ORIGINAL RESEARCH ARTICLE

FIVE-FACTOR RESPONSE SURFACE OPTIMISATION OF HYDROCHLORIC ACID DISSOLUTION OF ALUMINA FROM A NIGERIAN CLAY

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ARTICLE INFORMATION

ABSTRACT

The major challenge encountered in the process of making the acid dissolution of alumina from clays economically viable is the determination of the optimum conditions of the key process variables in order to enhance efficient recovery. High alumina recovery from clays can be achieved by determining the optimum conditions of the process variables during optimization. In this research, using the experimental design, the combined effects of five independent variables (calcination temperature, leaching temperature, acid concentration, stirring speed and liquid/solid ratio) on the yield of alumina from the local clay was studied and the second order polynomial regression equation was developed to evaluate the influence of the five independent variables on alumina yield. Model adequacy test was performed using the analysis of variance (ANOVA) and 0.9209 was obtained as the correlation value between the experimental responses and the predicted responses. The optimum yield of alumina was obtained as 80.07% at 677.27°C heat of activation; 65.18°C leaching temperature; 1.9mol/cm³HCl concentration; 10.36 liquid-solid weight ratio and 442.92rpm stirring speed.

1.0   Introduction

Aluminum is in high demand all over the world today due to some of its unique properties like hardness, strength and electrical conductivity making it extremely useful in so many engineering applications like electric power transmission (Olaremu, 2005). The expanded usage of the metal coupled with its ore depletion resulting from the increased mining of bauxite which is the most important ore from which aluminum is produced (Hosseini et al., 2011) is responsible for the high cost of the metal. For a continuous supply of the metal, there is need to explore alternative materials from which it can be produced (Al-Zahrani and Abdel-Majid, 2009; Orugba et al., 2020). Many Nigerian clays have been identified to be very rich in alumina content (Ogbuagu, et al., 2010; Ajemba and Onukwuli, 2012; Ajemba et al., 2012; Orugba et al., 2014; Udochukwu et al., 2020). Different methods have been adopted by researchers over the years to recover alumina from clays of which acid leaching has been an outstanding method (Al-Zahrani and Abdel-Majid, 2009). In the dissolution of alumina from the clays using acids or alkalis, it is important to investigate the process parameters in order to obtain their optimum conditions to enhance efficient recovery, in which case, the optimization of the dissolution process becomes very important. In order to determine the most appropriate values of process variables, optimization is very important. In a leaching process, the calcination temperature, leaching temperature, stirring speed, acid concentration and liquid-solid ratio have been identified as important parameters that control the yield of alumina from clays (Orugba et al., 2014, 2020). The chief aim in any optimization process is to obtain the minimum value or maximum value of a function depending on the circumstances. The need to search for the economic conditions of process variables is paramount because apart from cost reduction, certain process variables if not properly controlled can increase waste generation into the ecosystem. For example, the energy needed in the heat activation step is obtained from burning

of petroleum-based fuels which increases green house gases generation into the atmosphere posing risks to the environment (Orugba et al., 2019a, 2019b). In the chemical and process industry, the response surface methodology (RSM) has been employed for the purpose of either obtaining higher-value products or operating the process in a cost effective way and ensuring the process operates in a more reliable and stable way (Alam et al., 2007; Gunawan and Suhendra, 2008; Narayana et al., 2011; Sudamalla et al., 2012). Ajemba et al., (2012) performed the optimization of alumina dissolution from Ukpor clay in tetra-oxosulphate (vi) acid using the response surface methodology and obtained an optimum yield of 97.23% at the leaching conditions of calcination temperature of 729.54°C; leaching temperature of 103.25°C; acid concentration of 2.93mol/l; solid/liquid ratio of 0.027g/ml and stirring speed of 436.34 rpm. Orugba et al., (2014) studied the process modeling of sulphuric acid leaching of iron from a local Nigerian clay using the response surface methodology and obtained the iron yield of 84.7% at calcinations temperature of 650°C; leaching temperature of 70.02°C; acid concentration of 1.89mol/cm^3; liquid-solid ratio of 10.67 and stirring speed of 379.80rpm. Ohale et al., (2017) used Artificial Neural Network (ANN) and Response Surface Methodology based on a 25^{-1} fractional factorial design as tools for simulation and optimization of the dissolution process for a Nigerian local clay and obtained an optimal response of 81.45% yield of alumina at 4.6 M sulphuric acid concentration, 214 min leaching time, 0.085 g/ml dosage and 214 rpm stirring speed. Onukwuli et al., (2018) performed the process optimization of hydrochloric acid leaching of iron from Agbaja clay using the response surface methodology and obtained iron yield of 85.13 % at calcinations temperature of 800°C, leaching temperature of 53.7°C, HCl concentration of 2.34 mol/cm^3, liquid to solid ratio of 9.90cm^3/g and stirring speed of 250 rpm.

Every clay has its unique properties and responds to different leaching conditions in different ways hence the conditions that produce optimum ore yield for a particular clay may not produce same result for another clay sample. Therefore, generalizing leaching conditions for all clays is highly discouraged. The local clay studied in this work has not been studied extensively hence performing the optimization of its leaching process is important so as to obtain the values of process variables that guarantee optimum alumina yield. The response surface methodology (RSM) was used in the optimization study due to its robustness (Raissi and Farsani, 2009) while HCl was considered as the solvent because it is cheap and previous researches have recorded significant alumina yield with HCl (Al-Zahrani and Abdel-Majid, 2009). The aim of this research work is to investigate the influence of five process variables as well as their possible interactions on alumina yield from the local clay using hydrochloric acid so as to obtain their optimum conditions that guarantee maximum alumina yield. Using the response surface methodology, a suitable predictive model will be obtained for the dissolution process from the data obtained from few experiments through regression analysis.

2.0   Materials and Methods
2.1   Material preparation and leaching experiments
The local clay was obtained from Ozoro (6.24°N, 5.55°E) in Delta State Nigeria. The characterization of this clay by Orugba et al., (2014) and Orugba et al., (2020) revealed that the local clay has 33.90% of alumina making it a very viable source of the ore. The clay was soaked in water for two days in order to ease the removal of debris and stony materials. The dissolved clay was properly sieved and sun-dried for 24 hours then oven-dried at 60°C for 18 hours to aggregate the particles. The clay samples were subjected to heat activation in a muffle furnace at different temperatures ranging from 400°C to 900°C for a period of 1hr in order to study the influence of temperature of activation on alumina yield (Al-Zahrani and Abdel-Majid, 2003). The activated clay samples were ground to the same particle size of 0.045mm to increase the surface areas of the particles (Ozdemir and Cetisli, 2005). The prepared sample clay were properly labeled and subjected to leaching experiments. For each leaching experiment, 20g of

the activated clay was weighed into an already determined volume of the acid and heated in a round bottom flask for a period of 30minutes based on the conditions in the experimental design matrix shown in Table 1. At the end of the period, 2ml of each sample was collected and analyzed for alumina ion using the Atomic Adsorption Spectra (AAS) (Al-Zahrani and Abdel-Majid, 2003; Orugba et al., 2014).

2.2   Design of Experiment
The experimental design matrix was carried out using the central composite rotatable design of the Design Expert. Five independent variables (calcinations temperature, leaching temperature, acid concentration, liquid-solid ratio and stirring speed) were investigated at five levels and a total of 50 experiments were obtained from the design using the central composite rotatable design of 25 = 32 plus six centre points and (2 x 6 = 12) star points The yield of alumina from the ore was optimized using the Response Surface Methodology. Calcination temperature was varied from 400°C - 900°C, leaching temperature from 45°C - 85°C, acid concentration from 0.5mol/cm^3 - 3mol/cm^3, liquid-solid ratio from 4cm^3/g - 16cm^3/g and stirring speed from 90rpm-720rpm. The experimental matrix as well as alumina yield is presented in Table 1.

2.3   Statistical Analysis
The statistical analysis of the result was performed using Design expert software (Version V10) and a second order polynomial equation that represents the response (alumina yield) as a function of the five independent variables was developed based on Equation (1).

$$y = b_0 + \sum_{i=1}^{k} b_i x_i + \sum_{i=1}^{k} b_{ii} x_i^2 + \sum_{i=1}^{k} b_{ij} x_i x_j \qquad (1)$$

The alumina yield being the response is represented by y, b_0 is the response of the central point, while the main effects and the interactions of the variables x_i on the response y are measured by the other terms. The number of factors is represented by k while the independent variables under study are represented by x_i and x_j. The model adequacy was tested using the Analysis of variance (ANOVA) based on F-test.

3.0   Results and Discussion
3.1   Generation of the regression model equation
The influence of the interactions of the five independent variables on the yield of alumina from the local clay was investigated from the design matrix presented in Table 1.

Table 1: Experimental matrix and alumina yield

Std. order	Calcinations temp (°C) X_1	Leaching temp (°C) X_2	Acid conc. (mol/cm^3) X_3	Liquid/solid ratio (cm^3/g) X_4	Stirring speed (rpm) X_5	Yield (%) Exp. Value	Pred. value
1	400	45	0.5	4	90	39.8	46.0
2	850	45	0.5	4	90	59.5	56.6
3	400	90	0.5	4	90	57.6	56.5
4	850	90	0.5	4	90	66.1	64.9
5	400	45	3	4	90	54.9	56.9
6	850	45	3	4	90	67.3	64.4
7	400	90	3	4	90	66.5	63.8
8	850	90	3	4	90	70.1	69.1
9	400	45	0.5	16	90	58.2	55.3
10	850	45	0.5	16	90	64.9	64.8
11	400	90	0.5	16	90	57.8	62.3
12	850	90	0.5	16	90	72.4	69.7
13	400	45	3	16	90	67.9	66.9
14	850	45	3	16	90	71.2	73.4
15	400	90	3	16	90	73.2	70.3

16	850	90	3	16	90	73.5	74.6
17	400	45	0.5	4	720	47.3	49.1
18	850	45	0.5	4	720	58.6	58.4
19	400	90	0.5	4	720	61.2	56.9
20	850	90	0.5	4	720	63.9	63.9
21	400	45	3	4	720	62.9	58.3
22	850	45	3	4	720	64.9	64.6
23	400	90	3	4	720	59.2	62.4
24	850	90	3	4	720	66.9	66.4
25	400	45	0.5	16	720	52.8	50.1
26	850	45	0.5	16	720	55.2	58.4
27	400	90	0.5	16	720	51.9	54.4
28	850	90	0.5	16	720	62.3	60.5
29	400	45	3	16	720	54.2	60.1
30	850	45	3	16	720	69.3	65.4
31	400	90	3	16	720	61.7	60.7
32	850	90	3	16	720	69.2	63.8
33	89.9	67.5	1.75	10	405	57.4	54.5
34	1160	67.5	1.75	10	405	65.3	70.6
35	625	13.9	1.75	10	405	54.8	53.2
36	625	121	1.75	10	405	59.9	63.8
37	625	67.5	-1.22	10	405	51.3	50.4
38	625	67.5	4.72	10	405	63.9	67.2
39	625	67.5	1.75	-4.27	405	49.9	51.8
40	625	67.5	1.75	24.27	405	59.1	59.6
41	625	67.5	1.75	10	-344	64.3	64.9
42	625	67.5	1.75	10	1154	53.9	55.7
43	625	67.5	1.75	10	405	78.8	79.6
44	625	67.5	1.75	10	405	79.3	79.6
45	625	67.5	1.75	10	405	79.4	79.6
46	625	67.5	1.75	10	405	79	79.6
47	625	67.5	1.75	10	405	79	79.6
48	625	67.5	1.75	10	405	79.4	79.6
49	625	67.5	1.75	10	405	80	79.6
50	625	67.5	1.75	10	405	80	79.6

The technique of the central composite design was employed to generate a polynomial regression equation that shows the relationship that exists between the dependent variable (alumina yield) and the five independent variables (calcination temperature, leaching temperature, stirring speed, acid concentration and liquid-solid ratio) as shown in Equation (2).

$$Y_{Al2O3} = 79.56 + 3.39X_1 + 2.23X_2 + 3.54X_3 + 1.64X_4 - 1.94X_5 - 0.55X_1X_2$$
$$- 0.76X_1X_3 - 0.24X_1X_4 - 0.31X_1X_5 - 0.91X_2X_3 - 0.87X_2 - 0.70X_2X_5$$
$$+ 0.91X_3X_4 - 0.41X_3X_5 - 2.05X_4X_5 - 3.01X_1^2 - 3.72X_2^2 - 3.67X_3^2 - 4.22X_4^2$$
$$- 3.41X_5^2 \qquad (2)$$

3.2   Model adequacy test

In order to account for the adequacy of the model equation obtained, a model adequacy test using the Analysis of Variance (ANOVA) was performed as summarized in Table 2. The F-value and the p-value were used to evaluate the adequacy of the model because from statistical tests, the smaller the p-value and the larger the F-value, the higher the accuracy of the model (Rashid et al., 2011).

Table 2: The summary of the model adequacy test

Source	Sum of squares	Degree of freedom	Mean squares	F-value	P-value	Remarks
Sequential sum of squares						
Linear	1537.326	5	307.4652	4.484532	0.0022	Significant
2FI	241.09	10	24.109	0.295325	0.9776	Not significant
Quadratic	2415.38	5	483.0759	38.89003	< 0.0001	Significant
Cubic	239.5728	15	15.97152	1.853254	0.1282	Not significant
Source	Std Dev.	R^2	Adjusted R^2	Predicted R^2	PRESS	Remarks
Model summary statistics						
Linear	8.280173	0.337575	0.2623	0.232841	3493.658	Inadequate
2FI	9.035235	0.390515	0.121625	0.130424	3960.068	Inadequate
Quadratic	3.524428	0.920899	0.866347	0.708686	1326.65	Adequate
Cubic	2.935659	0.973506	0.907272	-0.22017	5556.674	Inadequate

When the p-value is less than 0.01, the model is highly significant, when the p-value lies between 0.01 and 0.05, it is significant but if it is greater than 0.05, it is not significant (Rashid et al., 2011). From the F-values of the different models presented in Table 3, it could be seen that the quadratic model with the highest F-value of 38.89 and with a p-value less than 0.0001adequately fits the experimental data.

The significant terms of the model equation generated in Equation (2) were selected based on their F and p-values as shown in Table 3.

Table 3: ANOVA for Response Surface Quadratic Model

Source	Sum of squares	Degree of freedom	F-value	P-value (prod.>F)
Model	4193.796	20	16.88107	< 0.0001
X_1	498.8237	1	40.1578	< 0.0001
X_2	216.0211	1	17.39078	0.0003
X_3	543.0555	1	43.71868	< 0.0001
X_4	115.995	1	9.338178	0.0048
X_5	163.4306	1	13.15698	0.0011
X_1X_2	9.68	1	0.779288	0.3846
X_1X_3	18.605	1	1.497795	0.2309
X_1X_4	1.805	1	0.145312	0.7058
X_1X_5	3.125	1	0.251578	0.6198
X_2X_3	26.645	1	2.145056	0.1538
X_2X_4	24.5	1	1.972372	0.1708
X_2X_5	15.68	1	1.262318	0.2704
X_3X_4	1.125	1	0.090568	0.7656
X_3X_5	5.445	1	0.43835	0.5131
X_4X_5	134.48	1	10.82631	0.0026
X_{12}	503.2336	1	40.51281	< 0.0001
X_{22}	767.5096	1	61.78836	< 0.0001
X_{32}	749.3644	1	60.32757	< 0.0001
X_{42}	989.7077	1	79.67641	< 0.0001
X_{52}	645.0513	1	51.92985	< 0.0001
Residual	360.2261	29		
Lack of Fit	358.8274	22	81.62455	< 0.0001
Pure Error	1.39875	7		
Cor Total	4554.022	49		

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The significance of each term of the model equation is judged based on its p-value as shown in Table 3. It could be revealed that the linear effects of all the independent variables (X_1, X_2, X_3, X_4, and X_5) as well as their quadratic effects (X_{12}, X_{22}, X_{32}, X_{42}, and X_{52}) are highly significant on the yield of alumina while their interactions are not significant. When only the significant terms in Equation (2) are considered, a final model equation was obtained as presented in Equation (3).

$$Y_{Al2O3} = 79.56 + 3.39X_1 + 2.23X_2 + 3.54X_3 + 1.64X_4 - 1.94X_5 - 2.05X_4X_5 - 3.01X_1^2 - 3.72X_2^2 - 3.67X_3^2 - 4.22X_4^2 - 3.41X_5^2 \tag{3}$$

The summary of the regression values of the final model equation is presented in Table 4.

Table 4: Summary of regression values

Item	value
Std. dev.	3.52
Mean	63.94
C.V%	5.51
PRESS	1326.65
R-squared	0.9209
Adj. R-squared	0.8863
Pre-R-squared	0.7087
Adeq precision	14.672

From Table 4, with the coefficient of variation value of 5.51%, the model can be considered reasonably reproducible. The signal to noise ratio which is given as the value of the adequacy precision is 14.672 indicates that an adequate relationship of signal to noise ratio exists.
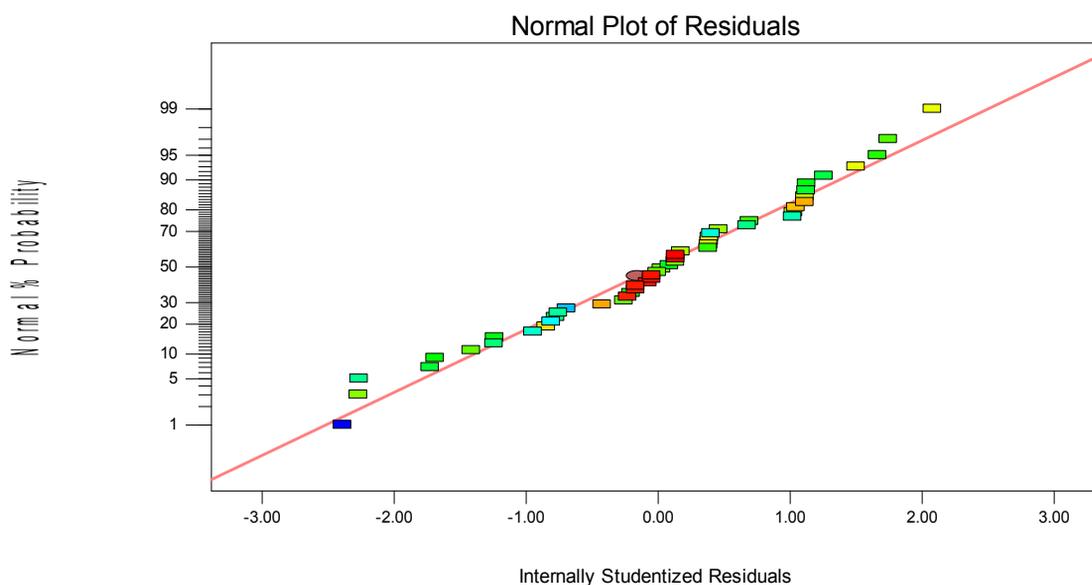
Figure 1: Plot of Normal probability against Studentized residual
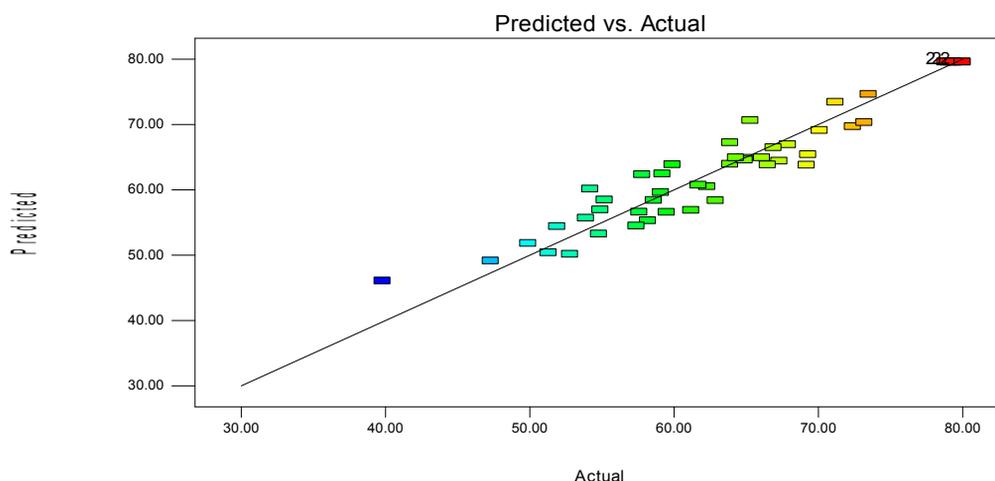
Figure 2: Plot of predicted yield values against actual yield values

The plot of Normal probability against Studentized residual given in Figure 1 and the plot of predicted yield values against actual yield values presented in Figure 2 revealed that a reasonable agreement exists between the actual and predicted alumina yield.

3.3   Response surface plots for the acid-dissolution of alumina

In order to study the combined effects of process variables on the yield of the ore, 3D response plot of any two independent variables is plotted while keeping another variable at its centre level as shown in Figures 3-12.

Figure 3 shows the variation of alumina yield with calcination temperature and leaching temperature. At low calcination temperatures and low leaching temperatures, very low alumina yield was recorded but as calcination temperature and leaching temperature increased, alumina yield increased. However, when calcination temperature was increased beyond 750°C, there was a decline in alumina yield. The decreased alumina yield at elevated calcination temperature could be due to the clay solid phase transformation and total dehydration (Al-Zahrani and Abdel-Majid, 2009)

The influence of acid concentration and calcination temperature on alumina yield is presented in Figure 4. From the figure, alumina yield increased with increase acid concentration and calcination temperature. Maximum alumina yield was obtained at the highest acid concentration of $3mol/cm^3$. The increased alumina yield with acid concentration could be due to the presence of more hydroxonium ions which increase leaching ability of the solvent (Poppleton and Sawyer, 1977)

The combined effect of liquid/solid ratio and calcination temperature on alumina yield is shown in Figure 5. As can be seen in the figure, high alumina yields were recorded with increased liquid/solid ratio and calcination temperature. Increased liquid/solid ratio makes more solvent available for proper dissolution of the ore and this enhances higher leaching rates (Ozdemir and Cetisli, 2005).

Figure 6 shows the influence of stirring speed and calcination temperature on alumina yield. From the figure, the yield of alumina increased as stirring speed and calcination temperature were increased. The lowest alumina yield was recorded at the lowest stirring speed of 90rpm and at the lowest calcination temperature of 400°C. As stirring speed increased, there is increased contact between the solid particles and the solvent which enhances ore dissolution (Orugba et al., 2014).

The combined influence of acid concentration and leaching temperature on alumina yield is presented in Figure 7. As shown in the figure, alumina yield increased as acid concentration and leaching temperature were increased. Higher acid concentrations means more solvent ions are made available for dissolution and this enhances dissolution rate. Also, high leaching temperatures increased the kinetic energy of the reacting species which also increase dissolution rate (Orugba et al., 2014).

Figure 8 shows the combined effect of liquid/solid ratio and leaching temperature on alumina yield. As can be seen from the figure, increasing liquid/solid ratio and leaching temperature increased alumina yield as the lowest alumina yield occurred at the lowest stirring speed and leaching temperature. At low leaching temperatures, the kinetic energies of the reacting species are low hence decreased reaction rate but this will increase as the leaching temperature increases. Also, low liquid/solid ratio implies less solvent to dissolve the solid particles hence reduced dissolution. Similar results were recorded by Al-Zahrani and Abdel-Majid, (2003).

The combined influence of liquid/solid ratio and acid concentration is shown in Figure 9. As shown in Figure 9, at low liquid/solid ratio and low acid concentration, the alumina yield is low but the yield was seen to increase at higher liquid/solid ratios and acid concentration.

Figure 10 shows the combined influence of stirring speed and acid concentration. The yield of alumina increased with increased stirring speed and acid concentration. The highest alumina yield occurred at the highest acid concentration of 3mol/cm^3.

The combined influence of stirring speed and liquid/solid ratio on alumina yield is presented in Figure 11. As shown in the figure, increasing both the stirring speed and liquid/solid ratio increased alumina yield as the highest alumina yield was obtained at the highest stirring speed and highest liquid/solid ratio.

Figure 12 shows the combined influence of stirring speed and leaching temperature on alumina yield. As both stirring speed and leaching temperature increased, alumina yield was also increased. The increased alumina yield with stirring speed is due to the increased contact between solid particles and solvent which enhances dissolution rate while increased leaching temperature increases the kinetic energy of the reacting species which also enhances dissolution rate.
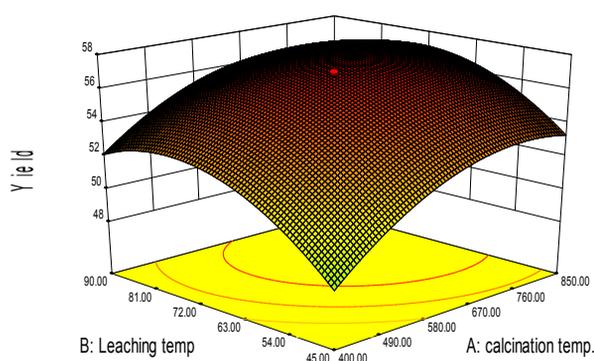

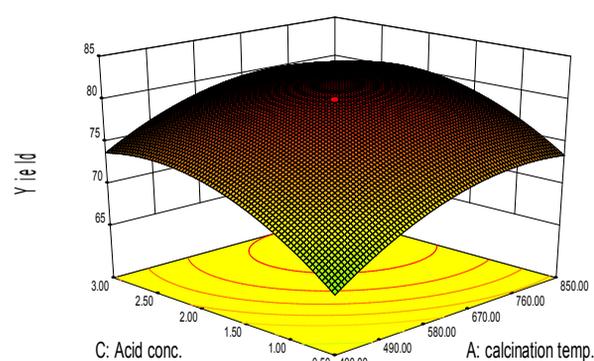Figure 3: Variation of alumina yield with calcination temperature and leaching temperature

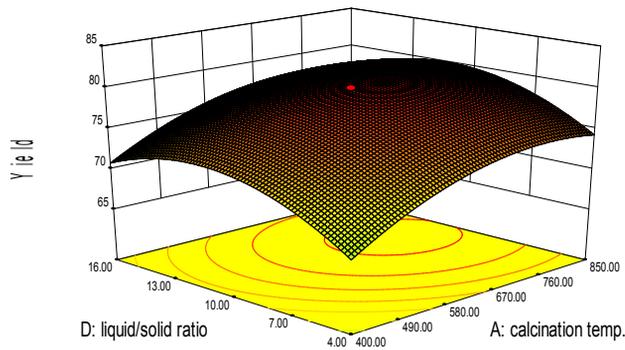Figure 4: Variation of alumina yield with calcination temperature and acid concentration.

Figure 5: Variation of alumina yield with calcination temperature and liquid-solid ratio
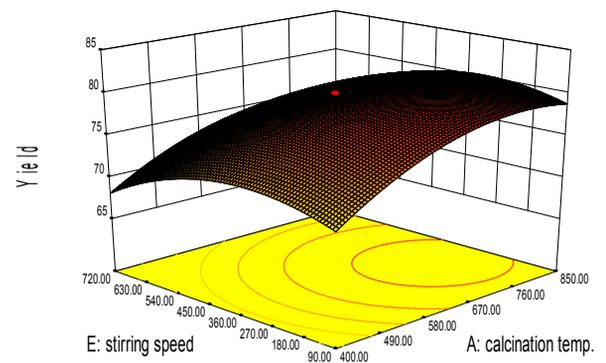

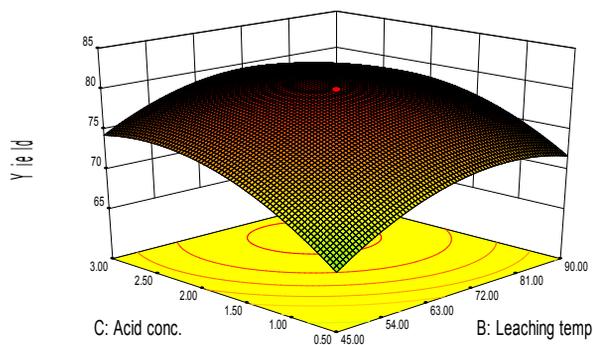Figure 6: Variation of alumina yield with calcination temperature and stirring speed

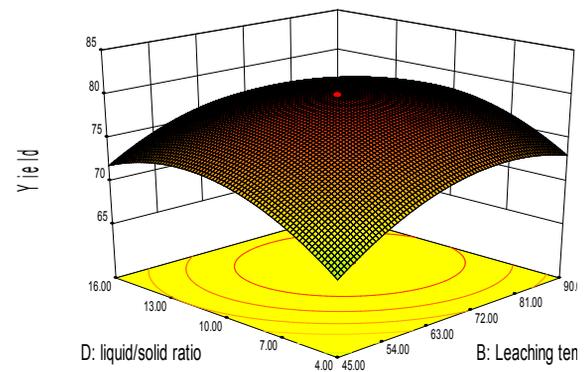Figure 7: Variation of alumina yield with leaching temperature and acid concentration

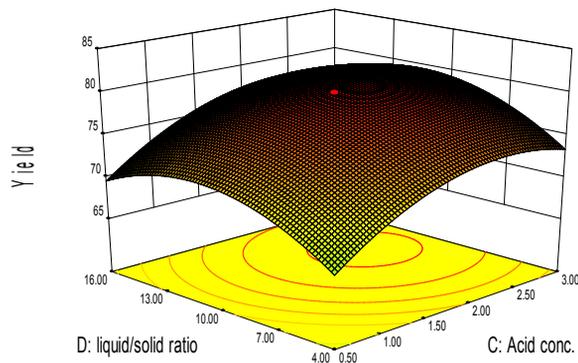Figure 8: Variation of alumina yield with leaching temperature and liquid-solid ratio

Figure 9: Variation of alumina yield with acid concentration and stirring speed
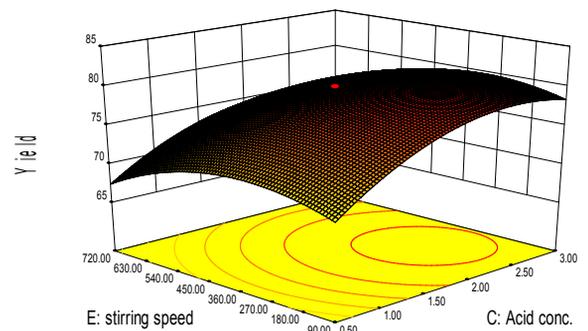

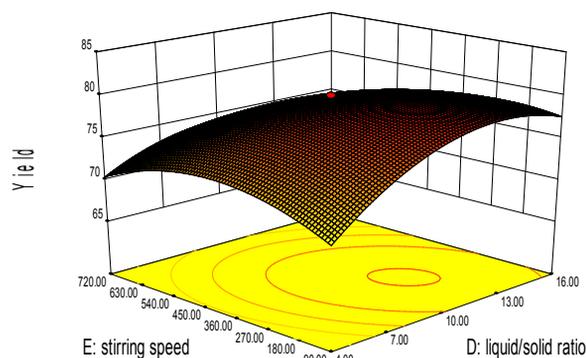Figure 10: Variation of alumina yield with acid concentration and stirring speed

Figure 11: Variation of alumina yield with solid - liquid ratio and stirring speed
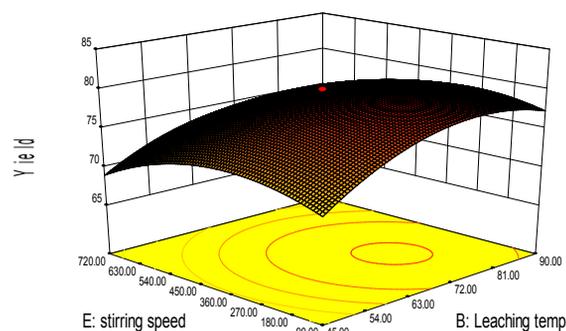
Figure 12: Variation of alumina yield with leaching temperature and stirring speed

3.4   Predicting the optimum condition of alumina yield

In order to further confirm the adequacy of the generated model in predicting the alumina yield, the optimum values of the five independent variables were used to perform a new set of experiments and the result is presented in Table 3.

Table 3: Comparing the experimental and predicted alumina yield using the optimum conditions

Variable	Optimum variables value	Alumina yield	
		Experimental	Predicted
Calcinations temp. (°C) X_1	677	80.07	78.91
Leaching temp. (°C) X_2	65	-	-
Acid conc. (mol/cm^3) X_3	1.9	-	-
Liquid-solid ratio (cm^3/g) X_4	10.4		
Stirring speed (rpm) X_5	442		

From the values of alumina yield obtained shown in Table 3, it can be established that a good agreement exists between the predicted alumina yield and the experimental alumina yield at the optimum levels and this confirms the validity of the generated model.

4.0   Conclusion

Developing a mathematical relationship to investigate the combined influence of different process variables on alumina yield is important in order to obtain the optimum conditions that guarantee high yield. In this research, a model equation that can be used to study the influence of five process variables on the yield of alumina from the local clay using HCl has been developed. The five process variables considered were calcination temperature, leaching temperature, stirring speed, liquid-solid ratio and acid concentration. Based on experimental design using the central composite design of the response surface methodology, the second-order polynomial regression equation appeared to fit the data most. The optimum alumina yield of 80.07% was obtained at calcinations temperature of 677.27°C; leaching temperature of 65.18°C; acid concentration of 1.9mol/cm^3; liquid-solid ratio of 10.36 and stirring speed of 442.92rpm. The correlation between the predicted and experimental responses was obtained as 0.9209. The developed model can guarantee optimal dissolution of the ore from the local clay.

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