E_a is <20 kJ/mol (that is, 18.368 kJ/mol), the anticipated mechanism is product diffusion (Ismael et al., 2022).

A semi-empirical kinetic model is provided below to explain the combined impact of the process factors on the Ogbunike clay's dissolving kinetics in HCl (Levenspiel, 1999):

$$1 + 2(1 - X) - 3(1 - X)^{2/3} = k_0 C_{HCl}^a dp^b (S/L)^c e^{(-E_a/RT)}, \quad (6)$$

where C_{HCl} represents the concentration of HCl (mol/m^3), X represents the fraction reacted, k_0 represents the exponential factors (s^{-1}), dp represents the particle size (μm), S/L represents the solid-to-liquid ratio (g/ml). The slope of the plots showing the correlation between the natural logarithm of the observed rate constant computed from **Figure 7**, **Figure 8**, and **Figure 9**, and the natural logarithm of the process variables a , b , and c , are presented in **Figure 11**, **Figure 12**, and **Figure 13**, for acid concentration, particle size and solid-to-liquid ratio, respectively.

The value for a , b , c , k_0 and E_a are $a = 2.7432$; $b = -2.0947$; $c = -1.3941$; $k_0 = 1.3139$; and $E_a = -18.368$. Substituting these values into Eq. 6 gives the kinetic model for the dissolution of alumina in ogbunike clay using HCl, as follows:

$$1 + 2(1 - X) - 3(1 - X)^{2/3} = 1.3139 C_{HCl}^{2.743} dp^{-2.094} (S/L)^{-1.394} e^{(-18.36/RT)} \quad (7)$$

The graphical and statistical analyses showed that the dissolution followed the "product layer diffusion-controlled equation" of the model:

$$1 + 2(1 - X) - 3(1 - X)^{2/3} = 1.3139 C_{HCl}^{2.743} dp^{-2.094} (S/L)^{-1.394} e^{(-18.36/RT)} \quad (8)$$

Table 3. Values of the rate constants and correlation coefficient for the hydrochloric acid leaching of alumina

Process parameter	Kinetic equation					
	$X = k_1 t$ (Film diffusion control)		$1 - (1 - X)^{1/3} = k_2 t$ (Chemical reaction control)		$1 + 2(1 - X) - 3(1 - X)^{2/3} = k_3 t$ (Product layer diffusion control)	
	K_1	R^2	K_2	R^2	K_3	R^2
Temperature (°C)						
30	0.0036	0.8315	0.0017	0.8885	0.0015	0.9475
50	0.0038	0.8437	0.0019	0.9204	0.0018	0.9830
70	0.0039	0.8210	0.0021	0.9261	0.0021	0.9977
80	0.0039	0.7881	0.0022	0.9064	0.0023	0.9898
90	0.0040	0.7479	0.0023	0.8796	0.0025	0.9729
Concentration (M)						
1.0	0.0015	0.9181	0.0005	0.9278	0.0002	0.9626
1.5	0.0016	0.9176	0.0006	0.9284	0.0002	0.9640
2.0	0.0035	0.7831	0.0018	0.8646	0.0016	0.9502
2.5	0.0033	0.6419	0.0017	0.7386	0.0016	0.8656
3.0	0.0042	0.5760	0.0038	0.7714	0.0046	0.8092
Particle size (µm)						
75.0	0.0041	0.8687	0.0022	0.9477	0.00210	0.9900
150	0.0025	0.8613	0.0011	0.8984	0.00060	0.9732
300	0.0010	0.9103	0.0004	0.9209	0.00009	0.9869
600	0.0009	0.8955	0.0003	0.9048	0.00007	0.9781
800	0.0003	0.9150	0.0001	0.9172	0.00001	0.9605
Solid-to-liquid ratio (g/ml)						
0.2	0.0042	0.7442	0.0028	0.9126	0.0022	0.9866
0.3	0.0040	0.7535	0.0024	0.8954	0.0032	0.9750
0.4	0.0039	0.7991	0.0022	0.9135	0.0017	0.9830
0.5	0.0036	0.8116	0.0019	0.9023	0.0026	0.9738
0.6	0.0029	0.8091	0.0013	0.8653	0.0009	0.9577

CONCLUSION

The optimal conditions for the alumina to dissolve in HCl include activation temperature of 70 °C for 60 minutes, leaching temperature of 90 °C, HCl concentration of 3 M, particle size of 75 µm, and solid-to-liquid ratio of 0.2 g/ml. The dissolution rate for alumina under these circumstances is 84.75%. **Table 3** shows the values of the rate constants and correlation coefficient for the hydrochloric acid leaching of alumina. The shrinking core kinetic model can describe the dissolution process with the product layer diffusion as the limiting step. Additionally, this research establishes that Ogbunike clay may be used as the main raw material in the industrial production of alumina. The apparent activation energy evaluated from the experimental data is 18.36 kJ/mol for leaching in hydrochloric acid.

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Declaration of interest: The authors declare that they have no competing interests.

Ethics approval and consent to participate: Not applicable.

Availability of data and materials: All data generated or analyzed during this study are available for sharing when appropriate request is directed to corresponding author.

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