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Optimization of Leaching Parameters and Kinetic Modelling for Leaching of Zinc from Zarara Hill Sulphide Ore in Different Acid Solutions

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Abstract

This research focused on optimization and kinetics of zinc dissolution from Zarara Hill sulphide ore using HNO_3, HCl and H_2SO_4. A polymetallic sulphide ore collected from Zarara hill, Kaduna State, Nigeria, was used for this study. Elemental analysis of the powdered sulphide ore using ICP-OES indicate that the primary components found are Fe (6.8440%), Zn (2.7477 %), Cu (2.6387%), Pb (2.5130%) and S (3.3017%) while the minor elements include Co (0.002454%), Ni (0.00134%), Mo (0.08337%), and Cd (0.00216%). Response Surface Methodology (RSM) was used to model, optimize and evaluate the effects and interactions of the influential specifications on the recovery of zinc using HNO_3, HCl and H_2SO_4. From the Design-Expert output, the quadratic model was not aliased. A regression model significant test and an individual model coefficient with lack of fit test were conducted in order to fit a good model. With a 95% confidence level, the significant components were sorted using the P-value (probability value). The smaller 'P' values from the ANOVA generated for both HNO_3, HCl and H_2SO_4 show more significant of the corresponding coefficients. Every R-squared value is near to one and each of the adjusted R-squared values is near to its corresponding R-square value. These confirm the adequacy of the developed model. Dissolution kinetics, studies show that for zinc dissolution, the order of reaction with regard to H^+ ion concentration using HCl, H_2SO_4 and HNO_3 were 0.53, 0.58 and 0.54 respectively. And the activation energy was calculated from Arrhenius plot and were found to be 24.3, 22.8 and 21.2 kJmol^{-1} for HCl, H_2SO_4 and HNO_3 dissolutions respectively.

1. Introduction

Complex sulphide ore is a common mineral and is found in almost all sulphide deposits and is often disseminated through igneous rock [1]. Processes available for the treatment of complicated sulphide ores involve sulphide oxidation to create water soluble sulphates or oxides. Procedures for Sulphur oxidation include roasting, pressure oxidation, chemical oxidation and bioleaching. The old processes are difficult and expensive. Hydrometallurgical method has remained the most prominent method in extracting many metals from their ores. The procedure

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usually entails winning metals from their ores by leaching into a suitable solution from which the metal can be easily collected by any other means such as solvent extraction, precipitation, ion exchange, and electro-winning. Various works were done on copper and lead leaching using different leaching solutions and with various leaching conditions by Ajibola & Jimoh [2]. As a result, emphasis has been focused to the use of readily available and inexpensive reagents like sulphuric acid (HCl), hydrochloric acid (H_2SO_4) and nitric acid (HNO_3) for base metal recovery and dissolution kinetics optimization.

Rasoul *et al.* [3] applied response surface methodology (RSM) to optimize the recovery of metal content (i.e., Cu, Fe, Zn, Pb, Ni, Sn, and Al) embedded in waste printed circuit boards (WPCBs) using a leaching agent without any additive or oxidative agent. Effective parameters and their levels, including leaching time (20–60 min), temperature (25–45°C), solid to liquid (S/L) ratio (1/8–1/20 g/ml), and acid molarity (1–2.7 M), were optimized. General optimum conditions for nine responses were introduced with the desirability of ≈ 85%. Another study aimed to develop the efficient technique for the production of clean coal by optimizing the operating parameters with the help of response surface methodology [4]. The effect of three independent variables such as hydrofluoric acid (HF) concentration (10–20% by volume), temperature (60–100°C), and time (90–180 min), for ash reduction from the low-grade coal was investigated. A quadratic model was proposed to correlate the independent variables for maximum ash reduction at the optimum process condition by using central composite design (CCD) method. It is evident from the studies that HF concentration was the most effective parameter for ash reduction at the expense of time and temperature.

Ayodele *et al.* [5] carried out the optimization of recovery of copper from Akiri hematite-dominated copper ore using hydrometallurgy. Effect of three independent factors like concentration, temperature and contact time for copper extraction from the hematite-dominated copper ore was studied. At optimized dissolution conditions, the level of copper extraction was maximized to obtain high grade copper by the reagent dissolution. The successful 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 activation energies for the copper dissolution were estimated as 13.20 kJ/mol and 22.67 kJ/mol for liquid film diffusion and diffusion product layer respectively, the values indicate that the leaching rate is controlled by diffusion process.

With the gradual depletion of rich ore deposits, it is becoming increasingly difficult in many situations to apply the conventional pyrometallurgical methods for metal extraction. So, hydrometallurgical processes are suited for lean and complex ores. If there are too much gangues in an ore, then, processing this is a problem, thus the ore processing can be carried out via hydrometallurgy. Zinc metal which is primarily used to galvanize iron and steel against corrosion as well as to produce brass and alloys for die-casting is present in significant amount in the sulphide ore. When exposed to the atmosphere, Zinc creates an impermeable oxide coating on the surface making the metal more resistant to ordinary atmosphere than iron and corroding at a far slower rate.

Therefore, this research will focus on optimization and kinetics of zinc dissolution from Zarara Hill sulphide ore using HNO_3, HCl and H_2SO_4.

2. Materials and Methods

2.1 Materials

A polymetallic sulphide ore collected from Zarara hill, Kaduna State, Nigeria, in March 2019 was used for this study. The ore samples were crushed, ground and sieved to a size of 0.1 mm which is fine enough to separate valuable materials using crusher, ball mill and automatic sieve shaker respectively. After pretreatment and beneficiation of the ore, it was then roasted at 1000°C for 2 hours in a suitable environment using a muffle furnace, roasting convert the metal sulphides to metal oxides which will be more amenable to leaching agents.

2.2 Design of Experiment

The experiments were design using Design Expert (Stat-Ease, Inc., Minneapolis, USA). Considering the literature and the preliminary experiments, effects of four parameters including acid concentration (HCl, 0.5M to 9M, HNO_3, 0.5M to 9M and H_2SO_4, 0.5M to 12M), leaching time (5–120 min), temperature (28–80°C), and stirring speed (100–720 rpm) on the leaching recovery of the metals from the ore were investigated using Central Composite Design (CCD) Response Surface Methodology (RSM) [3]. Table 1 shows the factors and their respective levels.

The response is Zn fraction. After each run, the mixture was filtered and diluted to 25 cm^3 and examined for Zn using Atomic Absorption Spectrometry (AAS) where the concentration in mg/L was then converted to mg/kg (Equation 1).

$$Recovery\left(\frac{mg}{kg}\,or\,ppm\right)=\frac{Conc.of\ metal\left(\frac{mg}{L}\right)X\,Vol.of\ sample\ (L)}{Sample\ weight\ (kg)} \tag{1}$$

Table 1 *Factors and their respective levels*

Factors	Low level	High level
Acid concentration (M)	0.5	12
Leaching time (mins)	5	120
Temperature (°C)	28	80
Stirring speed (rpm)	100	720

The fraction of each metal leached, was calculated (Equation 2) and used as the response variable.

$$Fraction\ of\ metal = \frac{metal\ extracted\ by\ leaching\ solution}{total\ amount\ of\ the\ metal\ in\ the\ ore} \quad (2)$$

From the output that the Design-Expert software program suggest, the quadratic model was not aliased. To ascertain statistical significance, analysis of variance (ANOVA) was employed. The precision of the fitted polynomial model was assessed using the coefficient of R^2. With a 95% confidence level ($\alpha = 0.05$), the significant model terms were evaluated using the P-value (probability value).

2.3 Leaching of Ore by Agitation Process

A 250 cm^3 beaker was used as the reactor for the leaching tests, and it was heated on a hot plate with a thermometer and a digitally controlled magnetic stirrer. The beaker with (say 100 mL) of leachant was placed on a hot plate. The ground ore sample was added to the beaker once the solution had achieved the appropriate temperature, and the contents were swirled at a determined pace. The solution mixture for each leaching experiment was made by dissolving 10 g/L of the roasted ore in the different molar concentration of HCl (0.5M to 9M), HNO_3 (0.5M to 9M) and H_2SO_4 (0.5M to 12M) at 28°C to 80°C [6-8].

In order to determine the best leachant, the amount of Zn dissolved was determined after 5, 10, 30, 60, and 120 minutes contact time and stirring speed of 100 to 720 revolution per minute (rpm) regarding the various concentrations of HCl, HNO_3 and H_2SO_4 acids, using Atomic Absorption Spectroscopy (AAS). Using this process, the impact of acid concentration, leaching period, temperature and stirring speed on zinc dissolution was examined.

The determination of the concentration of leached zinc was done by accurately withdrawing 5 cm^3 of the liquor and then filtered using a Whattman No. 42 filter paper. The filtrate was diluted to 25 cm^3 and then analysed. The percentage of zinc leached in each experimental set up were calculated from Equation 3.

$$R = \frac{M_1}{M_o} \times 100 \quad (3)$$

Where R is the percentage recovery of each of the metal M_0 is the weight of zinc in the ore sample from ICP-OES result and M_1 is the mass of each of the zinc leached [9].

The concentration of each of the leachants that resulted in the maximum zinc recovery was employed to optimize other parameters such as contact time, stirring speed and temperature. The Arrhenius plot (A plot of ln k versus 1/T (K^{-1})) was used to estimate the activation energy.

3. Results and Discussion

3.1 Ore Analysis

Elemental analysis of the powdered sulphide ore using ICP-OES are summarized in Table 2, which indicate that the primary elements found are Fe, Zn, Cu, Pb and S while the minor elements include Co, Ni, Mo, and Cd. The photomicrograph images of the ore (Figure 1) show further that the ore predominant minerals are quartz (SiO_2), Pyrite (FeS), Chalcopyrite (CuFeS), Sphalerite (ZnS), Galena (PbS).

Table 2 *Elemental composition of the sulphide ore*

	Fe	Cu	Pb	Zn	Co	Ni	Mo	Cd	S
Metal Conc. (mg/kg) ±0.3	68440	26387	25130	27477	24.54	13.4	833.7	21.6	33017

The mineralogical analysis revealed that the ore comprises sulphides in form of Pyrite (FeS_2), Chalcopyrite ($CuFeS_2$), Sphalerite (ZnS), Galena (PbS), Covellite (CuS) with magnetite (Fe_2O_4) and hematite (Fe_2O_3) in decreasing order of abundance, these ore minerals are accompanied by silicate gangue of quartz and feldspar with fluorides such as fluorite (CaF_2) [10].

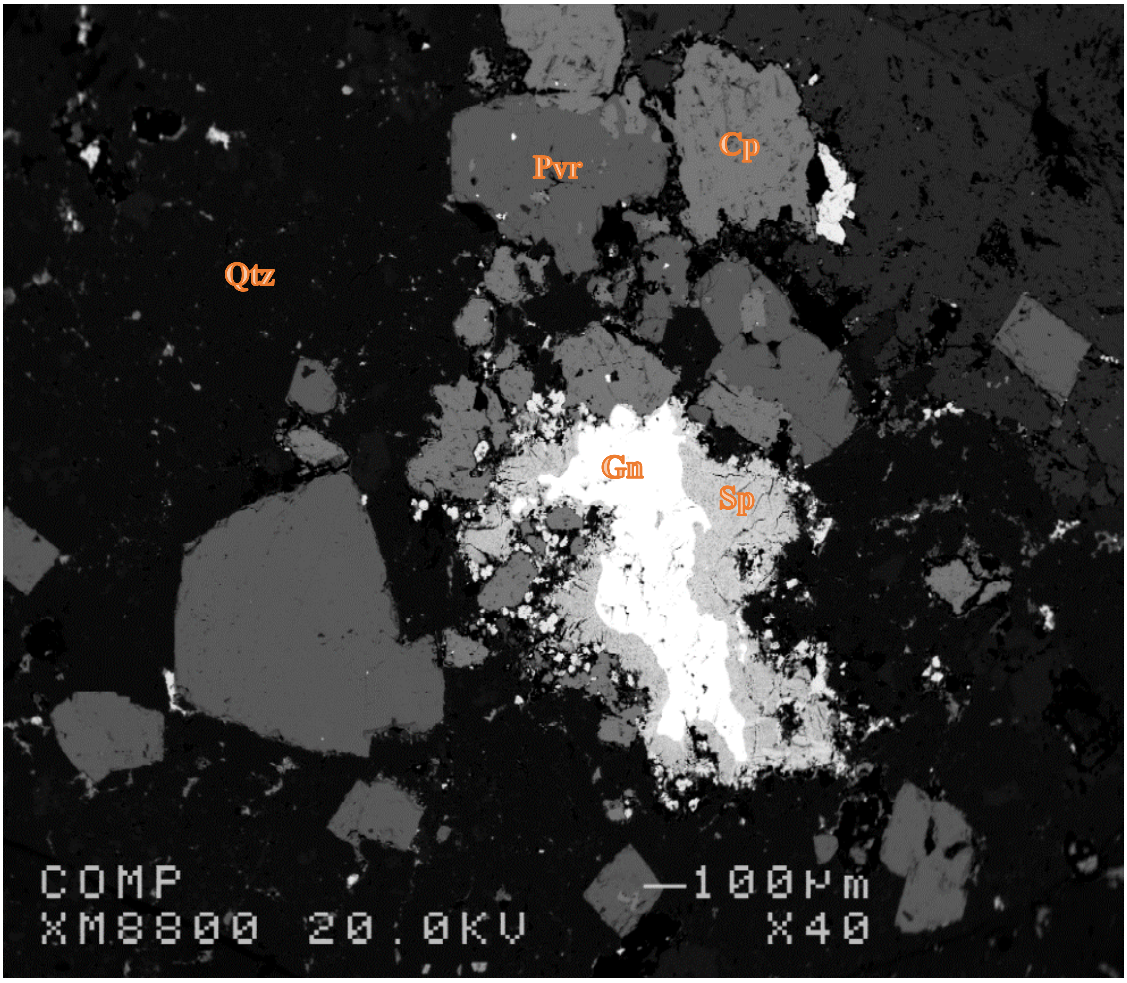

Fig. 1 *Photomicrograph of the raw Zarara Hill sulphide ore (Sp = Sphalerite, Pyr = Pyrite, Cpy = Chalcopyrite, Gn = Galena, and Qtz = Quartz)*

3.2 Leaching of Zn from Zarara Hill Sulphide Ore Using Surface Response Method

Surface Response Method results for leaching of zinc using HCl, H_2SO_4 and HNO_3 are provided in Table 3, 4 and 5 respectively. After modelling and proposing a fourth-degree polynomial model for each response, the adequacy of the proposed models was confirmed using ANOVA as presented in Table 6 for HCl, H_2SO_4 and HNO_3.

Table 3 *Experimental factors for HCl, actual units and experimental response*

Name	Units	Type	Std. dev.	Low	High
HCl Conc.	mol/dm^3	Factor	0	0.5	9
Time	mins	Factor	0	5	120
Temperature	°C	Factor	0	28	80
Stirring rate	rpm	Factor	0	100	720
Zinc	Fraction	Response	0.0233821	0.27	0.675

Table 4 *Experimental factors for H_2SO_4, actual units and experimental response*

Name	Units	Type	Std. dev.	Low	High
H_2SO_4	mol/dm^3	Factor	0	0.5	12
Time	mins	Factor	0	5	120
Temperature	°C	Factor	0	28	80
Stirring rate	rpm	Factor	0	100	720
Zinc	%	Response	0.0142526	0.136	0.475

Table 5 *Experimental factors for HNO_3, actual units and experimental response*

Name	Units	Type	Std. dev.	Low	High
HNO_3	mol/dm^3	Factor	0	0.5	9
Time	mins	Factor	0	5	120
Temperature	°C	Factor	0	28	80
Stirring rate	rpm	Factor	0	100	720
Zinc	Fraction	Response	0.0407948	0.190	0.800

Table 6 *The R-squares and adequate precision values of the experimental responses presented in Table 4, 5 and 6 for HCl, H_2SO_4 and HNO_3*

Response	Std. dev.	Mean	C. V. %	R-squared	Adj. R-squared	Adeq. precision	P-value
HCl	0.0234	0.4536	5.16	0.9858	0.9527	20.9878	0.0002
H_2SO_4	0.0143	0.3660	3.89	0.9943	0.9809	28.2044	<0.0001
HNO_3	0.0408	0.4386	9.30	0.9538	0.9077	21.4563	<0.0001

Std. dev. = Standard deviation, C.V = Coefficient of variance, Adj R-squared = Adjusted R-squared

From the output that the Design-Expert software program suggest, the quadratic model was not aliased for both HCl, H_2SO_4 and HNO_3. A regression model significant test and an individual model coefficient with lack of fit test were conducted in order to fit a good model. With a 95% confidence level, the significant components were sorted using the P-value (probability value). Table 6 shows the ANOVA (p-value) for the data generated. The smaller 'P' value for both HCl (0.0002), H_2SO_4 (<0.0001) and HNO_3 (<0.0001) show more significant of the corresponding coefficients. It can also be observed that every R-squared value is near to one and each of the adjusted R-squared values is near to its corresponding R-square value. These confirm the adequacy of the developed model. The more closely the R-square and adjusted R-square values in this statistical design resemble 1 and one another the more accurate the model is [11].

"Adequate precision" is the next criterion, which calculates the signal to noise ratio. Values higher than 4 indicate an adequate signal [3]. Table 6, indicates that the adequate precision for HCl as leachant is 20.9878. These values are much higher than four (4), which demonstrate the model's sufficiency. Also, from Table 6, the adequate precision values when H_2SO_4 was used as leachant is 28.2044 which are also far higher than four, confirming the accuracy of the model. The adequate precision values for the HNO_3 experimental responses are 21.4563.

In a similar study by Ayodele *et al.* [5], statistical variables from ANOVA having R^2 value of 0.9712 and adjusted R^2 values of 0.9280 confirms the accuracy of the model. Also, the adequate precision ratio of 15.735 is good due to this ratio being higher than 4. Therefore, the model developed can be applied to navigate the design space [5].

3.3 Response Surface Modelling Optimisation

The most crucial part of this work is estimating the ideal leaching conditions that will yield the maximum zinc recovery from the sulphide ore using HCl, H_2SO_4 and HNO_3. Using a numerical optimization technique, the leaching parameters optimization was investigated. Table 7 provides an illustration of the optimization conditions information.

Table 7 *Optimization conditions information and model validation of zinc fraction using numerical optimization technique*

Acid used	Acid concentration (mol/dm3)	Time (mins)	Temperature (°C)	Stirring speed (rpm)	Zinc fraction	
					Predicted	Experimental
HCl	8.98	112.4	79.2	471.25	0.703	0.675
H_2SO_4	8.00	111.1	52.9	386.26	0.480	0.475
HNO_3	8.99	108.4	79.9	100.05	0.801	0.800

Mins = minutes, °C = degree centigrade and rpm = revolution per minute

The predicted and experimental values obtained for the fraction of zinc leached from the sulphide ore using HCl were 0.705 and 0.675 respectively, 0.479 and 0.475 respectively for H_2SO_4 while the predicted and experimental values obtained for the fraction of zinc leached from the sulphide ore using HNO_3 was 0.808 and 0.800 at their various ideal leaching conditions (Table 7). The difference between the experimental and expected values is less than 1% for H_2SO_4 and HNO_3 and less than 5% for HCl. Consequently, the findings demonstrate that the created model is capable of accurately forecasting the recovery of zinc.

3.4 Dissolution Kinetics of the Metals from the Ore

Two shrinking core models were tested for zinc dissolution in HCl, H_2SO_4 and HNO_3 media. Some researchers have previously used the kinetic model [6,12-15]. The kinetic models are:

$$1-(1-X)^{1/3} = K_2 t \quad (4)$$

$$\left(1-\frac{2}{3}X\right)-(1-X)^{2/3} = K_3 t \quad (5)$$

All the studied data were found to best fit Equation 5 for diffusion control model with correlation coefficient (R^2) for both HCl, H_2SO_4 and HNO_3 dissolution being above 0.97. Therefore, the relation: $1 - (2/3)X - (1 - X)^{2/3} = K_3t$, gave a correlation coefficient ranges of 0.9983 – 0.9999, 0.9974 – 0.9998 and 0.9982 - 1 with straight line passing through the origin for zinc dissolutions in HCl, H_2SO_4 and HNO_3 respectively. Therefore, the dissolution kinetics studies shows that the order of reaction with regard to H^+ ion concentration using HCl, H_2SO_4 and HNO_3 is 0.53, 0.58 and 0.54 respectively. And the activation energy was calculated from Arrhenius plot and were found to be 24.3, 22.8 and 21.2 $kJmol^{-1}$ for HCl, H_2SO_4 and HNO_3 dissolution respectively.

The activation energy of zinc leaching using HCl (24.3 kJ/mol), H_2SO_4 (22.8 kJ/mol) and HNO_3 (21.2 kJ/mol) from the polymetallic sulphide ore containing sphalerite (ZnS) are all below 40 kJ/mol. According to the literature, chemical reaction controlled processes are highly reliant on the reaction temperature, whereas diffusion controlled processes are only marginally reliant. Diffusion controlled processes often have activation energies around 40 kJ/mol, whereas chemically controlled processes typically have activation energies above 40 kJ/mol [16].

Ayodele *et al.* [5] also reported that the activation energies estimated for leaching of copper in sulphuric acid leachant according to liquid film diffusion and diffusion through a product layer are 13.2 and 25.67 kJ/mol, respectively. The small values show that the dissolution rate is diffusion process controlled. Another experiment on the leaching of a Nigerian chalcopyrite ore by nitric acid for possible production of copper nitrate was examined by Baba *et al.* [17], kinetic data analysis showed that the dissolution mechanism followed diffusion as the rate controlling step. The reaction order was calculated to be 0.51 while the activation energy was deduced to be 29.99 kJ/mol.

4. Conclusion

The elemental composition of the sulphide ore showed that Fe (68440 mg/kg), Zn (27477 mg/kg), Cu (26387 mg/kg), Pb (25130 mg/kg) and S (33017 mg/kg) are the primary elements found. Other elements found in low to trace levels include Co, Ni, Mo, Cd. Photomicrography was used to determine its mineralogical composition and revealed that the ore predominant minerals in the polished sections are quartz (SiO_2), Pyrite (FeS), Chalcopyrite (CuFeS), Sphalerite (ZnS), Galena (PbS) with Covellite and Iron oxides in low amount.

From the output that the Design-Expert software program suggest, the quadratic model was not aliased. A regression model significant test and an individual model coefficient with lack of fit test gives a smaller 'P' value from the ANOVA generated for both HCl, H_2SO_4 and HNO_3 and shows more significant of the corresponding coefficients. It can also be observed that every R-squared value is near to one and each of the adjusted R-squared values is near to its corresponding R-square value. These confirm the adequacy of the developed model. The more closely the R-square and adjusted R-square values in this statistical design resemble 1 and one another the more accurate the model is.

Dissolution kinetics studies was carried out and found that the reaction rate is controlled by diffusion of zinc through the porous product layer. In light of this, the order of reaction with regard to H^+ ion concentration using HCl, H_2SO_4 and HNO_3 were 0.53, 0.58 and 0.54 respectively. And the activation energy was calculated from Arrhenius plot and were found to be 24.3, 22.8 and 21.2 kJ/mol for HCl, H_2SO_4 and HNO_3 dissolutions respectively. These values support the proposed diffusion controlled mechanism.

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Conflict of Interest

The authors declare that there are no conflicts of interest

Author Contribution

The authors confirm contribution to the paper as follows: ***study conception and design:*** *Mustapha Mukhtar, K. I. Omoniyi;* ***data collection:*** *Mukhtar Mustapha, Faizuan Abdullah;* ***analysis and interpretation of results:*** *Mustapha Mukhtar, K. I. Ominiyi, Faizuan Abdullah;* ***draft manuscript preparation:*** *Mustapha Mukhtar. All authors reviewed the results and approved the final version of the manuscript.*

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