OPTIMIZATION OF LEACHING PARAMETERS FOR THE EXTRACTION OF COPPER FROM HEMATITE-DOMINATED COPPER ORE USING RESPONSE SURFACE METHODOLOGY (RSM)

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Abstract

The optimization of recovery of copper from Akiri hematite-dominated copper ore using hydrometallurgy was investigated in this study. The Akiri copper ore deposit hosts a high-grade copper mineral from which copper metal can be extracted. However, the ore is dominated by gangue minerals that need to be mechanically reduced for efficient copper recovery. The purpose of this study is to optimize the extraction of the metal from hematite-dominated copper ore. The experiments that were carried out in the course of this study include crushing, pulverization,mineralogical and chemical characterizations of the sample and sulpheric acid leaching. Response surface methodology was used to optimize the system parameters namely;temperature, concentration of the leachant and contact time so that an efficient method will be developed for the extraction of copper.Chalcopyrite, covellite and cuprite are the copper minerals while the gangues minerals were quartz ,mica, hematite,etc.on the characterization of the copper ore. The major oxides in the ore are hematite ,copper oxide and silica and revealed that the ore contains 4.61% copper and 65.8% iron. Effect of three independent factors like concentration ,temperature and contact time for copper extraction from the hematite-dominated copper ore was studied.Central composite design method was applied to the proposed quadratic model that connect the factors used for best copper extraction at the best process condition. The work shows that concentration of the acid was the best efficient factor for copper extraction compare to temperature and contact time. This may be as a result of high value of F-statistics for the concentration of the leachant, which effects to high level of copper extraction.Experimental and predicted values for weight loss from the copper ore were obtained as 39.10% and 39.03% at optimum conditions, respectively. The optimum conditions of 1.5M acid concentration, 90°C reaction temperature and 90minutes contact time were obtained and from which 6.64%Cu at recovery of 92.0% and 2.31%Fe was obtained without stirring. Also, the ore was subjected to leaching with stirring at 400rpm with the optimum conditions obtained to know the effect. The grade obtained was 7.84%Cu at recovery of 83.51% and iron content 5.47%. This shows that leaching without stirring is the best option against leaching with stirring to extract the copper and to reduce the iron and other gangues contents in the copper ore.The activation energies were estimated as 13.20kJ/mol and 22.67kJ/mol for liquid film diffusion and diffusion product layer respectively, the values indicate that the leaching rate is controlled by diffusion process.

1.0 INTRODUCTION

Copper as a metal is a nonmagnetic base type with average concentration in the earth's crust of about 50ppm. The cutoff grade for exploitable concentration level for a deposit of copper is 0.4% based on average crustal abundance [1]. The estimated copper ore deposit in Nigeria is over ten million metric tons, and are distributed across the northern part of Nigeria in

Nasarawa, Bauchi, Plateau, Gombe and Zamfara States. This making it one of the most desired places in the world for buyers from outside the country to buy copper ore [2]. [3] reported that there are over 40 different types of economic minerals in Nigeria including copper found in about 500 locations, spread extensively across the country. Nevertheless, many of these occurrences can just be described as 'mineral showings'with no or little economic potential.

Copper as a base metal has many areas of applications in metallic forms and as well as in alloys form, in industries relating to mechanical, metallurgy, electrical and electronics, military, chemistry, paint and agriculture. The copper products demand globally is on the increase as a result to its growing applications in scientific and technological fields and as well as other innovative applications of the metal industry ([4];[5];[6]). The rising request for copper in the recent years was evident worldwide and the requisition for copper products can validly be due to stay high and keep increasing in the future years [7], This is because, the rising copper request has stimulated the unavoidable exploitation and application of oxide of copper ore. The effective use of oxides of copper from copper deposits is an essential subject of research [8]. The metals recovery from ores that are non-ferrous has been investigated on large-scale and the process determination relies upon both the localization and the ore composition ([9];[10];[11]). Oxides of copper, blended sulphide-oxide ores and copper sulphide ores with low grade, which for profitable cause, can never be improved by froth flotation technique,but can be treated by hydrometallurgical techniques [7].

Nevertheless, hydrometallurgy as a field of study, there have been notable transformation, growth and development for copper production from carbonate group ores of copper, like malachite and azurite ([12];[13]). The leaching media for the processing of copper usually of acids, alkaline, cyanides and inert reagents. Sulpheric acid is an inexpensive leaching agent for oxidized copper ores. [14] reported the origin and characteristics of Akiri copper ore deposit. The chemistry of the ores showed dominantly Cu and Fe with barium in places and the ores contain insignificant Pb and Zn which suggest the absence of the associated galena and sphalerite. The Akiri copper ore deposit hosts a high-grade copper mineral from which copper metal can be extracted. However, the ore is dominated by gangue minerals that need to be mechanically reduced for efficient copper recovery. The Nigerian copper ore has been reported to contain both chalcopyrite and pyrite while the content of pyrite is higher than that of chalcopyrite ([15] and [16]). The implication of these is that the grade of the ore needs to be improved berfore extraction.

Lately many experimental design methods mostly response surface methodology (RSM) is applied in the many engineering sectors for system parameters optimization. Complex variables process optimization in traditional method follows one-parameter at a time. Several tests are needed for normal methods, and these techniques do not describe the interaction effect. In order to estimate optimum level, additional data is required and take a longer period, which is undependable [17]. The purpose of the design of experiment (DOE) method was to know the combined effects among the factors, that will assist factors optimization in the experiment in order to produce the statistical models [18]. The optimization of leaching parameters for coal with hydrofluoric acid for ash reduction has been studied using response surface methodology [19]. From literature, it has been established that few studies have been conducted on statistical optimization of system condition for upgrading of copper and reduction of gangues minerals from copper ore by using hydrometallurgical method. Hence, this study aims to define the optimum leaching conditions for the extraction of copper and gangues reduction from Akiri hematite-dominated copper ore in sulpheric acid leaching with the aid of statistical optimization.

The interaction effects of leachant concentration, temperature of the reaction, and contact time on the dissolution process by applying response surface methodology with central composite design to optimized the variables was studied. Therefore, a design of experiment was applied to developed a statistical model in order to know the optimum dissolution conditions where copper recovery and iron reduction is highest to be obtained from the copper ore.

2.0 MATERIAL AND METHODS
2.1 Materials
Ore sample was obtained from Akiri copper mines in Akiri town, Awe local GovernmentArea of Nasarawa State, Nigeria. A representative sample from the bulk ore was crushed and ball milled and sieved with 250µm sieve to obtained 100% passing and the representative samples from the milled ore were used for both characterization and the leaching experiments. This particle size was used because it falls within the range of liberation sizes of the copper ore where copper particles are liberated.

2.2 Methods

The chemical and mineralogical characterizations were performed. These analyses of the test samples were conducted with   aid of X-Ray Fluorescence (XRF) spectrometer, X-ray Diffractometer (XRD) methods, Atomic Absorption Spectroscopy(AAS) and Ore Micrograph respectively. Chemical analysis of the copper ore was done using a high-performance sequential wavelength dispersive XRF spectrometer, Shimadzu EDXRF-702HS operated at 40kV and 18mA. Mineralogical analysis was performed using a Shimadu XRS2400H diffractometer with Cu anode, $\lambda 1_{CU}=1.541838$[A^{o}]. The ore micrographs analysis was conducted with Leica optical microscope equipped with high level resolution digital camera for both reflected and transmitted light [3] at the department of Materials Science and Engineering of Obafemi Awolowo University, Ile-Ife, Osun State. Both XRD and XRF analyses were conducted at Afe Babalola University, Ado Ekiti, Ekiti State Nigeria. AAS was conducted at Central Science Laboratory of Obafemi Awolowo University, Ile-Ife, Osun State. These analyses were conducted to find the classification of individual minerals, the extent of association with each other and mode of occurrence.

The leaching reagent was prepared with analytic grade H_2SO_4 (Probus R.A $\geq$ 98% purity) and deionized water. The experiments were performed in 50ml conical flasks which were heated in electric oven under temperature control for different conditions to establish the best conditions for the leaching process. The leaching tests in bench scale were performed by using 3^3 factorial design experiments using the test parameters in the Table 1. A sample (5g) was prepared for each leaching test. The leachant was prepared in-predetermined concentrations stated in the Table 1. Then, based on 1:5g/ml solid-to-liquid ratio, 25ml of solution was poured into the conical flask. Then, sample of the ore was added into the flask and the content of the flask was mixed by stirring for 2 minutes. The mixture was heated at the set temperature for the required time.

After each experiment, solution was allowed cooled and then filtered into 250ml conical flask. Then residual collected, cleansed towards neutrality in-deionized water, air dried for 24hrs, oven dried at about 105°C then weighed. The disparity in weight was noted for determining the fraction of the copper that had been dissolved. The solution of the experiment with the least weight was analyzed using AAS. The copper recovery into the copper loaded solution was estimated using the equation below:

$$Cu\ recovery = \frac{Cu\ leached\ per\ gram\ of\ copper\ ore}{Total\ Cu\ present\ per\ gram\ of\ raw\ copper\ ore}\ x\ 100\% \qquad (1)$$

Copper leached = (xxmg/L*dilution factor*volume of the filtrate per gram of ore) [20].

The test was repeated on a magnetic stirrer hotplate and an agitated reactor to examine the effects of agitation rate on the leaching rate. This was performed in a 300ml reactor prepared with mechanical stirrer with a digital controller unit, thermostat and a timer. The reaction medium temperature was set as required. The contents in the reactor were heated initially with mild stirring at stage of heating and on reaching the set temperature, and then the stirring continued until the desired reaction time was reached. After the experiment, the content in the reactor was filtered immediately and the amount of copper and iron in the filtrate was determined by AAS.

Table1: Sulphuric acid LeachingTest parameters

Label	Parameters	LEVELS		
		1	2	3
A	Concentration(M)	0.5	1.0	1.5
B	Temperature(ºC)	50	70	90
C	Time(min)	30	60	90

2.3 Design of Experiment

Parameters used for copper dissolution from the Akiri hematite-dominated copper ore by H_2SO_4 leaching were evaluated with central composite design, a typical RSM design. RSM technique is appropriate to fits a quadratic surface and as well optimize the variables of the system with a least number of tests and to examine the synergy among the variables [21].

RSM is a statistical technique that is used for creating the empirical model, optimizing and improving process variables and find the synergy affecting variables [21]. The methodology involves designing experiments, creating models; and analyzing data in order to know the most significant input variables, and to examine the range of values that will yield the maximum possible outcome for the output variables [22].

2.4 Statistical Analysis and Model Fitting

Analysis of variance is a statistical tool for graphical analysis of information obtained from the experiment to explain the interaction among the parameters and the yields to calculate the statistical variables of the process. The analytical software was used on data from the experiments to plot the response surfaces, the regression analysis, and contour plot at the optimal conditions. Statistical weight was scanned with the F-

test, polynomial model fitting accuracy was evaluated with the R^2 coefficient and important model terms were estimated P-value (Probability value) with confidence interval of 95%.

3.0 RESULTS AND DISCUSSION
3.1 Characterization and Sulpheric acid Leaching Mechanism of the Copper Ore

Figure 1 shows the ore micrographs of the copper ore sample. From the result, three mineral phases can be recognized under plane polarized light, the phases are chalcopyrite (brassyellow), hematite (reddishbrown) and silica mineral (whitish). This confirmed that the mineral of interest copper is actually present in the Akiri copper ore sample. Nevertheless, the sample was subjected to further analysis for confirmation as follows.

The XRD pattern shows that the ore contains chalcopyrite $(CuFeS_2)$(-222) as the main copper mineral with 2.24% in composition,while covellite (CuS)(212), and cuprite (Cu_2O)(622) are the associated copper minerals with 1.76 and 1.29% respectively as shown in Figure 2. The XRD analysis also revealed the gangue minerals present in the ore namely; quartz (002), mica (011), plagioclase (022), K-Fieldspar (412), chlorite (222) and calcite (141). Calcite $(CaCO_3)$ was present as a main acid consuming mineral. The XRD analysis indicated that the ore is made up of four major mineral groups that is sulphide, silicate, carbonates and oxide. This result is similar to the study reported by [23] that, Akiri copper ore contains chalcopyrite, siderite, azurite, quartz, hematite, goethite, pyrite and malachite from XRD analyses of different samples from the deposit, and interms of frequency of occurrence, $CuFeS_2$, $FeCO_3$ and SiO_2 are the most dominant minerals in the samples.
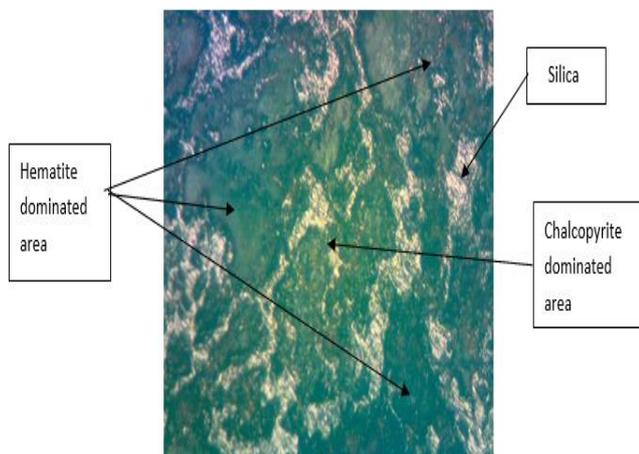

Figure 1: Micrograph of Head Sample under plane polarized light (x100)

The major oxides in the samples according to XRF analysis are hematite (Fe_2O_3), copper oxide (CuO) and quartz (Si_2O) as shown in Figure 3. The sulphur that is present means that the deposit is a sulphide ore deposit. A high content of Fe in combination with S and O is confirming that pyrite and hematite present. Deposits of chalcopyrite are mostly found with pyrite and hematite as major associated gangues. There are other trace elements,which constitute impurities in the ore Mn,Si,Ca,Ni etc. The AAS analysis result of the raw ore shows that, the ore contains 4.61% Cu and 22.48% Fe.
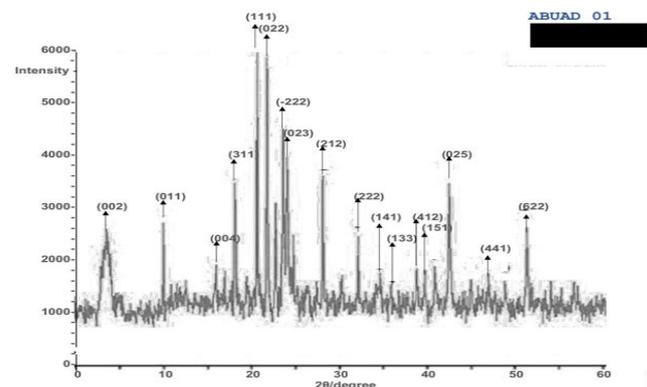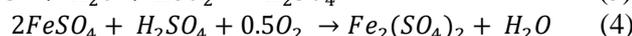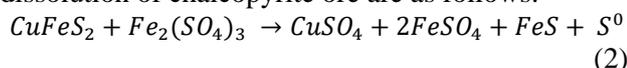

Figure 2: XRD pattern of the raw ore
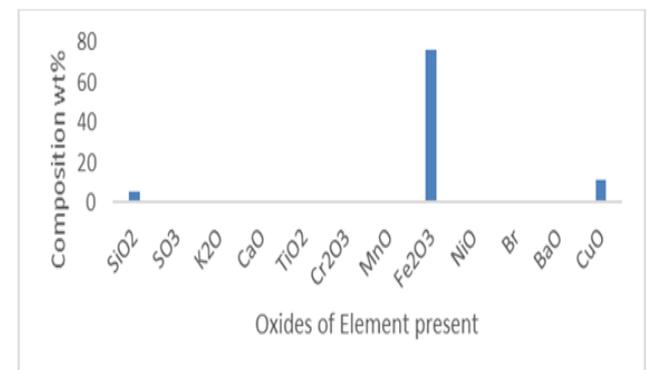
Figure 3: Chemical analysis of the Head Sample by XRF

The leaching behaviour of minerals is in response to temperature, concentration and contact time. The most used effective media with higher latent of extraction is sulpheric acid as compared with other substances that are used for leaching agents. The basis behind for greater degree of leaching may be as a result of the high attraction of hydrogen ion (H^+) towards the copper matrix minerals. Copper oxide ores contain Cu in the divalent state, for example, malachite, chrysocolla, tenorite and azurite and these are usually soluble in H_2SO_4 at 25ºC [24]. Hence, when sulphuric acid is applied as leaching reagent to dissolve Cu from the copper ore, elements apart from

copper in the matrix of the ore as well leached along with the copper in the course of dissolution process. The impurities inside the filtrate need to be separated by solvent extraction before to the copper electro-winning step to raise the process current efficiency as well as the final product quality [25].

The reaction that always takes place during H_2SO_4 dissolution of chalcopyrite ore are as follows:

$$CuFeS_2 + Fe_2(SO_4)_3 \rightarrow CuSO_4 + 2FeSO_4 + FeS + S^0 \tag{2}$$

$$S^0 + H_2O + 1.5O_2 \rightarrow H_2SO_4 \tag{3}$$

$$2FeSO_4 + H_2SO_4 + 0.5O_2 \rightarrow Fe_2(SO_4)_2 + H_2O \tag{4}$$

The significant economic factor in the dissolution of oxidized ore of copper is the cost of the acid. Limestone and dolomite, which are carbonate rocks that are present in the ores, caused consumption of more acid. Hydrometallurgical method has become the most applied technique for treatment of tailings and low-grade ores because of the small fund as against that of high cost of pyrometallurgical method that demand serious environmental controls [26].

3.2 Model Development

A statistical ''design-expert 6.0.6" software was used to examine the regression analysis of the data obtained from the experiments and to sketch the response surface plot. Statistical variables were calculated by applying analysis of variance. The sulpheric acid

dissolution study, the needed scope of the experiment and parameters coded level are given in Tables 2 and 3, which shows the experimental design together with the outcomes from the tests.

As the yield suggested by the software, the quadratic model was not named. The final empirical model interms of a coded factor for copper extraction (weight loss, P%) is shown in Equation 5:

$$Wt.Loss = +21.84 + 2.14A + 1.18B + 5.00C + 0.76A^2 + 1.41B^2 + 3.73AC - 0.013BC \tag{5}$$

Where the + signify the synergistic effects whereas - signify the antagonistic effects.

Each model coefficients with lack of fit test and test for significance of the regression model were performed in order to fit a good model. Generally, the significant parameters were positioned based on the Model F-value or Probability value (P-value) with confidence level of 95%. As shown in Table 4, the analysis of variance for the data produced by Equation 5 for the copper recovery from the copper ore. The bigger F-value and the smaller 'P' value (Prob.>F), show more significant of the corresponding coefficient [27]. The F-value of the model is 22.50 implies that the model is significant. Also, the model coefficient terms are significant only when the values of ''Prob.>F'' are > 0.05. In this work, A, C, C^2 and AC are the significant model terms.

Table 2: Experimental independent parameters and their coded levels for the central composite design

Independent variables	Symbol	Levels of coded variables		
		Low	**Medium**	**High**
		-1	0	+1
Temperature(ºC)	A	50	70	90
Time (min)	B	30	60	90
Concentration(M)	C	0.5	1.0	1.5

Table 3: Experimental factors in actual unit and experimental responses

| Std	Run	Block	Independent variables			Wt. LossP(%)
			A(degC)	**B(Min)**	**C(M)**	
6	1	1	90	30	1.50	37.82
2	2	1	90	30	0.50	19.42
5	3	1	50	30	1.50	24.68
13	4	1	70	60	0.50	21.20
11	5	1	70	30	1.00	22.40
9	6	1	50	60	1.00	21.98
14	7	1	70	60	1.50	28.37
12	8	1	70	90	1.00	23.72
10	9	1	90	60	1.00	22.84
3	10	1	50	90	0.50	24.38
1	11	1	50	30	0.50	21.62
4	12	1	90	90	0.50	21.96
8	13	1	90	90	1.50	39.10
16	14	1	70	60	1.00	21.50
15	15	1	70	60	1.00	22.94
7	16	1	50	90	1.50	27.82

Table 4: Analysis of variance (ANOVA) for response surface quadratic model for copper recovery in leaching of the crude copper ore

Source	Sum of Squares	Df	Mean Square	F value	Prob>F(value)	Remark

|						
Model	495.40	9	55.04	22.50	0.0006	Significant
A	45.97	1	45.97	18.79	0.0049	Significant
B	13.97	1	13.97	5.71	0.0541	
C	249.90	1	249.90	102.13	<0.0001	Significant
A^2	1.53	1	1.53	0.63	0.4590	
B^2	5.26	1	5.26	2.15	0.1931	
C^2	25.94	1	25.94	10.60	0.0173	Significant
AB	0.21	1	0.21	0.086	0.7788	
AC	111.15	1	111.15	45.43	0.0005	Significant
BC	1.250E-003	1	1.250E-003	5.109E-004	0.9827	
Residual	14.68	6	2.45			
LackofFit	13.64	5	2.73	2.63	0.4354	Insignificant
PureError	1.04	1	1.04			
CorTotal	510.08	15				

The ''Lack of fit F-value''of 2.63 means the Lack of Fit is significant comparative to the pure error. There is a 43.54% chance that a "Lack of Fit F-value" this large could take place due to noise. Non-significant lack of fit is good. Each parameter level was estimated based on initial swelling. The distance from the center to the axial points was ±1.6817 and it was estimated from $a=(2^k)^{0.25}$ where k denotes the number of parameters. Adequacy precision measured the signal to noise ratio, which contains both the average prediction error and predicted value at the design points. In this work, adequacy precision ratio is 15.735 and is good due to this ratio has been higher than 4. Therefore, the model developed can be applied to navigate the design space.
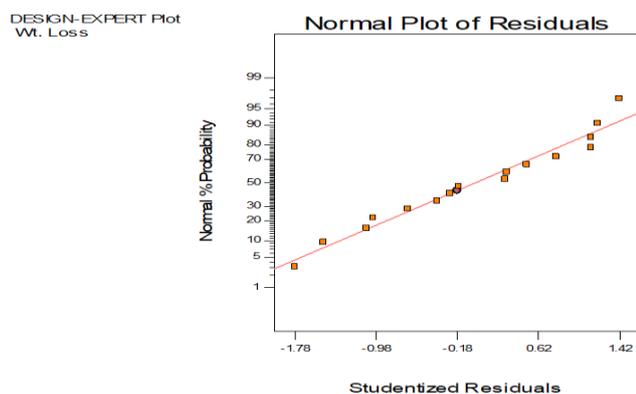

Figure 4: Studentized residuals and normal perce-ntage probability plot for weight loss in leaching the copper ore

The adequateness of the built model was the primary step to examine the test data analysis. The studentized residual and normal probability plot was illustrated in Figure 4 for copper extraction % from the copper ore. From Figure 4, it can be seen that there was neither apparent problem nor response transformation with normality. Figure 5 illustrates the plot of internally studentized residual versus predicted copper extraction %. This shows that the entire values of the random scatter plot, the variance of original observation and the response is stable, and this was a sign that, transformation of response parameters was

not needed. The actual and the predicted % for copper recovery was shown in Figure 6. 97% and 93% were the values for R^2 and adjusted R^2 (R_{adj}) found respectively.
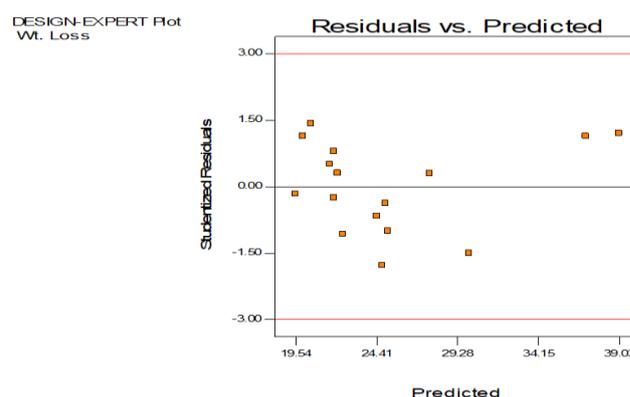
Figure 5: Predicted weight loss of the copper ore and studentized residuals plot
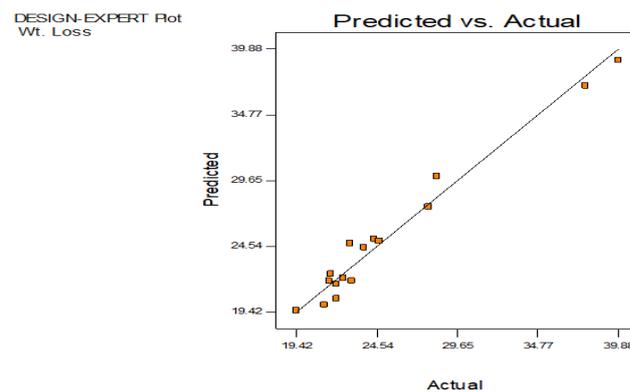

Figure 6: Actual and predicted plot of weight loss in leaching of the copper ore

R^2 value obtained explains upto what level good model can calculate the experimental data points and the value of the R_{adj} estimated the quantity of variation about mean spell out by the model. The value of predicted R^2 for Equation 5 was 0.7733, and relatively closes to the value of R^2. The data from the experiment for copper recovery % from the copper ore fitted well with the value predicted from the model as revealed. The model standard deviation is 1.56. The standard

deviation small value proves good model that gives close value between actual and predicted values for the responses. Table 6 illustrates the statistical parameters obtained from analysis of variance.

The pareto (Figure 7) was plotted using coefficients in Equation 5. Acid concentration (*C*) was major parameter that has impacts on the dissolution of copper from the ore. Since the coefficient for *C* in Equation 5 carries a + signs mean that the copper recovery increases as the concentration increases. Also, the most significant interaction term is AC. This means that the copper extraction is favourably influenced by combined variation in both temperature and concentration. From Equation 5 and Figure 7, increasing the entire variables can have positive effects on the extraction of copper.
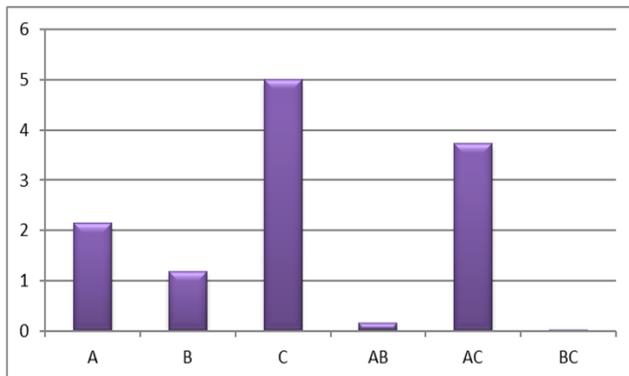
Figure 7: Pareto chart for copper extraction

Similarly, copper extraction is adversely affected by combined variation in both temperature and contact time, although these two parameters individually increase copper recovery, the term AB negative sign shows that more iron will be released as gangue as contact time is lengthen, depending on the temperature of the sample. Nevertheless, in the equation, the effect of BC terms interaction is minimal. The quadratic terms in the equation indicate the presence of curvatures. The + signs in Equation 5 shows that the quadratic curves for A^2, B^2 and C^2 are convex. This shows that copper extraction will be raising as the respective parameters are increased until maximum extraction is achieved.

Table 5: Staistical variables obtained from analysis of variance (ANOVA) for the models for weight loss percentage in leaching the crude ore

Insignificant factors excluded	Weight loss P(%)
Standarddeviation(S.D)	1.56
Mean	25.16
Coefficientofvariance(C.V)%	6.22
Adeqprecision	15.762
R^2	0.9712
AdjustedR^2	0.9280
PredictedR^2	0.7733

PRESS	115.63

Coefficients of the quadratic term's magnitude indicates the decent of the curvature. Hence, it from Table 5 and Equation 5 that C^2 is steeper than A^2 and B^2. The correlation coefficient of R² of 0.9712 value shows that only 2.88% of the entire variation could not be explained by the empirical model.
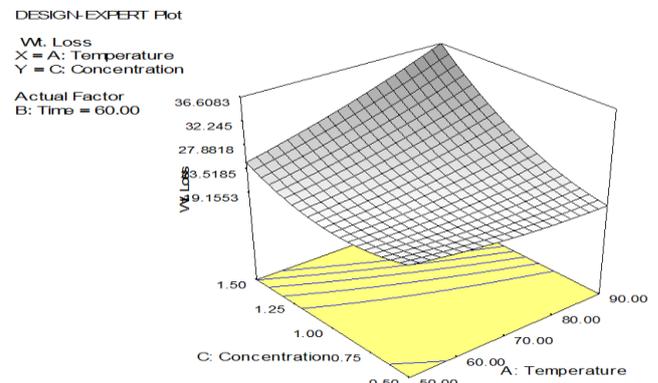
Figure 8: Combined effect of temperature and concentration on the copper recovery from of the copper ore

3.3 Combination Effect of Concentration, Temperature and Time on Copper Extraction

RSM was applied to examine the interaction and each factor effect of the 3-factor on copper extraction from the copper ore. According to analysis of variance, the results that were obtained, the effects of test factors on copper extraction, corresponding 3-D response surface plots were illustrated in Figures 8–10 and the response model was illustrated in Equation 5. The factors, temperature, time and concentration have significant effects on the copper extraction from the copper ore. By the application of RSM technique, it was discovered in Table 4 that the leaching factors affects the copper extraction (P) from the ore.

From Table 4, sulphuric acid concentration (C) was the factor that has major impact on the dissolution of copper from the copper ore compared to other factors, which may be as a result of the large F-value of 102.13 for sulphuric acid. Nevertheless, both temperature and contact time have less significant impacts for copper extraction from the ore. Though, the combined interaction between the parameters has no impacts on the copper extraction; only the concentration was discovered to be more effective on copper extraction. The dissolution concentration and temperature quadratic function demonstrated nearly alike impacts on the response, which was the small level significance in comparison to the quadratic function of time.

Response surface methodology was applied to examine the 3-D response plots that were produced from the impacts of the 3 parameters on copper extraction from the copper ore by sulphuric acid leaching. The ANOVA for a response surface quadratic model for copper extraction from the copper ore is shown in Table 5. According to RSM, the dissolution factors were involved more or less in increasing the efficacy of copper extraction (P).
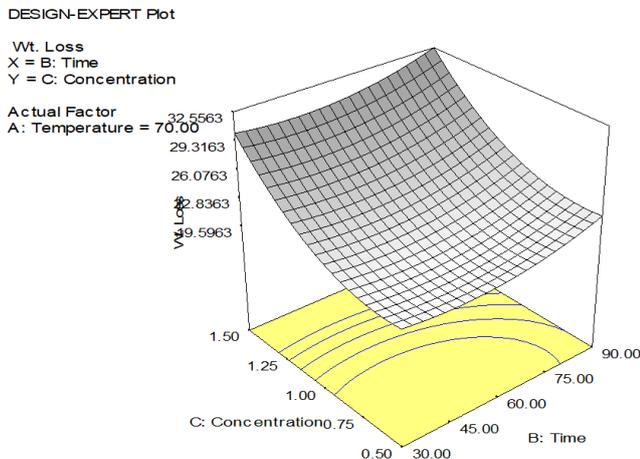

Figure 9: Interaction effect of time and concentration on the leaching of the copper ore
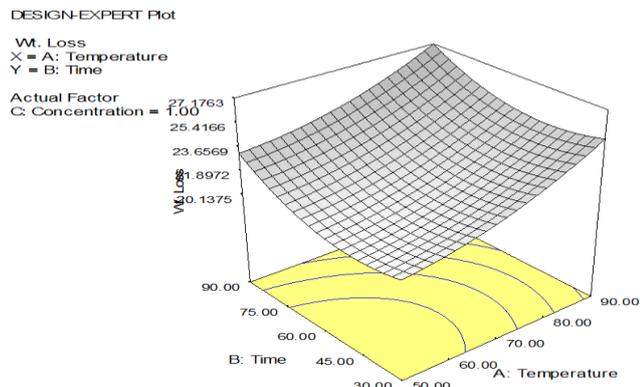

Figure 10: Interaction effect of time and temperature on the leaching of the copper ore

Figures 8 - 10 demonstrate the synergy among the factors in 3-D response surface plots. The scale values of the axis in the Figures 8-10 are real values. Figure 8 illustrates the interaction effect of temperature (A) and concentration (C) on copper extraction at constant contact time (60min). Copper dissolution increased due to the effects of concentration and temperature at constant contact time. The highest copper recovery of 92% was obtained. The synergy between the temperature and concentration was the most efficient factor for the copper extraction in the leaching process. Figure 8 and 10, showed that, the copper recovery increases with rise in temperature. Because, rise in temperature leads to the increase in

reaction rate and as well as the reaction activation energy. When the leaching time is constant, the increase of leachant concentration from 0.5 to 1.5M and temperature rises from 50°C to studied range, this made the copper recovery to increase from 61 to 90.38%. The case for raised copper extraction discover from the analysis of variance table was the larger 'F'value of temperature and acid concentration, which showed the larger extent of dissolution effect [28]. The interaction effect of time (B) and concentration (C) on copper extraction from the copper ore at stable temperature (70°C) was illustrated in Figure 9. The copper recovery of 81.11% was obtained by the effect of time and concentration at constant temperature.
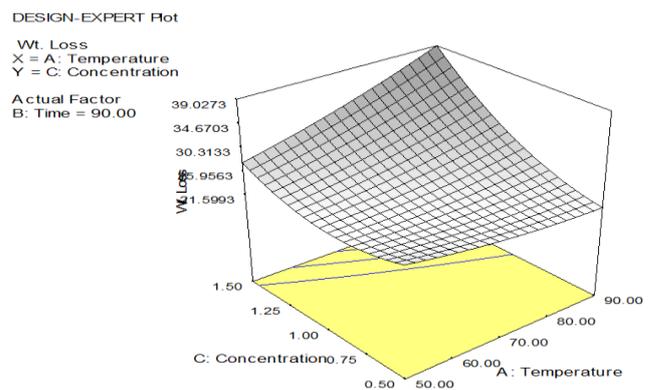

Figure 11: Optimum region on the concentration and temperature for maximization of copper recovery from the copper ore
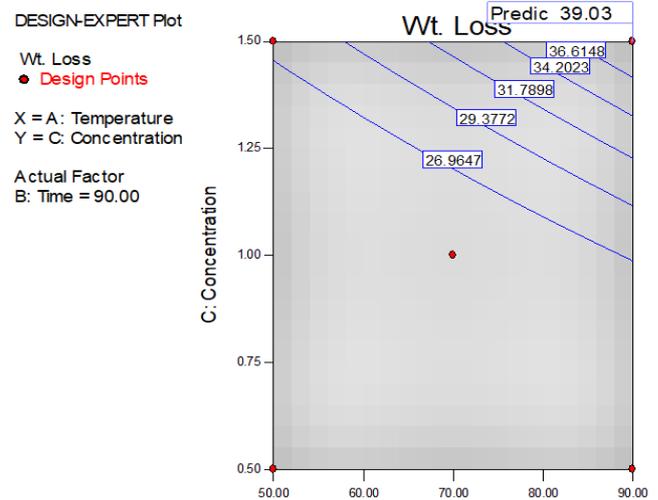

Figure 12: Contour plot of temperature and concentration on the leaching of the copper ore at optimum conditions

Also, the interaction effect of contact time (B) and temperature (A) on copper extraction at 1M concentration and the related 3-D response surface were showed in Figure 10. The results show that, temperature and contact time has fewer effects on

copper extraction from the copper ore because of the small value of F statistics. Copper recovery of 75.03% was obtained by the effect of temperature and contact time at 1M concentration.

3.4 Response Surface Modelling Optimization
To estimate the leaching optimum conditions where the highest copper recovery can be obtained from the copper ore is the most important aspect of this work. The leaching parameters optimization was examined in a numerical optimization technique. Figure 11 and 12 illustrate the response surface and contour plot at best leaching conditions for highest copper recovery respectively. Therefore, 1.5M acid concentration;

90°C temperature and 90min contact time were the optimum process conditions for the extraction of copper in the leaching of the copper ore. The experimental and predicted values obtained for the weight loss from the copper ore were 39.10% (92% copper recovery) and 39.03% (91.8% copper recovery) at the optimum leaching conditions, respectively. A contrast betwixt the predicted and experimental results shows <0.08% error. Therefore, the results show that the developed model can correctly predict the copper recovery. Optimization conditions information is illustrated in Table 6.

Table 6: Optimal processing conditions and model validation of percentage weight loss from numerical optimization

Parameters	TemperatureA(°C)	TimeB(Min)	ConcentrationC(M)	Weight LossP(%)	
				Predicted	Exptal
Optimum conditions	90.00	90.00	1.5	39.03	39.10

Table 7: Result of leaching crude ore with Stirring

Operation	%Cu	%Fe	R_{Cu}(%)	R_{Fe}(%)
Feed(-250μm)	4.68	11.33	***	***
LeachingwithoutStirring	6.64	2.31	92.00	57.30
LeachingwithStirring	7.84	5.47	83.51	24.07

3.5 Effects of Stirring Rate on the Extraction of Copper
To study the impact of rate of stirring on the extraction of copper, experiment was conducted using the optimum conditions (Acid concentration, 1.5M, Temperature, 90°C and Contact time, 90minutes) and with optimum stirring rate of 400rpm [29]. In Table 7, the copper extraction efficiency increased with stirring rate. The copper grade increased from 6.64% Cu (leaching without stirring) to 7.84% Cu (leaching with stirring). But the copper recovery decreased from 92% (without stirring) to 83.51% (with stirring). The stirring action also affected the iron contents by increasing it from 2.31% to 5.47% Fe. It shows that, more iron was dissolved when the solution was stirred. According to hydrometallurgy kinetics principle ([30];[4]), reported that the rate of stirring has a strong impact on the leaching reaction efficiency (liquid-solid reaction) when the control step is fluid film diffusion, which can generally improve the dissolution efficiency greater than 40%. Although, the effect of stirring on the copper extraction efficiency is unnoticeable, shows that the rate-controlling step is not diffusion through the fluid film [31].

3.6 Validity of Shrinking Core Model (SCM) Test
The optimum leaching conditions found was applied to determine leaching kinetics of copper ore minerals. When liquid film diffusion (LFD) model controlled

the leaching rate of the particle, then the integrated rate equation of this step is Equation 6 [31].

$$x = kt \tag{6}$$

When diffusion through a product layer (DLP) model controlled the leaching rate, the integrated rate equation of this step is Equation 7 [32].

$$1 - 3(1-x)^{\frac{2}{3}} + 2(1-x) = kt \tag{7}$$

The activation energies estimated for leaching of copper in sulphuric acid leachant according to LFD and DLP are 13.2 and 25.67kJ/mol^{-1}, respectively. The small values show that the dissolution rate is diffusion process controlled ([33];[34];[35]).

Hence, the activation energy value obtained in this work affirms that the leaching process is diffusion controlled through the product layer and liquid film controlled. Hence, the mathematical expression involving the factors in the experiment to represent the kinetics of the dissolution process can be written as follows:

$$x + 1 - 3(1-x)^{\frac{2}{3}} + 2(1-x) = 0.90944 \times$$
$$e^{\frac{-25.67 \times 10^3}{8.314 \times T}} \times t \tag{8}$$

4.0 CONCLUSION
A simplistic single-step technique was used to activate sulpheric acid dissolution into hematite-dominated copper ore obtained from Akiri copper

mines. At optimized dissolution conditions, the level of copper extraction was maximized to obtain high grade copper by the reagent dissolution. The victorious activation of H_2SO_4 leaching on the copper ore shows that the copper can be beneficiated by hydrometallurgy to increase the copper grade from 4.68% to 6.64% at high copper recovery of 92% and reduced iron contents from 11.33% to 2.31%.

The maximal copper extraction from the copper ore at optimum dissolution conditions by using central composite design together with response surface methodology was examined. The optimization of variables and the regression analysis were estimated with the aid of design expert software to predicts the yield in the investigative regions. Regression analysis, statistical significance and response surface study was conducted using the values obtained from tests at factor working conditions. The dissolution test parameters were correlated to the responses by the model developed. Response surfaces were derived based on the models. The acid concentration used was the most significant effect on copper extraction from the copper ore to produce nearly pure copper metal with reduced iron and other gangues minerals. The dissolution system optimization was done and values obtained from experiments conducted for the copper extraction and obtained values predicted by the models were nearly closer. The optimal working conditions for maximizing the copper extraction were got at 90°C temperature, 1.5M concentration and 90min contact time. Then, 0.08% was obtained as the absolute error. The acid wastes resulting from the leaching processes was treated by adding proper proportion of caustic soda and soda ash to ensure safe and clean environment.

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