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PROCESS MODELING OF SULPHURIC ACID LEACHING OF IRON FROM OZORO CLAY

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Abstract

The local clay from Ozoro (6.24^{o}N, 5.55^{o}E) in Delta State Nigeria is relatively rich in iron content. The traditional method of obtaining optimum leaching conditions by varying just one factor at a time while keeping the rest constant is cumbersome and does not save time This research work was aimed at investigating the combined effects of calcinations temperature, leaching temperature, acid concentration, liquid-solid ratio and stirring speed on iron yield from the local clay in H_2SO_4using the Response Surface Methodology. The result obtained showed that the second order polynomial regression equation fitted the experimental data more appropriately with a correlation coefficient R^2 of 0.9189. The optimum conditions were calcinations temperature of 650^{o}C; leaching temperature of 70.02^{o}C; acid concentration of 1.89mol/cm^3; liquid-solid ratio of 10.67 and stirring speed of 379.80rpm at an optimum yield of 84.7%.

Keywords: Clay, leaching, iron oxide, sulphuric acid, calcinations, ANOVA

Introduction

Iron in form of oxide is a major component of clay. The characterization of several clays by different researchers revealed that most Nigerian clays are rich in iron oxide (Ogbemudia et al., 2010; Ajemba and Onukwuli, 2012; Ogbuagu et al., 2007; Lori et al., 2007). Ozoro in Delta

State Nigeria has huge deposits of clay that is very rich in iron oxide. Suong Oh Lee et al., (2006) found that even traces of iron oxide can be removed by acid washing. Most dissolution studies of iron oxides have been confined to their preferential dissolution from soils so that other minerals could be concentrated and studied (Mitchell et al., 1964). Ambikadevi and Lalithambika (2000) in their investigation of several organic acid leaching of iron from clay found that oxalic acid is more efficient. The extent of the dissolution reaction depends on both clay mineral type and reaction conditions, such as the acid/clay ratio, acid concentration, time, and temperature of the reaction (Abali et al, 2006; Lui et al, 2010). Many researchers like Ozdemir and Cetisli, (2005), Ajemba and Onukwuli, (2012), Poppleton and Sawyer, (1977), Eisele, (1983), Al-Zahrani and Abdul, (2009) have identified calcinations temperature, leaching temperature, acid concentration, liquid-solid ratio and stirring speed to be very important process parameters that affects the leaching of minerals from clays. In the process optimization of sulphuric acid leaching of alumina from Nteje clay Using Central Composite Rotatable Design, Ajemba and Onukwuli, (2012) found the optimum conditions to be 675^0C for calcinations temperature; 97^0C for leaching temperature; 2.97 for mol/l acid concentration; 0.03 g/ml for solid-to-liquid ratio; and 476 rpm for stirring speed to achieve 81.87% alumina yield. In the extraction of alumina from local Saudi clay, Al-Zahrani and Abdul-Majid, (2009), obtained optimum calcinations temperature of 650^oC. Ambikadevi and Lalithambika, (2000) found oxalic acid (0.05-0.15M) to be the best extractant for removing iron from iron compounds. The dissolution was found to increase with acid concentration within the range (0.05-0.15M) studied.

Classical and conventional methods of studying a process by maintaining other parameters involved at an unspecified constant level does not depict the combined effect of all the parameters involved (Kumar, Prasad & Mishra, 2008). This is referred as one factor at a time. This method is also time consuming and requires large number of experiments to determine optimum levels, which are unreliable. These limitations can be avoided by optimizing all the parameters collectively by statistical experimental design such as response surface methodology (Ko, Porter, & Mc kay, 2000). Response surface methodology is based on polynomial surface analysis and it is a collection of mathematical and statistical techniques that are useful for the modeling and analysis of problems in which a response of interest is influenced by several variables (Park and Ahn, 2004). The main objective of response surface methodology is to determine the optimum operational conditions for the process (Kumar et al., 2008).

The application of statistical experimental design techniques in leaching process development can result in improved product yields, closer confirmation of the output response to nominal and target requirement reduced process variability and reduced development time and overall costs (Annadurai, Juang, and Lee, 2002).

In this work, the optimum process conditions for the leaching of iron from calcined Ozoro clay in hydrochloric acid is studied applying the central composite design of the response surface methodology.

Materials and methods

Sample preparation

The local clay used in this work was obtained from Ozoro (6.24^{o}N, 5.55^{o}E) in Delta State Nigeria. The mined clay was soaked in water for two days after which the impurities were removed and the clay sun-dried for 24 hours then oven dried at 60^{O}C for 18hours to enhance aggregation. The clay was subjected to a calcinations process in the temperature range of 400^{o}C-900^{o}C for a period of 1hr. the resulting samples were all ground to the same particle size of 0.054mm and were properly labeled.

Leaching experiment

Leaching experiments were carried based on the experimental plan presented in Table 2 in a reflux system on a magnetic stirrer model SH-85-2 and temperature was measured using thermometer. 12g of the calcined sample was added to already determine volume of the acid based on the liquid-solid ratio for each run and heated while stirring continuously. The reaction was performed for 90min. At the end of the reaction, 1ml of leaching solution was taken out of the round bottom flask by a pipette. The collected sample of leached liquor was cooled, filtered and used for alumina estimation using the Atomic Adsorption Spectrophotometer (AAS). The dissolution fraction of the alumina in the slurry was calculated by:

$$X = \frac{\text{amount of } Al^{3+} \text{ in the solution}}{\text{total amount of } Al^{3+} \text{ in original sample}} \times 100 \qquad (1)$$

Design of experiment for the optimization study

To investigate the combine effects of the five different factors (independent variables) calcinations temperature, leaching temperature, acid concentration, liquid-solid ratio and stirring speed on iron yield and derive a model, a Central Composite Rotatable Design of $2^5 = 32$ plus six centre points and (2x6=12) star points leading to a total of 50 experiments were performed.

The range of the five independent variables were presented in Table 1 while the experimental design plan obtained from the Central Composite Design were presented in Table 2.

Table 1.Range of the independent variables.

Independent variables	Symbols	Range and levels				
		-α	-1	0	+1	+α
Calcinations temp. (^oC)	X_1	89.9	400	625	900	1160
Leaching temp. (^oC)	X_2	13.9	45	67.5	85	121
Acid conc. (mol/cm^3)	X_3	-1.22	0.5	1.75	3.0	4.72
Liquid-solid ratio (cm^3/g)	X_4	-4.27	4	10	16	24.27
Stirring speed (rpm)	X_5	-344	90	405	720	1154

Table 2 Experimental design for iron yield with sulphuric acid.

Std. order	Calcinations temp for 1hr. (^oC) X_1		Leaching temp (^oC) X_2		Acid conc. (mol/cm^3) X_3		Liquid/solid ratio (cm^3/g) X_4		Stirring speed (rpm) X_5	
	Coded	Real	Coded	Real	Coded	Real	Coded	Real	Coded	Real
1	-1	400	-1	45	-1	0.5	-1	4	-1	90
2	+1	850	-1	45	-1	0.5	-1	4	-1	90
3	-1	400	+1	90	-1	0.5	-1	4	-1	90
4	+1	850	+1	90	-1	0.5	-1	4	-1	90
5	-1	400	-1	45	+1	3	-1	4	-1	90
6	+1	850	-1	45	+1	3	-1	4	-1	90
7	-1	400	+1	90	+1	3	-1	4	-1	90
8	+1	850	+1	90	+1	3	-1	4	-1	90
9	-1	400	-1	45	-1	0.5	1	16	-1	90
10	+1	850	-1	45	-1	0.5	1	16	-1	90
11	-1	400	+1	90	-1	0.5	1	16	-1	90
12	+1	850	+1	90	-1	0.5	1	16	-1	90
13	-1	400	-1	45	+1	3	1	16	-1	90
14	+1	850	-1	45	+1	3	1	16	-1	90
15	-1	400	+1	90	+1	3	1	16	-1	90
16	+1	850	+1	90	+1	3	1	16	-1	90
17	-1	400	-1	45	-1	0.5	-1	4	1	720
18	+1	850	-1	45	-1	0.5	-1	4	1	720
19	-1	400	+1	90	-1	0.5	-1	4	1	720
20	+1	850	+1	90	-1	0.5	-1	4	1	720
21	-1	400	-1	45	+1	3	-1	4	1	720
22	+1	850	-1	45	+1	3	-1	4	1	720
23	-1	400	+1	90	+1	3	-1	4	1	720
24	+1	850	+1	90	+1	3	-1	4	1	720
25	-1	400	-1	45	-1	0.5	1	16	1	720
26	+1	850	-1	45	-1	0.5	1	16	1	720
27	-1	400	+1	90	-1	0.5	1	16	1	720
28	+1	850	+1	90	-1	0.5	1	16	1	720
29	-1	400	-1	45	+1	3	1	16	1	720
30	+1	850	-1	45	+1	3	1	16	1	720
31	-1	400	+1	90	+1	3	1	16	1	720
32	+1	850	+1	90	+1	3	1	16	1	720
33	-2	89.8568	0	67.5	0	1.75	0	10	0	405
34	+2	1160	0	67.5	0	1.75	0	10	0	405
35	0	625	-2	13.9	0	1.75	0	10	0	405
36	0	625	+2	121	0	1.75	0	10	0	405
37	0	625	0	67.5	-2	-1.22	0	10	0	405
38	0	625	0	67.5	+2	4.72	0	10	0	405

39	0	625	0	67.5	0	1.75	-2	-4.27	0	405
40	0	625	0	67.5	0	1.75	2	24.27	0	405
41	0	625	0	67.5	0	1.75	0	10	-2	-344
42	0	625	0	67.5	0	1.75	0	10	2	1154
43	0	625	0	67.5	0	1.75	0	10	0	405
44	0	625	0	67.5	0	1.75	0	10	0	405
45	0	625	0	67.5	0	1.75	0	10	0	405
46	0	625	0	67.5	0	1.75	0	10	0	405
47	0	625	0	67.5	0	1.75	0	10	0	405
48	0	625	0	67.5	0	1.75	0	10	0	405
49	0	625	0	67.5	0	1.75	0	10	0	405
50	0	625	0	67.5	0	1.75	0	10	0	405

Results and discussions

The combined effects of the process parameters on the experimental leaching efficiency of iron were studied. It was observed that the efficiency of removal of iron from the clay increased with increasing calcinations temperature, leaching temperature, acid concentration, liquid/solid ratio and stirring speed. The experimental result of the combined effect of the five process variables on the response (iron yield) were presented in Table 3.

Table 3. Effect of the five process variables on the response (iron yield).

Std. order	Calcinations temp for 1hr (^oC) X_1		Leaching temp (^oC) X_2		Acid conc. (mol/cm^3) X_3		Liquid/solid ratio (cm^3/g) X_4		Stirring speed (rpm) X_5		Yield (%)	
	Coded	Real	Coded	Real	Coded	Real	Coded	Real	Coded	Real	Exp. Values	Predicted values
1	-1	400	-1	45	-1	0.5	-1	4	-1	90	42.6	48.9
2	+1	850	-1	45	-1	0.5	-1	4	-1	90	62.9	60.5
3	-1	400	+1	90	-1	0.5	-1	4	-1	90	61.7	60.3
4	+1	850	+1	90	-1	0.5	-1	4	-1	90	70.5	69.1
5	-1	400	-1	45	+1	3	-1	4	-1	90	58.7	61.1
6	+1	850	-1	45	+1	3	-1	4	-1	90	72	69.2
7	-1	400	+1	90	+1	3	-1	4	-1	90	70.9	68
8	+1	850	+1	90	+1	3	-1	4	-1	90	74.8	73.2
9	-1	400	-1	45	-1	0.5	1	16	-1	90	62.1	59.4
10	+1	850	-1	45	-1	0.5	1	16	-1	90	70.2	69.4
11	-1	400	+1	90	-1	0.5	1	16	-1	90	61.7	67.2
12	+1	850	+1	90	-1	0.5	1	16	-1	90	77.3	74.3
13	-1	400	-1	45	+1	3	1	16	-1	90	73.1	71.6
14	+1	850	-1	45	+1	3	1	16	-1	90	75.9	78.1
15	-1	400	+1	90	+1	3	1	16	-1	90	78.1	74.9
16	+1	850	+1	90	+1	3	1	16	-1	90	76.5	78.4
17	-1	400	-1	45	-1	0.5	-1	4	1	720	49.8	51.7
18	+1	850	-1	45	-1	0.5	-1	4	1	720	63.5	62.9
19	-1	400	+1	90	-1	0.5	-1	4	1	720	65.4	60.4
20	+1	850	+1	90	-1	0.5	-1	4	1	720	68.3	68.8
21	-1	400	-1	45	+1	3	-1	4	1	720	67.1	62.6
22	+1	850	-1	45	+1	3	-1	4	1	720	72.1	70.3
23	-1	400	+1	90	+1	3	-1	4	1	720	63.2	66.8
24	+1	850	+1	90	+1	3	-1	4	1	720	71.4	71.5
25	-1	400	-1	45	-1	0.5	1	16	1	720	56.3	53.1

26	+1	850	-1	45	-1	0.5	1	16	1	720	58.9	62.7
27	-1	400	+1	90	-1	0.5	1	16	1	720	55.4	58.2
28	+1	850	+1	90	-1	0.5	1	16	1	720	67.1	64.8
29	-1	400	-1	45	+1	3	1	16	1	720	57.9	64
30	+1	850	-1	45	+1	3	1	16	1	720	73.2	70.1
31	-1	400	+1	90	+1	3	1	16	1	720	65.8	64.5
32	+1	850	+1	90	+1	3	1	16	1	720	73.9	67.6
33	-2	89.8568	0	67.5	0	1.75	0	10	0	405	61.3	58.3
34	+2	1160	0	67.5	0	1.75	0	10	0	405	70.4	75.9
35	0	625	-2	13.9	0	1.75	0	10	0	405	58.8	57.3
36	0	625	+2	121	0	1.75	0	10	0	405	63.9	68.0
37	0	625	0	67.5	-2	-1.22	0	10	0	405	54.7	53.8
38	0	625	0	67.5	+2	4.72	0	10	0	405	68.2	71.7
39	0	625	0	67.5	0	1.75	-2	-4.27	0	405	53.2	55.5
40	0	625	0	67.5	0	1.75	2	24.27	0	405	63.1	63.4
41	0	625	0	67.5	0	1.75	0	10	-2	-344	68.6	69.1
42	0	625	0	67.5	0	1.75	0	10	2	1154	57.6	59.7
43	0	625	0	67.5	0	1.75	0	10	0	405	84.1	84.7
44	0	625	0	67.5	0	1.75	0	10	0	405	84.6	84.7
45	0	625	0	67.5	0	1.75	0	10	0	405	84.7	84.7
46	0	625	0	67.5	0	1.75	0	10	0	405	84.7	84.7
47	0	625	0	67.5	0	1.75	0	10	0	405	84.3	84.7
48	0	625	0	67.5	0	1.75	0	10	0	405	84.7	84.7
49	0	625	0	67.5	0	1.75	0	10	0	405	84.5	84.7
50	0	625	0	67.5	0	1.75	0	10	0	405	84.1	84.7

Model generation

The data generated from the experiments (Table 3) were statistically analyzed to identify the significant main interactions and quadratic effects. Multi regression analysis was performed on the data to obtain quadratic response surface model for the leaching of iron from the clay. The final second order (quadratic model) polynomial predictive equation obtained for the analysis of iron leaching with H_2SO_4 from Ozoro clay is presented in equation (2) as follows:

$$Y\ Fe2O3 = 84.67 + 3.70X1 + 2.26X2 + 3.76X3 + 1.66X4 - 1.98X5 - 0.73X1X2 - 0.90X1X3 - 0.42X1X4 - 0.12X1X5 - 1.14X2X3 - 0.92X2X4 - 0.70X2X5 - 0.003125X3X4 - 0.35X3X5 - 2.28X4X5 - 3.10X12 - 3.90X_2^2 - 3.88X3^2 - 4.46X4^2 - 3.56X5^2$$

Test of Adequacy of the Model

Table 4. Adequacy test of the model

Source	**Sum of squares**	**Degree of freedom**	**Mean squares**	**F-value**	**P-value**	**Remarks**
Sequential sum of squares						
Linear	1718.061	5	343.6122	4.461693	0.0023	Significant
2FI	303.9606	10	30.39606	0.335035	0.9651	Not significant
Quadratic	2415.38	5	534.1342	37.41713	< 0.0001	Significant
Cubic	239.5728	15	19.1954	2.132017	0.0826	Not significant
Source	**Std Dev.**	**R^2**	**Adjusted R^2**	**Predicted R^2**	**PRESS**	**Remarks**
Model summary statistics						
Linear	8.775754	0.336435	0.26103	0.229072	3936.874	Inadequate
2FI	9.524966	0.395957	0.129467	0.1323	4431.06	Inadequate
Quadratic	3.778244	0.918934	0.863026	0.702305	1520.232	Adequate
Cubic	3.000567	0.975317	0.91361	-0.20025	6129.295	Inadequate

The adequacy of the model was tested using the sequential model sum of squares and the model summary statistics (Table 4). At 0.05 level of significant, both the linear terms and the quadratic models are significant but with coefficient of correlation (R^2), only the quadratic model is significant which gave the regression coefficient of 91.89 showing that the model adequately explained 91.89% of the variation and also, the R^2 adjusted of 0.8630 is in reasonable agreement with the R^2 predicted of 0.7023 for the quadratic model.

ANOVA and regression analysis for the response

Table 5. ANOVA for the Quadratic model

Source	Sum of squares	Degree of freedom	F-value	P-value (prod.>F)
Model	4692.693	20	16.43661	< 0.0001
X_1	593.5779	1	41.58128	< 0.0001
X_2	220.9622	1	15.47883	0.0005
X_3	613.4732	1	42.97498	< 0.0001
X_4	119.839	1	8.39495	0.0071
X_5	170.2089	1	11.92346	0.0017
X_1X_2	17.25781	1	1.208943	0.2806
X_1X_3	25.74031	1	1.803159	0.1897
X_1X_4	5.695313	1	0.398968	0.5326
X_1X_5	0.427813	1	0.029969	0.8638
X_2X_3	41.63281	1	2.916459	0.0984
X_2X_4	26.82781	1	1.87934	0.1809
X_2X_5	15.54031	1	1.088629	0.3054

X_3X_4	0.000313	1	2.19E-05	0.9963
X_3X_5	3.850313	1	0.269722	0.6075
X_4X_5	166.9878	1	11.69782	0.0019
X_1^2	534.4926	1	37.44224	< 0.0001
X_2^2	843.849	1	59.11325	< 0.0001
X_3^2	836.2103	1	58.57815	< 0.0001
X_4^2	1106.624	1	77.52113	< 0.0001
X_5^2	715.1867	1	50.10021	< 0.0001
Residual	413.9786	29		
Lack of Fit	413.4999	22	274.816	< 0.0001
Pure Error	0.47875	7	16.43661	
Cor Total	5106.671	49	41.58128	

The analysis of variance (NOVA) was presented in Table 5. The P values were used as a tool to check the significance of each of the coefficients, which in turn are necessary to understand the pattern of the mutual interactions between the test variables (Shrivastava, Saudagar, Bajaj, and Singhal, 2008). The larger the magnitude of F-test value and the smaller the magnitude of P-values, the higher the significance of corresponding coefficient (Alam, Muyibi, Kamaldin, 2008). Values of P less than 0.05 indicate that the model terms are significant. The final mathematical model by eliminating the insignificant terms and interactions is expressed in equation (3).

$$Fe_2O_3 = 84.67 + 3.70X_1 + 2.26X_2 + 3.76X_3 + 1.66X_4 - 1.98X_5 - 2.28X_4X_5 - 3.10{X_1}^2 - 3.90{X_2}^2 - 3.88{X_3}^2 - 4.46{X_4}^2 - 3.56{X_5}^2$$

Further validation of the quadratic model was done with the Normal probability of residuals plot (Fig.1) and plot of predicted versus actual (Fig.2). The residuals can be judged as normally distributed; therefore normality assumptions of the response is satisfied

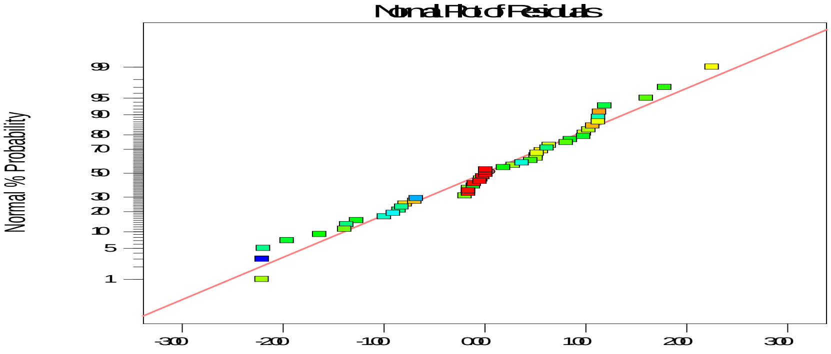

Fig.1. Plot of normal probability Vs residuals

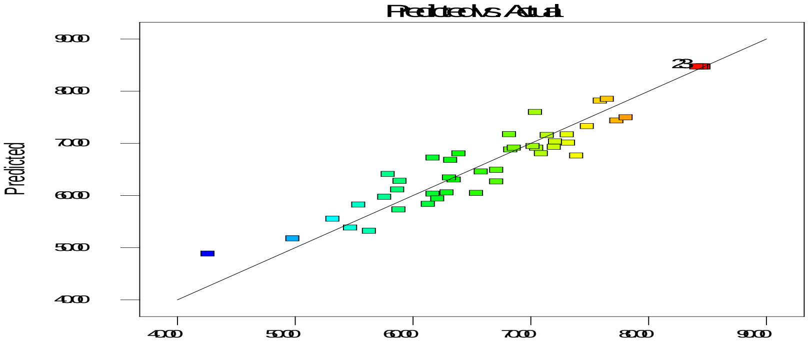

Fig.2. plot of predicted values Vs actual values

Response Surface Plots of Iron Dissolution in H_2SO_4

The interactive effects of the process variables on the percentage iron yield were studied by plotting three dimensional surface curves against any two independent variables, while keeping other variables at their central (0) level. The 3D curves of the response (percentage yield) and contour plots from the interactions between the variables are shown in Figures 3 – 13.

The interactive effect of calcinations on iron yield (Figures 3, 4, 5 and 6) revealed that iron yield increased with increased calcinations temperature up to 650^0C after which it began to decrease.

The interactive effect of leaching temperature (Figures 4, 8, 9 and 10) showed that increase in leaching temperature increased yield of iron for up to around 70^0C, and further increase had no significant improvement in iron yield The interactive effect of acid concentration (Figures 5, 8, 11 and 12) revealed that iron dissolution increased as acid concentration increased. Optimum iron yield was obtained at around acid concentration of 1.9mol/cm^3, and further increase had no significant improvement in iron yield. The interactive effect of acid-clay ration (Figures 6, 9, 11 and 13) showed increased iron yield with increase in acid-clay ratio. The optimum result was achieved with the ratio of 10.5. The interactive effect ofstirring speed (Figures 7, 10, 12 and 13) revealed an increase in iron yield with stirring speed up to the optimum value of 380rmp, and further increase had no significant improvement on iron yield.

Validation of Optimization result

The optimum conditions predicted for obtaining 84.7% yield in the dissolution of iron with H_2SO_4 from Ozoro clay were as follows: calcinations temperature of 650^oC; leaching temperature of 70.02^oC; acid concentration of 1.89mol/cm^3; liquid-solid ratio of 10.67 and stirring speed of 379.80rpm. The optimization was performed using the numerical method of the Design Expert version 8.1 by State Ease U.S.A. this value is in close agreement with the experimental value of 83.9% performed at the same optimum values of the process variables.

Conclusion

Response surface methodology was used to study the effect of key parameters on percentage iron yield. Process optimization was accomplished by applying Box Wilson design. A central composite rotatable design with 50 assays was successfully employed for experimental design.

From the result obtained in this work, a yield of 84.7% can be achieved at the following optimum conditions: calcinations temperature of 650^{o}C; leaching temperature of 70.02^{o}C; acid concentration of 1.89mol/cm^3; liquid-solid ratio of 10.67 and stirring speed of 379.80rpm.

This study clearly shows that Box Wilson design is undoubtedly a good technique for studying
the effect of major process parameters on response factor by significantly reducing the number of experiments in the batch study of a leaching process.

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Appendix

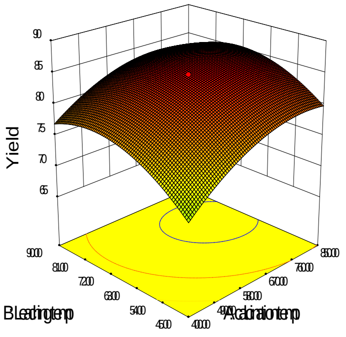

Fig.3. Effect of calc. temp and leaching temp on iron yield

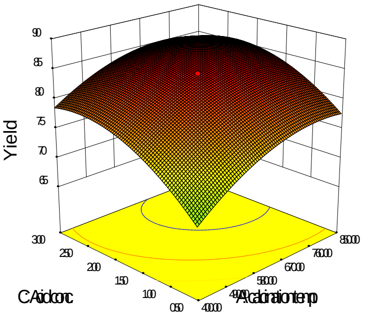

Fig.4. Effect of calc. temp and acid conc on iron yield

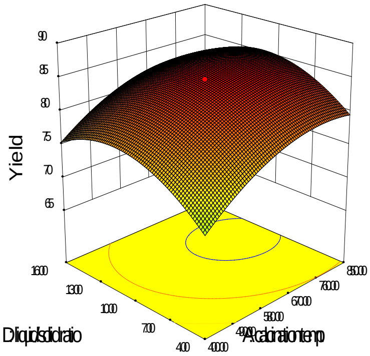

Fig.5. Effect of calc. temp and liquid-solid ratio on iron yield

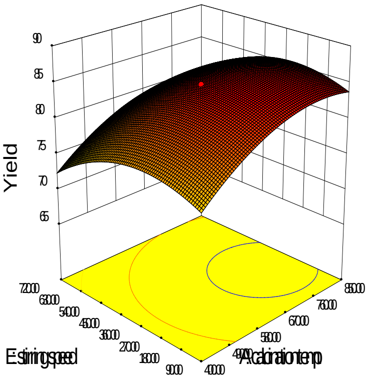

Fig.6. Effect of calc. temp and stirring speed on iron yield

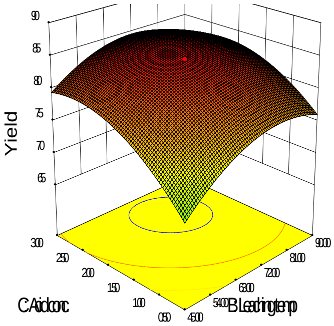

Fig.7. Effect of leaching temp and acid conc on iron yield

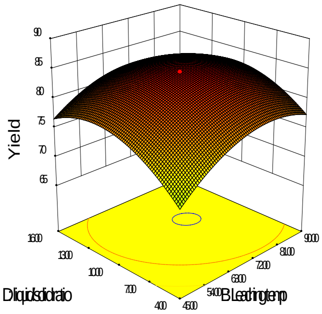

Fig.8. Effect of leaching temp and liquid-solid ratio on iron yield

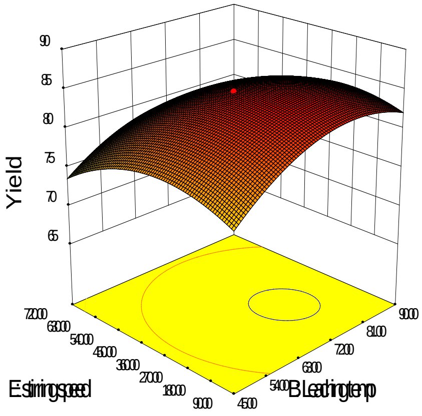

Fig.9. Effect of leaching temp and stirring speed on iron yield

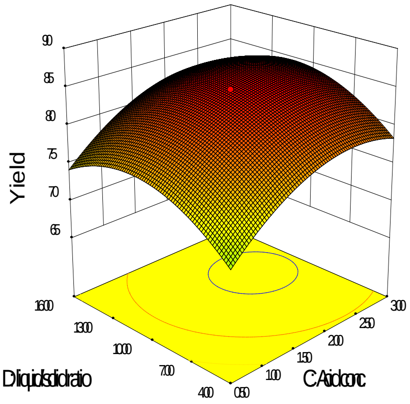

Fig.10.Effect of acid conc and liquid-solid ratio on iron yield

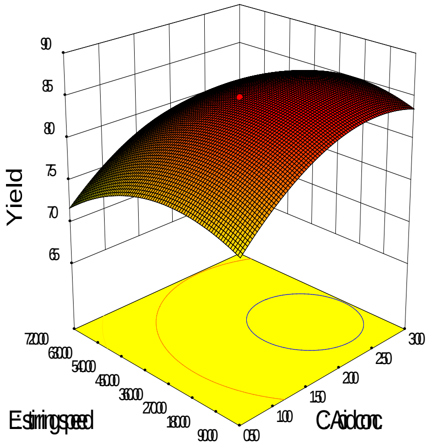

Fig.11. Effect of acid conc and stirring speed on iron yield

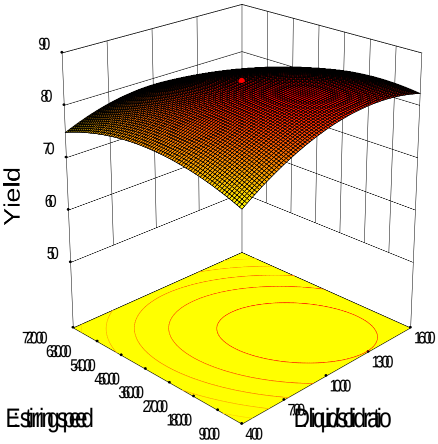

Fig.12. Effect of liquid-solid ratio and stirring speed on iron yield