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# Determination of Optimal Yield of Alumina From Local Clays by Factorially-Designed Experiments

Kenneth Kekpugile.Dagde* Nwankwo Chiedozie Onyebuchi
Department of Chemical and Petrochemical Engineering, Rivers State University, Port Harcourt, Rivers State, Nigeria

**Abstract**
Leaching by use of factorial design experiment was used to investigate the effect of process variables on the yield of alumina obtainable from four local clays from four different locations in Nigeria. Characterization of these clays obtained from previous work was observed in this order: Ikot-Abasi, Enito II, Akpugo and Awgbu containing 56.00%, 8.45%, 25.28% and 14.43% $Al_2O_3$ respectively. Fractional factorial design was used to develop a mathematical model used in the investigation. The process variables whose effect on alumina leaching investigated were: acid concentration (2M and 10M), calcinations Temperature (200$^oC$ and 1000$^oC$), calcinations time (15 and 75 minutes), leaching time (20 and 100 minutes) and particle size (75 and 1000μm) with the experiment conducted at constant boiling temperatures of the acids used (Nitric and Acetic acid). On optimization of the model developed from the factorial design experiment, optimal leaching conditions for each clay samples were obtained with corresponding yields of alumina presented as follows: Ikot-Abasi clay and nitric acid – yield of 78.86% alumina; Ikot-Abasi and Acetic – yield of 50.26% alumina; Enito II and nitric acid – yield of 53.14% alumina; Enito II clay and acetic acid – yield of 30.23% alumina; Awgbu clay and nitric acid – 62.74% alumina yield; Awgbu clay and acetic acid – alumina yield of 43.24%; Akpugo clay and nitric acid – alumina yield 75.43%; Akpugo clay and acetic acid – alumina yield 41.98%. The values of the yields obtained from the model optimization were validated by conducting the leaching experiment again in the laboratory under the optimized process conditions and were observed to closely match with a deviation ranging from 0.21 to 5.55%. From the results obtained, it was observed that the best yield was gotten from Ikot-Abasi clay which contained the highest percentage of alumina content.
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## 1. Introduction

In line with the recent need for diversification and improvement in our manufacturing industries as well as export, exploring other cheap and economic alternatives to well-known expensive ones for the manufacture of alumina is most welcome. The industrial application of speciality aluminium oxides in refractories, ceramics, polishing and abrasive applications. Large tonnages of aluminium hydroxide, from which alumina is derived, are used in the manufacture of zeolites, coating titania pigments, and as a fire retardant/smoke suppressant. (Osabor et al., 2009) Nigeria, is known to be blessed with vast amount of clay deposits in various geographical and regional locations, and it is growing in knowledge that most of these clays have significant alumina content which are extractible. (Mark 2010)

Several studies to this effect have been gathering momentum. Ajemba and Onukwuli, 2012 studied the effects of optimization of extracting alumina from the same clay (Nteje clay) using sulphuric acid. Response surface methodology was employed to optimize the sulphuric acid leaching of alumina from the clay based on the central composite rotatable design. Following this, a model was developed with the adequacy tested using the sequential model sum of squares. The optimum conditions generated for the process parameters showed that 81.87% was extractible.

In another recent development, the kinetic study of hydrochloric acid leaching of alumina from Agbaja clay (Kogi State, Nigeria) was investigated. It was observed that obtaining alumina from the clay was seriously hindered due to small surface area and presence of negative surface charges but by improved thermal activation and effects of most other process variables excluding particle size the yield substantially increased. A kinetic equation and optimal conditions were developed with activation energy calculated to be 34KJ/mol (Uchenna *et al*, 2015).

In the kingdom of Saudi Arabia, kaolinitic clay obtained from Riyadh area (N: 24 00'36", E: 47"44"03") was subjected to leaching using hydrochloric acid. The analysis of the aluminium ion present after leaching was carried out using the solar MS Atomic Absorption Spectrophotometer. The results of their experiment showed that 62.9% of alumina present in the local clay was extracted under optimum calcinations conditions of 600$^oC$ and 1 hour (Al-Zahrani and Abdul-Majid, 2009).

However, Ajemba and Onukwuli (2012), the other experiments performed have not been able to give a comprehensive outlook of having variable factors working together rather than individually.

In this study, the use of factorial design which is a quite simple approach, to give a new outlook of the process using half-fraction factorial design will be used. The factorial design helps us to study how the yield of alumina is affected by variance of all the operating factors simultaneously where the variation will only be the highest and lowest values of these factors. The results should provide us with the optimum operating process conditions necessary for extraction of alumina from these clays.

## 2. Materials and Methods

### 2.1 Factorial Design Experiment

This experiment was carried out using the fractional factorial design given by $2^k$, where k represents the number of process factors (5 in this case). By applying boundary conditions for the highest and lowest values of each process variable, the output (yield) for each case was obtained. The factorial design experiment provides a comprehensive check on the yields obtained with systematic values of the five variables (acid concentration, leaching time, calcinations temperature, calcinations time and particle size) used in the experiment. The un-coded values of the variable $X_5$, is obtained from the relation: $X_5 = -(X_1X_2X_3X_4)$; where, (-) represents the lowest value for a variable and (+) represents the highest value.

The results obtained from the four clays using the two different acids are presented in Tables 1 and 2. From the experiments, it was observed that the optimal yield of alumina from the factorial experiment was at the stage where the highest values of $X_1$, $X_2$, $X_3$ and $X_4$ and lowest value of $X_5$ were combined. The optimum variables were: $X_1$ – Leaching time – 100 minutes; $X_2$ – Calcination temperature – 1000°C; $X_3$ – Calcination time – 75 minutes; $X_4$ – Acid concentration – 10M and $X_5$ – Particle size - 75µm.

Further we represented each of the clays thus for simplicity: Ikot -Abasi Clay – A; Enito II Clay – B; Akpugo Clay – C and Awgbu Clay – D.

The experiment is presented in table 1 to 8:

Table 1: Factorial design results for the clays and nitric acid

| s/no | $X_1$ | $X_2$ | $X_3$ | $X_4$ | $X_5$ | Yield, Y (%) (A) | Yield, Y (%) (B) | Yield, Y (%) (C) | Yield, Y (%) (D) |
|---|---|---|---|---|---|---|---|---|---|
| 1 | - | - | - | - | - | 27.20 | 13.62 | 17.6 | 15.52 |
| 2 | + | - | - | - | + | 38.07 | 16.01 | 20.70 | 20.22 |
| 3 | - | + | - | - | + | 18.93 | 19.65 | 21.34 | 23.00 |
| 4 | + | + | - | - | - | 31.84 | 22.86 | 24.84 | 24.02 |
| 5 | - | - | + | - | + | 25.10 | 15.70 | 32.66 | 30.84 |
| 6 | + | - | + | - | - | 31.54 | 29.09 | 45.22 | 43.42 |
| 7 | - | + | + | - | - | 39.52 | 33.29 | 48.26 | 48.50 |
| 8 | + | + | + | - | + | 54.14 | 41.42 | 62.04 | 39.78 |
| 9 | - | - | - | + | + | 32.99 | 28.31 | 36.60 | 33.98 |
| 10 | + | - | - | + | - | 57.74 | 29.81 | 38.54 | 35.12 |
| 11 | - | + | - | + | - | 40.96 | 30.83 | 30.88 | 36.00 |
| 12 | + | + | - | + | + | 47.81 | 32.02 | 39.86 | 37.80 |
| 13 | - | - | + | + | - | 65.26 | 31.11 | 41.40 | 43.04 |
| 14 | + | - | + | + | + | 44.76 | 42.08 | 46.92 | 39.24 |
| 15 | - | + | + | + | + | 54.35 | 33.58 | 48.24 | 42.98 |
| 16 | + | + | + | + | - | 77.20 | 65.43 | 70.72 | 66.66 |

Table 2: Factorial design results for the clays and acetic acid

| s/no | $X_1$ | $X_2$ | $X_3$ | $X_4$ | $X_5$ | Yield, Y (%) (A) | Yield, Y (%) (B) | Yield, Y (%) (C) | Yield, Y (%) (D) |
|---|---|---|---|---|---|---|---|---|---|
| 1 | - | - | - | - | - | 17.55 | 9.82 | 11.64 | 9.94 |
| 2 | + | - | - | - | + | 25.01 | 11.31 | 13.86 | 7.92 |
| 3 | - | + | - | - | + | 16.50 | 12.24 | 15.84 | 9.98 |
| 4 | + | + | - | - | - | 20.95 | 22.85 | 18.02 | 13.92 |
| 5 | - | - | + | - | + | 16.74 | 15.27 | 27.10 | 22.18 |
| 6 | + | - | + | - | - | 21.07 | 22.78 | 27.16 | 27.72 |
| 7 | - | + | + | - | - | 26.35 | 26.19 | 31.20 | 28.28 |
| 8 | + | + | + | - | + | 35.68 | 28.63 | 38.86 | 35.46 |
| 9 | - | - | - | + | + | 25.44 | 12.89 | 13.94 | 16.26 |
| 10 | + | - | - | + | - | 41.22 | 22.19 | 18.10 | 18.04 |
| 11 | - | + | - | + | - | 28.93 | 22.64 | 42.82 | 19.62 |
| 12 | + | + | - | + | + | 31.45 | 21.90 | 37.38 | 35.24 |
| 13 | - | - | + | + | - | 45.39 | 26.79 | 28.60 | 30.90 |
| 14 | + | - | + | + | + | 31.29 | 27.07 | 28.10 | 33.80 |
| 15 | - | + | + | + | + | 34.50 | 22.94 | 29.18 | 31.92 |
| 16 | + | + | + | + | - | 60.65 | 37.08 | 49.50 | 48.36 |

Based on the experimental results performed and with the aid of the ANOVA model, a two level factorial model for the five factors were developed in the following format:

$$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \cdots + \beta_5 X_5 + \beta_{12} X_1 X_2 + \ \ldots \qquad (1)$$

The optimal conditions required to give the best yield of alumina was obtained by evaluation of the model equation using linear programming techniques with the aid of MATLAB software.

Using the factorial design, the notation for a multiple linear regression model for a fractional two level experiment with five variables interaction would be:

$$\begin{aligned} Y = \beta_O + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_4 X_4 + \beta_5 X_5 + \beta_{12} X_1 X_2 + \beta_{13} X_1 X_3 + \beta_{14} X_1 X_4 + \beta_{15} X_1 X_5 + \beta_{23} X_2 X_3 \\ + \beta_{24} X_2 X_4 + \beta_{25} X_2 X_5 + \beta_{34} X_3 X_4 + \beta_{35} X_3 X_5 + \beta_{45} X_4 X_5 + \beta_{123} X_1 X_2 X_3 + \beta_{124} X_1 X_2 X_4 \\ + \beta_{125} X_1 X_2 X_5 + \beta_{234} X_2 X_3 X_4 + \beta_{235} X_2 X_3 X_5 + \beta_{134} X_1 X_3 X_4 + \beta_{135} X_1 X_3 X_5 + \beta_{145} X_1 X_4 X_5 \\ + \beta_{245} X_2 X_4 X_5 + \beta_{345} X_3 X_4 X_5 + \beta_{1234} X_1 X_2 X_3 X_4 + \beta_{1235} X_1 X_2 X_3 X_5 + \beta_{1245} X_1 X_2 X_4 X_5 \\ + \beta_{1345} X_1 X_3 X_4 X_5 + \beta_{2345} X_2 X_3 X_4 X_5 + \beta_{12345} X_1 X_2 X_3 X_4 X_5 \\ \in \qquad (2) \end{aligned}$$

where $\beta_i$ represents the coefficients of the variables which could be the overall mean, the independent effect of each factor or the effects of the various interactions between the factors depending on what contextual form they appear.

Simplifying the model equation by assuming that the various interactions between the factors led to negligible terms (i.e. considering only independent events), equation can thus be reduced to:

$$Y = \beta_O + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_4 X_4 + \beta_5 X_5 + \in \qquad (3)$$

where, $\beta_o$ represents the overall mean, $\beta_1$, $\beta_2$, $\beta_3$, $\beta_4$ and $\beta_5$ represents the independent effects of the factors $X_1$, $X_2$, $X_3$, $X_4$ and $X_5$ respectively. $\in$ is the random error term or residual and Y is the yield of alumina.

The 'best' values of the coefficients were determined by setting up the sum of squares of the residuals given by,

$$S_r = \sum_{i=1}^{n} (Y_i - \beta_O - \beta_1 X_{1i} - \beta_2 X_{2i} - \beta_3 X_{3i} - \beta_4 X_{4i} - \beta_5 X_{5i})^2 \qquad (4)$$

By minimizing the derivatives of the squares of the residuals by setting all to zero, normal equations were developed from which the matrix was of the form:

$$\begin{bmatrix} n & \sum X_{1i} & \sum X_{2i} & \sum X_{3i} & \sum X_{4i} & \sum X_{5i} \\ \sum X_{1i} & \sum X_{1i}^2 & \sum X_{2i} X_{1i} & \sum X_{3i} X_{1i} & \sum X_{4i} X_{1i} & \sum X_{5i} X_{1i} \\ \sum X_{2i} & \sum X_{1i} X_{2i} & \sum X_{2i}^2 & \sum X_{3i} X_{2i} & \sum X_{4i} X_{2i} & \sum X_{5i} X_{2i} \\ \sum X_{3i} & \sum X_{1i} X_{3i} & \sum X_{2i} X_{3i} & \sum X_{3i}^2 & \sum X_{4i} X_{3i} & \sum X_{5i} X_{3i} \\ \sum X_{4i} & \sum X_{1i} X_{4i} & \sum X_{2i} X_{4i} & \sum X_{3i} X_{4i} & \sum X_{4i}^2 & \sum X_{5i} X_{4i} \\ \sum X_{5i} & \sum X_{1i} X_{5i} & \sum X_{2i} X_{5i} & \sum X_{3i} X_{5i} & \sum X_{4i} X_{5i} & \sum X_{5i}^2 \end{bmatrix} \begin{pmatrix} \beta_O \\ \beta_1 \\ \beta_2 \\ \beta_3 \\ \beta_4 \\ \beta_5 \end{pmatrix} = \begin{pmatrix} \sum Y_i \\ \sum Y_i X_{1i} \\ \sum Y_i X_{2i} \\ \sum Y_i X_{3i} \\ \sum Y_i X_{4i} \\ \sum Y_i X_{5i} \end{pmatrix} \qquad (5)$$

Using the above 6 x 6 matrix, the constants can be solved for with the un-coded form of the factorial design table. For the Awgbu clay and nitric acid, after solving for the constants, the model developed:

$$\boldsymbol{Y = 36.2575 + 2.0250X_1 + 3.5850X_2 + 8.0500X_3 + 5.5950X_4 - 2.7775X_5} \quad (6)$$

Awgbu clay and acetic acid:

$$\boldsymbol{Y = 24.3463 + 3.2113X_1 + 3.5013X_2 + 7.9813X_3 + 4.9213X_4 - 0.2513X_5} \quad (7)$$

Akpugo clay and nitric acid:

$$\boldsymbol{Y = 39.1138 + 4.4913X_1 + 4.1588X_2 + 10.3188X_3 + 5.0313X_4 - 0.5688X_5} \quad (8)$$

Akpugo clay and acetic acid:

$$\boldsymbol{Y = 26.9563 + 1.9163X_1 + 5.8938X_2 + 5.5063X_3 + 3.9963X_4 - 1.4238X_5} \quad (9)$$

Enito II clay and nitric acid:

$$\boldsymbol{Y = 30.3006 + 4.5394X_1 + 4.5844X_2 + 6.1619X_3 + 6.3456X_4 - 1.7044X_5} \quad (10)$$

Enito II clay and acetic acid:

$$\boldsymbol{Y = 21.4119 + 2.8144X_1 + 2.8969X_2 + 4.4319X_3 + 2.7756X_4 - 2.3806X_5} \quad (11)$$

Ikot-Abasi clay and nitric acid:

$$\boldsymbol{Y = 42.9631 + 4.8931X_1 + 2.6306X_2 + 6.0206X_3 + 9.6706X_4 - 3.4444X_5} \quad (12)$$

Ikot-Abasi clay and acetic acid:

$$\boldsymbol{Y = 29.9200 + 3.4950X_1 + 1.9563X_2 + 4.0388X_3 + 7.4388X_4 - 2.8438X_5} \quad (13)$$

Optimization of these models were carried out by method of linear programming using the MATLAB software. The model can be re-written in this format:

$$\boldsymbol{Y = 36.2575 + f(x)} \quad (14)$$

where $\boldsymbol{f(x)}$ was then optimized to give the results.

Table 3 were used for calculating the coefficient of determination and correlation coefficient which was used to test the accuracy of the models developed.

Table 3: Regression analysis of Awgbu clay and nitric acid

| s/no | $Y_i$ | $(Y_i-Y_m)^2$ | $U = \beta_0 + \beta_1X_1 + \beta_2X_2 + \beta_3X_3 + \beta_4X_4 + \beta_5X_5$ | $(Y_i - U)^2$ |
|---|---|---|---|---|
| 1 | 15.52 | 430.044 | 19.78 | 18.1476 |
| 2 | 20.22 | 257.201 | 18.275 | 3.783025 |
| 3 | 23 | 175.761 | 21.395 | 2.576025 |
| 4 | 24.02 | 149.756 | 31 | 48.7204 |
| 5 | 30.84 | 29.3493 | 30.325 | 0.265225 |
| 6 | 43.42 | 51.3014 | 39.93 | 12.1801 |
| 7 | 48.5 | 149.879 | 43.05 | 29.7025 |
| 8 | 39.78 | 12.408 | 41.545 | 3.115225 |
| 9 | 33.98 | 5.18701 | 25.415 | 73.359225 |
| 10 | 35.12 | 1.29391 | 35.02 | 0.01 |
| 11 | 36 | 0.06631 | 38.14 | 4.5796 |
| 12 | 37.8 | 2.37931 | 36.635 | 1.357225 |
| 13 | 43.04 | 46.0023 | 47.07 | 16.2409 |
| 14 | 39.24 | 8.89531 | 45.565 | 40.005625 |
| 15 | 42.98 | 45.192 | 48.685 | 32.547025 |
| 16 | 66.66 | 924.312 | 58.29 | 70.0569 |
| **Total** | **580.12** | **2289.03** | **580.12** | **356.6466** |

Using the formula:

$$r^2 = \frac{(S_t - S_r)}{S_t} \quad \text{where,} \quad (21)$$

$$S_t = \sum(Y_i - Y_m)^2 \ and \ S_r = \sum(Y_i - U)^2 \quad (22)$$

$r^2 = 0.8442, and\ r = 0.9403$

This showed that 84.42% of the original uncertainty has been explained by this model.

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Table 4: Regression analysis of Awgbu clay and acetic acid

| s/no | $Y_i$ | $(Y_i-Y_m)^2$ | $U = \beta_0 + \beta_1X_1 + \beta_2X_2 + \beta_3X_3 + \beta_4X_4 + \beta_5X_5$ | $(Y_i-U)^2$ |
|---|---|---|---|---|
| 1 | 9.94 | 207.541 | 4.9824 | 24.5778 |
| 2 | 7.92 | 269.823 | 10.9024 | 8.8947 |
| 3 | 9.98 | 206.391 | 11.4824 | 2.2572 |
| 4 | 13.92 | 108.708 | 18.4076 | 20.1386 |
| 5 | 22.18 | 4.69286 | 20.4424 | 3.0193 |
| 6 | 27.72 | 11.3819 | 27.3676 | 0.1242 |
| 7 | 28.28 | 15.474 | 27.9476 | 0.1105 |
| 8 | 35.46 | 123.514 | 33.8676 | 2.5357 |
| 9 | 16.26 | 65.3882 | 14.3224 | 3.7543 |
| 10 | 18.04 | 39.7694 | 21.2476 | 10.2887 |
| 11 | 19.62 | 22.3379 | 21.8276 | 4.8735 |
| 12 | 35.24 | 118.673 | 27.7476 | 56.1361 |
| 13 | 30.9 | 42.951 | 30.7876 | 0.0126 |
| 14 | 33.8 | 89.3724 | 36.7076 | 8.4541 |
| 15 | 31.92 | 57.3609 | 37.2876 | 28.8111 |
| 16 | 48.36 | 576.658 | 44.2128 | 17.1993 |
| **Total** | **389.54** | **1960.04** | **389.5408** | **191.1877** |

$$r^2 = 0.9025 \text{ and } r = 0.95$$

This showed that 90.25 percent uncertainty has been explained by the model.

Table 5: Regression analysis of Akpugo clay and nitric acid

| s/no | $Y_i$ | $(Y_i-Y_m)^2$ | $U = \beta_0 + \beta_1X_1 + \beta_2X_2 + \beta_3X_3 + \beta_4X_4 + \beta_5X_5$ | $(Y_i-U)^2$ |
|---|---|---|---|---|
| 1 | 17.6 | 462.844 | 15.6824 | 3.6772 |
| 2 | 20.7 | 339.068 | 23.5274 | 7.9942 |
| 3 | 21.34 | 315.908 | 22.8624 | 2.3177 |
| 4 | 24.84 | 203.741 | 32.9826 | 66.3019 |
| 5 | 32.66 | 41.6515 | 35.1824 | 6.3625 |
| 6 | 45.22 | 37.2857 | 45.3026 | 0.0068 |
| 7 | 48.26 | 83.653 | 44.6376 | 13.1218 |
| 8 | 62.04 | 525.611 | 52.4826 | 91.3439 |
| 9 | 36.6 | 6.31919 | 24.6074 | 143.8225 |
| 10 | 38.54 | 0.32925 | 34.7276 | 14.5344 |
| 11 | 30.88 | 67.7955 | 34.0626 | 10.1289 |
| 12 | 39.86 | 0.55681 | 41.9076 | 4.1927 |
| 13 | 41.4 | 5.22671 | 46.3826 | 24.8263 |
| 14 | 46.92 | 60.9368 | 54.2276 | 53.4010 |
| 15 | 48.24 | 83.2875 | 53.5626 | 28.3301 |
| 16 | 70.72 | 998.952 | 63.6828 | 49.5222 |
| **Total** | **625.82** | **3233.17** | **625.8208** | **519.8841** |

$$r^2 = 0.8392 \text{ and } r = 0.9161$$

Thus, 83.92% uncertainty has been explained by the model.

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Table 6: Regression analysis of Akpugo clay and acetic acid

| s/no | $Y_i$ | $(Y_i-Y_m)^2$ | $U = \beta_0 + \beta_1X_1 + \beta_2X_2 + \beta_3X_3 + \beta_4X_4 + \beta_5X_5$ | $(Y_i-U)^2$ |
|---|---|---|---|---|
| 1 | 11.64 | 234.589 | 11.0674 | 0.3279 |
| 2 | 13.86 | 171.513 | 12.0524 | 3.2674 |
| 3 | 15.84 | 123.572 | 20.0074 | 17.3672 |
| 4 | 18.02 | 79.8575 | 26.6876 | 75.1273 |
| 5 | 27.1 | 0.02065 | 19.2324 | 61.8991 |
| 6 | 27.16 | 0.04149 | 25.9126 | 1.5560 |
| 7 | 31.2 | 18.009 | 33.8676 | 7.1161 |
| 8 | 38.86 | 141.698 | 34.8526 | 16.0593 |
| 9 | 13.94 | 169.424 | 16.2124 | 5.1638 |
| 10 | 18.1 | 78.434 | 22.8926 | 22.9690 |
| 11 | 42.82 | 251.657 | 30.8476 | 143.3384 |
| 12 | 37.38 | 108.654 | 31.8326 | 30.7736 |
| 13 | 28.6 | 2.70175 | 30.0726 | 2.1686 |
| 14 | 28.1 | 1.30805 | 31.0576 | 8.7474 |
| 15 | 29.18 | 4.94484 | 39.0126 | 96.6800 |
| 16 | 49.5 | 508.218 | 45.6928 | 14.4948 |
| **Total** | **431.3** | **1894.64** | **431.3008** | **507.0559** |

$$r^2 = 0.7324 \; and \; r = 0.8558$$

Meaning that 73.24% of the uncertainty has been explained by the model.

Table 7: Regression analysis of Enito II clay and nitric acid

| s/no | $Y_i$ | $(Y_i-Y_m)^2$ | $U = \beta_0 + \beta_1X_1 + \beta_2X_2 + \beta_3X_3 + \beta_4X_4 + \beta_5X_5$ | $(Y_i-U)^2$ |
|---|---|---|---|---|
| 1 | 13.62 | 278.242 | 14.4117 | 0.6268 |
| 2 | 16.01 | 204.221 | 15.3967 | 0.3761 |
| 3 | 19.65 | 113.435 | 23.3517 | 13.7026 |
| 4 | 22.86 | 55.3625 | 30.0319 | 51.4361 |
| 5 | 15.7 | 213.178 | 22.5767 | 47.2890 |
| 6 | 29.09 | 1.46555 | 29.2569 | 0.0279 |
| 7 | 33.29 | 8.93651 | 37.2119 | 15.3813 |
| 8 | 41.42 | 123.641 | 38.1969 | 10.3884 |
| 9 | 28.31 | 3.96249 | 19.5567 | 76.6203 |
| 10 | 29.81 | 0.24069 | 26.2369 | 12.7670 |
| 11 | 30.83 | 0.28026 | 34.1919 | 11.3024 |
| 12 | 32.02 | 2.95634 | 35.1769 | 9.9660 |
| 13 | 31.11 | 0.65513 | 33.4169 | 5.3218 |
| 14 | 42.08 | 138.754 | 34.4019 | 58.9532 |
| 15 | 33.58 | 10.7545 | 42.3569 | 77.0340 |
| 16 | 65.43 | 1234.07 | 49.0371 | 268.7272 |
| **Total** | **484.81** | **2390.16** | **484.8096** | **659.9200** |

$$r^2 = 0.7239 \; and \; r = 0.8508$$

This means that 72.39% of the uncertainty in the model has been removed.

Table 8: Regression analysis of Enito II clay and acetic acid

| s/no | $Y_i$ | $(Y_i\text{-}Y_m)^2$ | $U = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_4 X_4 + \beta_5 X_5$ | $(Y_i\text{-}U)^2$ |
|---|---|---|---|---|
| 1 | 9.82 | 134.372 | 10.8737 | 1.1103 |
| 2 | 11.31 | 102.048 | 11.7413 | 0.1860 |
| 3 | 12.24 | 84.1237 | 11.9063 | 0.1114 |
| 4 | 22.85 | 2.06813 | 22.2963 | 0.3066 |
| 5 | 15.27 | 37.7229 | 14.9763 | 0.0863 |
| 6 | 22.78 | 1.8717 | 25.3663 | 6.6889 |
| 7 | 26.19 | 22.8302 | 25.5313 | 0.4339 |
| 8 | 28.63 | 52.101 | 26.3989 | 4.9778 |
| 9 | 12.89 | 72.6228 | 11.6637 | 1.5038 |
| 10 | 22.19 | 0.60544 | 22.0537 | 0.0186 |
| 11 | 22.64 | 1.50823 | 22.2187 | 0.1775 |
| 12 | 21.9 | 0.23824 | 23.0863 | 1.4073 |
| 13 | 26.79 | 28.924 | 25.2887 | 2.2539 |
| 14 | 27.07 | 32.0141 | 26.1563 | 0.8348 |
| 15 | 22.94 | 2.33509 | 26.3213 | 11.4332 |
| 16 | 37.08 | 245.489 | 36.7113 | 0.1359 |
| **Total** | **342.59** | **820.875** | **342.5904** | **31.6662** |

$$r^2 = 0.9614 \; and \; r = 0.9805$$

This means that 96.14% of the uncertainty has been explained by the model.

Table 9: Regression analysis of Ikot-Abasi clay and nitric acid

| s/no | $Y_i$ | $(Y_i\text{-}Y_m)^2$ | $U = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_4 X_4 + \beta_5 X_5$ | $(Y_i\text{-}U)^2$ |
|---|---|---|---|---|
| 1 | 27.2 | 248.475 | 23.1926 | 16.0593 |
| 2 | 38.07 | 23.9424 | 26.09 | 143.5204 |
| 3 | 18.93 | 577.59 | 21.565 | 6.9432 |
| 4 | 31.84 | 123.723 | 38.24 | 40.9600 |
| 5 | 25.1 | 319.09 | 28.345 | 10.5300 |
| 6 | 31.54 | 130.487 | 45.02 | 181.7104 |
| 7 | 39.52 | 11.8549 | 40.495 | 0.9506 |
| 8 | 54.14 | 124.923 | 43.3924 | 115.5109 |
| 9 | 32.99 | 99.4627 | 35.645 | 7.0490 |
| 10 | 57.74 | 218.357 | 52.32 | 29.3764 |
| 11 | 40.96 | 4.01241 | 47.795 | 46.7172 |
| 12 | 47.81 | 23.4924 | 50.6924 | 8.3082 |
| 13 | 65.26 | 497.152 | 54.575 | 114.1692 |
| 14 | 44.76 | 3.22885 | 57.4724 | 161.6051 |
| 15 | 54.35 | 129.661 | 52.9474 | 1.9673 |
| 16 | 77.2 | 1172.17 | 69.6224 | 57.4200 |
| **Total** | **687.41** | **3707.62** | **687.4096** | **942.7974** |

$$r^2 = 0.7457 \; and \; r = 0.8635$$

This means that 74.57% of the uncertainty has been explained by the model equation (18).

Table 10: Regression analysis of Ikot-Abasi clay and acetic acid

| s/no | $Y_i$ | $(Y_i-Y_m)^2$ | $U = \beta_0 + \beta_1X_1 + \beta_2X_2 + \beta_3X_3 + \beta_4X_4 + \beta_5X_5$ | $(Y_i-U)^2$ |
|---|---|---|---|---|
| 1 | 17.55 | 153.017 | 15.8349 | 2.9416 |
| 2 | 25.01 | 24.1081 | 17.1373 | 61.9794 |
| 3 | 16.5 | 180.096 | 14.0599 | 5.9541 |
| 4 | 20.95 | 80.4609 | 26.7375 | 33.4952 |
| 5 | 16.74 | 173.712 | 18.2249 | 2.2049 |
| 6 | 21.07 | 78.3225 | 30.9025 | 96.6781 |
| 7 | 26.35 | 12.7449 | 27.8251 | 2.1759 |
| 8 | 35.68 | 33.1776 | 29.1275 | 42.9353 |
| 9 | 25.44 | 20.0704 | 25.0249 | 0.1723 |
| 10 | 41.22 | 127.69 | 37.7025 | 12.3728 |
| 11 | 28.93 | 0.9801 | 34.6251 | 32.4342 |
| 12 | 31.45 | 2.3409 | 35.9275 | 20.0480 |
| 13 | 45.39 | 239.321 | 38.7901 | 43.5587 |
| 14 | 31.29 | 1.8769 | 40.0925 | 77.4840 |
| 15 | 34.5 | 20.9764 | 37.0151 | 6.3257 |
| 16 | 60.65 | 944.333 | 49.6927 | 120.0624 |
| **Total** | **478.72** | **2093.23** | **478.72** | **560.8225** |

$$r^2 = 0.7321 \text{ and } r = 0.8556$$

This shows that 73.21% of the uncertainty has been removed by the generated model.

## 3. Results and Discussion

Table 11: Comparison between model predictions and experimental yields of alumina

| | % Yield (MODEL) | % Yield (EXPERIMENT) | % Deviation |
|---|---|---|---|
| Awgbu Clay and Nitric Acid | 62.74 | 60.08 | 2.66 |
| Awgbu Clay and Acetic Acid | 43.24 | 39.65 | 3.59 |
| Akpugo Clay and Nitric Acid | 75.43 | 75.22 | 0.21 |
| Akpugo Clay and Acetic Aicd | 41.98 | 36.43 | 5.55 |
| Enito II Clay and Nitric Acid | 53.14 | 51.09 | 2.05 |
| Enito II Clay and Acetic Acid | 30.23 | 28.66 | 1.57 |
| Ikot-Abasi Clay and Nitric Acid | 78.66 | 74.33 | 4.33 |
| Ikot-Abasi Clay and Acetic Acid | 50.26 | 47.29 | 2.97 |

Table 11 shows the yields of alumina obtained by the developed optimization model of the factorial design experiment at optimum operating conditions generated from the developed model. The comparison between optimal model predictions and experimental data showed a percentage deviation ranges from 0.21% to 5.55% indicating very reasonable agreement. The Ikot-Abasi Clay had the highest yields of 78.66% model predictions as compared with 74.33% from experiment with nitric acid while the least yields was obtained from Enito 11 clay with 30.23% model predictions and 28.66% experimental value with acetic acid.

Also, in terms of the leaching experiments conducted in PART A of this journal, comparing the optimal yield, we have

Table 12: Comparison between yield of alumina obatined from model and leaching experiment

| | % Yield (MODEL) | % Yield ( LEACHING EXPERIMENT) |
|---|---|---|
| **Awgbu Clay and Nitric Acid** | 62.74 | 70.72 |
| **Awgbu Clay and Acetic Acid** | 43.24 | 49.50 |
| **Akpugo Clay and Nitric Acid** | 75.43 | 53.47 |
| **Akpugo Clay and Acetic Aicd** | 41.98 | 43.23 |
| **Enito II Clay and Nitric Acid** | 53.14 | 65.43 |
| **Enito II Clay and Acetic Acid** | 30.23 | 37.08 |
| **Ikot-Abasi Clay and Nitric Acid** | 78.66 | 68.10 |
| **Ikot-Abasi Clay and Acetic Acid** | 50.26 | 38.07 |

From Table 12, it is seen that comparing the results from the model with the optimal values from the leaching experiment, the later showed better yield of alumina except for Enito II clay. However, the marginal difference between the yields obtained for the Enito II clay was not very high.

The accuracy of the models developed above were tested as seen by finding their 'coefficient of determination' which was seen for all to lie above 70% making the model to be quite accurate as a large percentage of uncertainty had been explained (70% and above).

Optimization of the models using MATLAB is presented in the Table 13 for each of the sample:

Table 13: Optimal process variable and model predictions from MATLAB simmulation

| **Process Variables** | **$X_1$ (mins)** | **$X_2$ (°C)** | **$X_3$ (mins)** | **$X_4$ (M)** | **$X_5$ (µm)** | **Y (%)** |
|---|---|---|---|---|---|---|
| **Awgbu Clay and Nitric Acid** | 31.06 | 427.5 | 194.11 | 4.1 | 32.21 | 62.74 |
| **Awgbu Clay and Acetic Acid** | 40.72 | 348.4 | 27.96 | 3.0 | 7.99 | 43.24 |
| **Akpugo Clay and Nitric Acid** | 59.34 | 634.7 | 219.05 | 1.6 | 14.84 | 41.98 |
| **Akpugo Clay and Acetic Aicd** | 43.72 | 1475.0 | 63.68 | 1.3 | 14.84 | 41.98 |
| **Enito II Clay and Nitric Acid** | 118.5 | 919.7 | 92.56 | 9.4 | 190.00 | 53.14 |
| **Enito II Clay and Acetic Acid** | 37.69 | 247.8 | 107.16 | 2.0 | 4.90 | 30.23 |
| **Ikot-Abasi Clay and Nitric Acid** | 3.67 | 336.4 | 82.19 | 26.8 | 50.81 | 78.66 |
| **Ikot-Abasi Clay and Acetic Acid** | 43.05 | 46.4 | 42.06 | 18.8 | 65.33 | 50.26 |

It was observed that apart from Akpugo clay and nitric acid, Ikot-Abasi clay and acetic acid where the deviation for the yields were quite high, all others were close in values.

## 4. Conclusion

The results obtained from the factorial design experiment showed that the dependent variable, i.e. the percent yield of alumina, is significantly affected by the five variables investigated - leaching time, calcination time, calcination temperature, acid concentration and particle size. The models developed from this design showed a simpler and more efficient path in achieving optimal yield of alumina after these models had been optimized. Comparing the values obtained from optimization of the models with experimental results showed not very much deviation. The results obtained showed clearly that Ikot-Abasi, Akpugo and Awgbu clays provided the best yield of alumina achievable from these clays and these could also be tied to the analysis where it was seen that these clays had the highest alumina content as 56.00, 25.28 and 14.23% respectively. The factorial experiment was designed in a way that helped to reduce the total number of experiments where a good number of factors were involved while still achieving a better yields of alumina. From the factorial experiments conducted, it was seen that, the best yield was achieved from the last experiment where the upper limit of all values of factors minus particle size were combined interactively.

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