Modelling and optimization of zinc recovery from Enyigba sphalerite in a binary solution of hydrochloric acid and hydrogen peroxide

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Synopsis

This work focused on the prediction of optimal conditions for zinc recovery from sphalerite in a binary solution of hydrochloric acid and hydrogen peroxide. The sphalerite sample was characterized with X-ray fluorescence spectrometry (XRF), X-ray diffractometry, and Fourier transform infrared analysis (FTIR). The central composite design of response surface methodology (RSM) developed in Design Expert software and the genetic algorithm (GA) tool in matlab, were deployed for the optimization exercise. The leaching temperature, acid concentration, stirring rate, leaching time, and hydrogen peroxide concentration were defined as input variables, while zinc yield was the response. An ideal zinc yield of 90.89% could be obtained with a leaching temperature of 84.17°C, HCl concentration of 3.14 M, stirring rate of 453.08 r/min, leaching time of 107.55 minutes, and hydrogen peroxide concentration of 3.93 M using RSM; while a yield of 87.73% was obtained using GA. Analysis of the post-leaching residue revealed the presence of sulphur, zircon, fluorite, gahnite, anatase, and sylvite.

Keywords
sphalerite leaching, genetic algorithm, optimization, response surface methodology.

Introduction

Sphalerite (ZnS), also known as zinc blende, is the primary mineral of zinc. It is usually found with other sulphide minerals such as galena (PbS), pyrite (FeS_2), chalcopyrite ($CuFeS_2$), and barite ($BaSO_4$). Zinc has been recovered from sphalerite concentrate for decades via the roast-leach-electrowinning (RLE) process (Souza, Pina, and Leao, 2007). However, the RLE process has a major shortcoming arising from the roasting stage, as it emits SO_2 which causes pollution. To avert this problem, two similar routes were proposed in the 1970s as alternatives to the RLE procedure. The first involves direct leaching with oxidative reagents such as acids, ferric salts, hydrogen peroxide, or magnesium oxide (Guler, 2016); while the second process involves pressure leaching carried outs in autoclaves at 14–15 atm. oxygen pressure (Souza, Pina, and Leao, 2007; Baldwin and Demapolis, 1995). Zinc and its compounds have found application as anti-corrosion agent, in paint production, in the manufacture of rubber, in photocopying products, in organic synthesis, among others (Marks, Pearse, and Walker, 1985).

The leaching of sphalerite in a non-oxidizing but complexing medium like HCl would form zinc chloride and hydrogen sulphide according to Equation [1] (Baba and Adekola, 2010), while sphalerite leaching in a binary solution of hydrochloric acid and hydrogen peroxide leads to the formation of zinc chloride, water, hydrogen sulphide, and elemental sulphur as illustrated in Equation [2].

$$ZnS_{(s)} + 2HCl_{(aq)} \rightarrow ZnCl_{2\,(aq)} + H_2S_{(g)} \quad [1]$$

$$2ZnS_{(s)} + 4HCl_{(aq)} + H_2O_{2(aq)} \rightarrow 2ZnCl_{2(aq)} + 2H_2O_{(l)} + H_2S_{(g)} + S^{o}_{(s)} \quad [2]$$

The well-known method for optimizing a process by changing one parameter at a time while maintaining the others at a constant but unspecified level does not reveal the overall impact of all the parameters involved (Nnanwube and Onukwuli, 2020). This 'one factor at a time' approach is tedious and requires a vast number of experiments, which can be misleading. These limitations can be avoided by optimizing all the parameters together by statistical experimental design such as the response surface methodology (RSM) (Ko, Porter, and Mckay, 2000). RSM is based on polynomial surface analysis and is a collection of mathematical and statistical techniques that are helpful for the modelling and analysis of problems in which a response of interest is affected by a number of factors. The major goal of RSM is to establish the optimum operational conditions for a process. The most generally applied of RSM designs is the central composite design (CCD) (Box, Hunter, and Hunter, 1978).

Genetic algorithms (GAs), on the other hand, are a group of computational models inspired by evolution. These algorithms encode a potential solution to a particular problem on a basic chromosome-like data structure and apply recombination operators to these structures in order to preserve critical information. They are frequently viewed as function optimizers, although the scope of problems to which GAs have been applied is very wide (Nix and Vose, 1992). GAs, as opposed to conventional methods, work concurrently with a population of individuals, exploring a number of new areas in the search space in parallel, thus reducing the probability of being trapped in a local minimum. As in nature, individuals in a population compete with each other for survival so that fitter individuals tend to advance into new generations, while the poor ones usually die out (Matous *et al.*, 2000). The GA technique has been found to be an efficient optimization tool. It has been applied in analysing leaching data for low-grade manganese ore (Pettersson *et al.*, 2009), software testing (Sharma, Patani, and Aggarwal, 2016), wireless sensor networks (Liu and Ravishankar, 2011), as well as predicting recovery during column leaching of copper oxide ore (Hoseinian *et al.*, 2020), among others.

Although some work had been reported on the dissolution of sphalerite in acids and oxidative reagents (Souza, Pina, and Leao, 2007; Baba and Adekola, 2010; Nnanwube, Udeaja, and Onukwuli, 2020), studies on the application of soft computing techniques in modelling the process are scant. Hence there is need for more research in this area. It was the purpose of this research to assess the efficiency of RSM and GAs in predicting the recovery of zinc from sphalerite in a binary solution of hydrochloric acid and hydrogen peroxide. This work will serve as a good reference material on the modelling of zinc recovery from sphalerite.

Materials and methods

The sphalerite used for this investigation was sourced from Enyigba in southeastern Nigeria. The sample was finely pulverized and sieved to <75 µm. Solutions of HCl and H_2O_2 were prepared with analytical grade reagents and deionized water.

Elemental analyses were carried out by X-ray fluorescence spectrometry (XRF) using an X-supreme 600 from Oxford instruments. Mineralogical examination was carried out with an ARL X'TRA X-beam diffractometer from Thermoscientific (serial number 197492086). The FTIR analysis was carried out using a Shimadzu 8400S FTIR spectrophotometer, while the Varian AA240 model was used to conduct the AAS analysis.

Experimental procedure

Leaching experiments were performed in a 500 mL glass glass reactor fitted with a condenser to avoid losses through evaporation. A magnetically-stirred hot plate (model 78HW 1) was used for the experiments. For every leaching experiment, the solution was prepared by dissolving 20 g/L of the sample in hydrochloric acid/hydrogen peroxide binary solution at the required temperature and stirring rate, as determined by the experimental design. At the end of the reaction, the undissolved material in the suspension was allowed to settle and separated by filtration. The solutions obtained were diluted and analysed for zinc using atomic absorption spectrophotometer (AAS) (Nnanwube, Udeaja, and Onukwuli, 2020).

Experimental design and RSM model development

The effects of leaching temperature, acid concentration, stirring rate, leaching time, and hydrogen peroxide concentration on the recovery of zinc from sphalerite were assessed using RSM. A central composite rotatable design (CCRD) with five levels and five factors was deployed for the modelling and optimization studies. Table I gives the variables and their levels while the coded CCRD for the 32 experimental runs is presented in Table II. The experimental runs were randomized to minimize the impacts of unexpected variability in the observed responses. The method employed generates a second-order polynomial that describes the process. To connect the response to the independent variables, multiple regressions were used to fit the coefficient of the second-order polynomial model of the response. The quality of the fit of the model was assessed using a test of significance and analysis of variance. In RSM, the most generally utilized second-order polynomial equation created to fit the experimental data and classify the applicable model terms is presented in Equation [3].

$$Y = \beta_0 + \sum_{i=1}^{n} \beta_i x_i + \sum_{i=1}^{n-1} \sum_{j=2}^{n} \beta_{ij} x_i x_j + \sum_{i=1}^{n} \beta_{ii} x_i^2 + \varepsilon \qquad [3]$$

where

- Y is the predicted response variable, which is the percentage yield of zinc in this investigation
- β_0 is the constant coefficient
- β_i is the *i*th linear coefficient of the input variable x_i
- β_{ii} is the *i*th quadratic coefficient of the input variable x_i
- β_{ij} is the different interaction coefficients between the input factors x_i and x_j
- ε is the error of the model.

Design Expert software package version 10.0 (Stat-Ease Inc., Minneapolis, MN, USA) was deployed for regression analysis and analysis of variance.

Optimization using genetic algorithms (GAs)

A GA is a search heuristic premised on biological evolution principles to explore the solution space to locate the global minimum of a function (Yaghoobi *et al.*, 2016). GAs are mostly

Table I

Levels of independent variables for CCD experimental design

Independent variable	Unit	Symbol	Coded variable levels				
			−α	−1	0	+1	+α
Leaching temp.	°C	A	45	60	75	90	105
Acid concn.	M	B	0.25	1.5	2.75	4.0	5.25
Stirring rate	r/min	C	100	250	400	550	700
Leaching time	min	D	30	60	90	120	150
Hydrogen peroxide	M	E	0.25	1.5	2.75	4.0	5.25

Table II

Coded fractional central composite design for sphalerite dissolution in HCl/H_2O_2 binary solution

Run	Temperature (°C)	Acid conc. (M)	Stirring rate (r/min)	Leaching time (min)	Hydrogen peroxide conc. (M)
1	+1	–1	–1	+1	+1
2	+1	+1	+1	–1	–1
3	–1	+1	–1	–1	–1
4	0	0	–2	0	0
5	+1	+1	+1	+1	+1
6	–1	–1	–1	+1	–1
7	0	–2	0	0	0
8	0	0	0	0	0
9	–2	0	0	0	0
10	+1	+1	–1	+1	–1
11	–1	–1	+1	+1	+1
12	0	+2	0	0	0
13	0	0	0	0	0
14	0	0	0	–2	0
15	–1	+1	+1	+1	–1
16	0	0	0	+2	0
17	–1	+1	–1	+1	+1
18	+2	0	0	0	0
19	0	0	0	0	0
20	–1	–1	–1	–1	+1
21	0	0	0	0	0
22	+1	–1	+1	–1	+1
23	0	0	+2	0	0
24	–1	+1	+1	–1	+1
25	+1	–1	–1	–1	–1
26	+1	+1	–1	–1	+1
27	0	0	0	0	–2
28	–1	–1	+1	–1	–1
29	0	0	0	0	0
30	+1	–1	+1	+1	–1
31	0	0	0	0	0
32	0	0	0	0	+2

used for optimization purposes. GAs emulate Charles Darwin's theory of 'survival of the fittest', and are employed to resolve complex optimization problems (Goldberg and Holland, 1988). GAs have gained popularity over conventional optimization methods since they can resolve irregular or nondifferentiable fitness functions proficiently (Singh *et al.*, 2009; Gupta and Sexton, 1999; Shen, Wang, and Li, 2007). Pillay and Banzhaf (2009) applied an informed GA for the examination of a timetabling problem. Alba, Luque, and Araujo (2006) studied natural language tagging with genetic algorithms. Tseng *et al.* (2008) applied GA rule-based methodology for land-cover classification. The GA often changes the group for the individual solutions of the problem, and these changes are known as evolution. In each step of this evolution, two individuals from the group are chosen arbitrarily as the parent and child, and they are considered for the next generation. In this fashion the group advances toward an optimal solution.

To optimize the problem in the present investigation, an objective function for optimizing zinc recovery was established by experimental tests (Fayyazi *et al.*, 2015). By performing a number of trials, a suitable choice for the initial range, fitness scaling, selection, elite count, crossover fraction, mutation function, crossover function, migration, and stopping criteria was made, and lastly, the optimized solution was assessed (Ou, 2012).

Results and discussion

Characterization

The results of the XRF analysis of the sphalerite sample were reported earlier (Nnanwube, Udeaja, and Onukwuli, 2020). The results, shown in Figure 1, revealed that ZnO, SO_3, Na_2O, and Fe_2O_3 were the major oxides present in the ore; oxides such as SiO_2, CaO, Al_2O_3, Mn_2O_3, and MgO were present in minor quantities, while the other constituents occurred in traces.

The XRD results revealed that sphalerite (ZnS) was the dominant mineral with three major peaks at 28.56, 47.50, and 56.37° 2θ. The results also revealed the presence of cerium germanium sulphide (Ce_2GeS_2) with three major peaks at 30.15, 43.16, and 26.03° 2θ, respectively, as shown in Figure 2 (Nnanwube, Udeaja, and Onukwuli, 2020).

The FTIR spectrum of Enyigba sphalerite is shown in Figure 3. The spectrum show the functional groups present in the ore. The band at 504.10 cm^{-1} is ascribed to C-N-C and C-O-C bending. The band at 839.08 cm^{-1} is attributed to C-Cl and Si-C stretches, while the band at 1066.67 cm^{-1} is ascribed to SO_3 symmetrical stretch as well as Si-O-Si antisymmetrical stretch. The band at

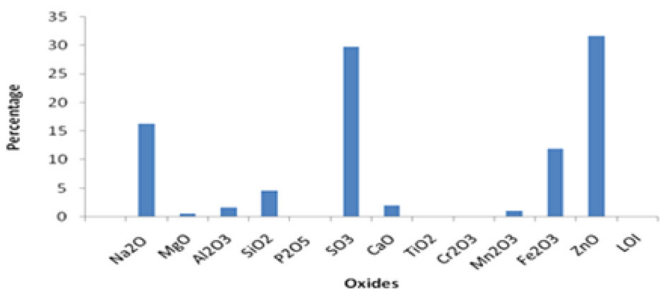

Figure 1—XRF analysis of Enyigba sphalerite

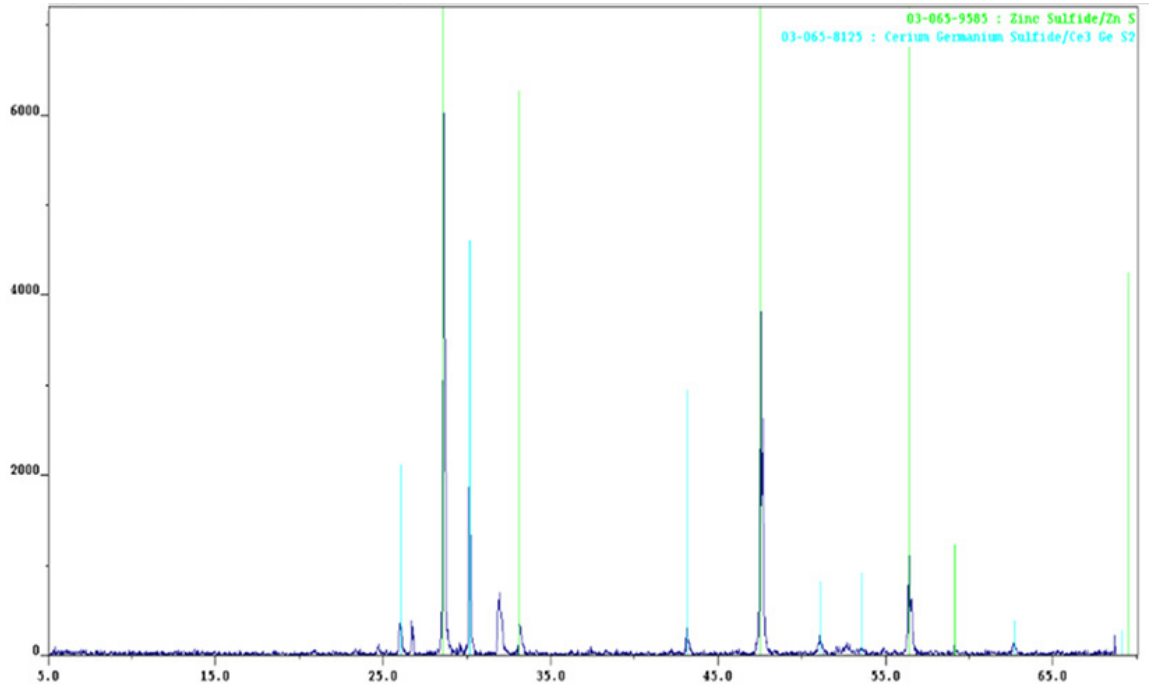

Figure 2—X-ray diffraction pattern of Enyigba sphalerite

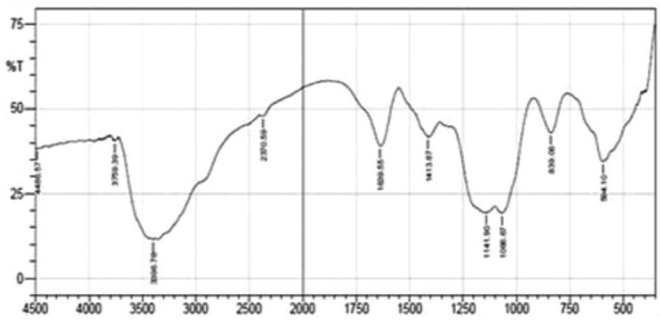

Figure 3—FTIR spectrum of Enyigba sphalerite

1141.90 cm^{-1} is ascribed to C-C-N bending and C-O-C stretch, while the band at 1413.87 cm^{-1} is ascribed to C-N stretch and OH bending. The band at 1639.55 cm^{-1} is ascribed to C=O and C=C stretches, while the band at 3396.70 cm^{-1} is ascribed to OH stretch.

RSM modelling and statistical analysis

The central composite design for the leaching of zinc from sphalerite with a binary solution of hydrochloric acid and hydrogen peroxide is shown in Table II. The five experimental factors gave a sum of 32 experimental runs with 6 centre points, 10 star points, and 16 fractional factorial design points. The responses established from different experimental runs were very distinctive, showing that all the factors had appreciable impact on the response.

Models analysed include the linear, quadratic, 2FI (two factors interaction), and cubic model. The quality of the models can be compared based their R^2 values and other parameters such as standard deviation (SD), R^2 adjusted, R^2 predicted, prediction error sum of squares (PRESS), and F- and P-values. The closer the R^2 value to unity, the better the model's fit (Ameer *et al.*, 2017). From the model analyses presented as model summary statistics in Table III the quadratic model, with the highest regression coefficient (R^2 value of 0.9934) and standard deviation of 1.07, shows better correlation between the observed and model- predicted data.

The results were further analysed using the analysis of variance (ANOVA) appropriate for the experimental design used and presented in Table IV. The model F-value of 83.27 suggested the model to be significant and there is just 0.01% chance that an F-value this large could occur due to noise. The F-value for a term is the test for comparing the change associated with that term with the residual variance. The F-values of the independent variables A, B, C, D, and E were 272.65, 260.16, 263.87, 237.23, and 265.12 respectively, demonstrating that the effects of every single independent variable on the response were considerably high.

The model P-value (Prob. > F) is low, which also demonstrates that the model is significant. The P-values were used as a means of verifying the significance of each one of the model coefficients. The smaller the P-value, the more significant the corresponding coefficient. Estimations of $P < 0.05$ confirm the model expressions to be significant. The estimations of P for the coefficients reveal that among the tested variables used in the design, A, B, C, D, E. A^2, B^2, C^2, D^2, E^2 (where A = leaching temperature, B = acid concentration, C = stirring rate, D = leaching time, and E = hydrogen peroxide concentration) are significant model terms.

Table III

Model summary statistics

Source	Std. dev.	R^2	Adjusted R^2	Predicted R^2	PRESS
Linear	4.06	0.7748	0.7315	0.7172	539.53
2FI	5.10	0.7821	0.5778	-2.1464	6003.78
Quadratic	1.07	0.9934	0.9815	0.8823	224.59
Cubic	0.93	0.9973	0.9858	0.4220	1102.90

Table IV

ANOVA for response surface quadratic model

Source	Coefficient estimate	Sum of squares	Df	F-value	P-value (Prob > F)
Model	85.29	1895.62	20	83.27	< 0.0001
A	3.60	310.32	1	272.65	< 0.0001
B	3.51	296.10	1	260.16	< 0.0001
C	3.54	300.33	1	263.87	< 0.0001
D	3.35	270.01	1	237.23	< 0.0001
E	3.55	301.75	1	265.12	< 0.0001
AB	–0.31	1.50	1	1.32	0.2752
AC	–0.42	2.81	1	2.47	0.1447
AD	–0.12	0.23	1	0.20	0.6648
AE	–0.24	0.95	1	0.84	0.3804
BC	–0.23	0.86	1	0.75	0.4044
BD	–0.056	0.051	1	0.044	0.8368
BE	–0.13	0.28	1	0.24	0.6323
CD	–0.37	2.18	1	1.91	0.1942
CE	–0.54	4.73	1	4.16	0.0663
DE	–0.12	0.23	1	0.20	0.6648
A2	–1.86	101.75	1	89.40	< 0.0001
B2	–1.78	92.42	1	81.20	< 0.0001
C2	–2.10	129.36	1	113.65	< 0.0001
D2	–1.85	100.39	1	88.21	< 0.0001
E2	–1.96	112.97	1	99.26	< 0.0001
Residual		12.52	11		
Lack of fit		8.30	6	1.64	0.3021
Pure error		4.22	5		
Cor. Total		1908.14	31		

The 'lack of fit F-value' of 1.64 infers that the lack of fit is not significant with respect to the pure error. There is a 30.21% chance that a lack of fit F-value this large could occur due to noise. A non-significant lack of fit demonstrates that the model is well fitted. Since many insignificant model terms have been eliminated, the improved model can be used to predict successfully the responses of the percentage recovery of zinc from sphalerite. The model with the significant coefficient is presented in Equation [4].

$$\text{Yield} = 85.29 + 3.60A + 3.51B + 3.54C + 3.35D + 3.55E - 1.86A2 - 1.78B2 - 2.10C2 - 1.85D2 - 1.96E2 \quad [4]$$

In terms of the actual factors, the model equation is shown in Equation [5]:

$$\text{Yield} = -86.65 + 1.66 \times \text{Leaching temperature} + 11.14 \times \text{Acid concentration} + 0.13 \times \text{Stirring rate} + 0.55 \times \text{leaching time} + 12.40 \times \text{Hydrogen peroxide concn.} - 8.28 \times 10^{-3} \times \text{Leaching temperature}^2 - 1.14 \times \text{Acid concentration}^2 - 9.33 \times 10^{-5} \times \text{Stirring rate}^2 - 2.06 \times 10^{-3} \times \text{Leaching time}^2 - 1.26 \times \text{Hydrogen peroxide concn.}^2 \quad [5]$$

The summary of regression values is presented in Table V. The CV estimation of 1.37% shows that the model can be considered reasonably reproducible (Chen *et al.*, 2010). The signal-to-noise ratio, which is given as the adequate precision, is 31.308 (Table V). This shows that an adequate relationship of signal-to-noise ratio exists. The result demonstrates that the model can be used to explore the design space.

The results were also analysed to check the correlation between the experimental and predicted zinc yields, as presented in Figure 4. The result shows a good correlation between the experimental and predicted values of the response. This demonstrates that the model selected is appropriate and that the central composite rotatable design (CCRD) can be deployed for the optimization exercise.

Table V

Summary of regression values

Std. dev.	Mean	CV (%)	PRESS	Adequate precision
1.07	78.12	1.37	224.59	31.308

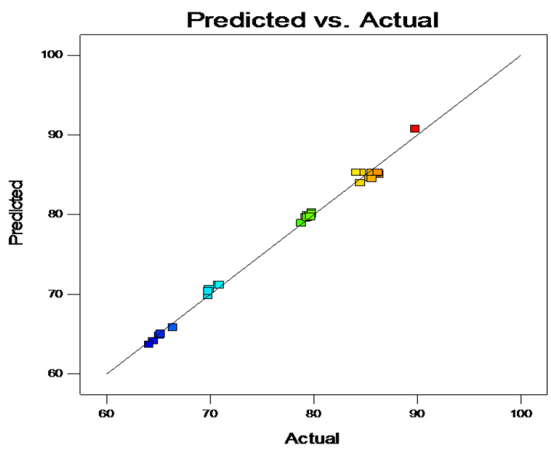

Figure 4—Plot of predicted values *versus* experimental values

Response surface plots

The combined effects of adjusting the process variables within the design space were observed by constructing 3D surface plots (Figure 5). Figure 5a shows the effect of acid concentration and leaching temperature on zinc yield. From Figure 5a, as

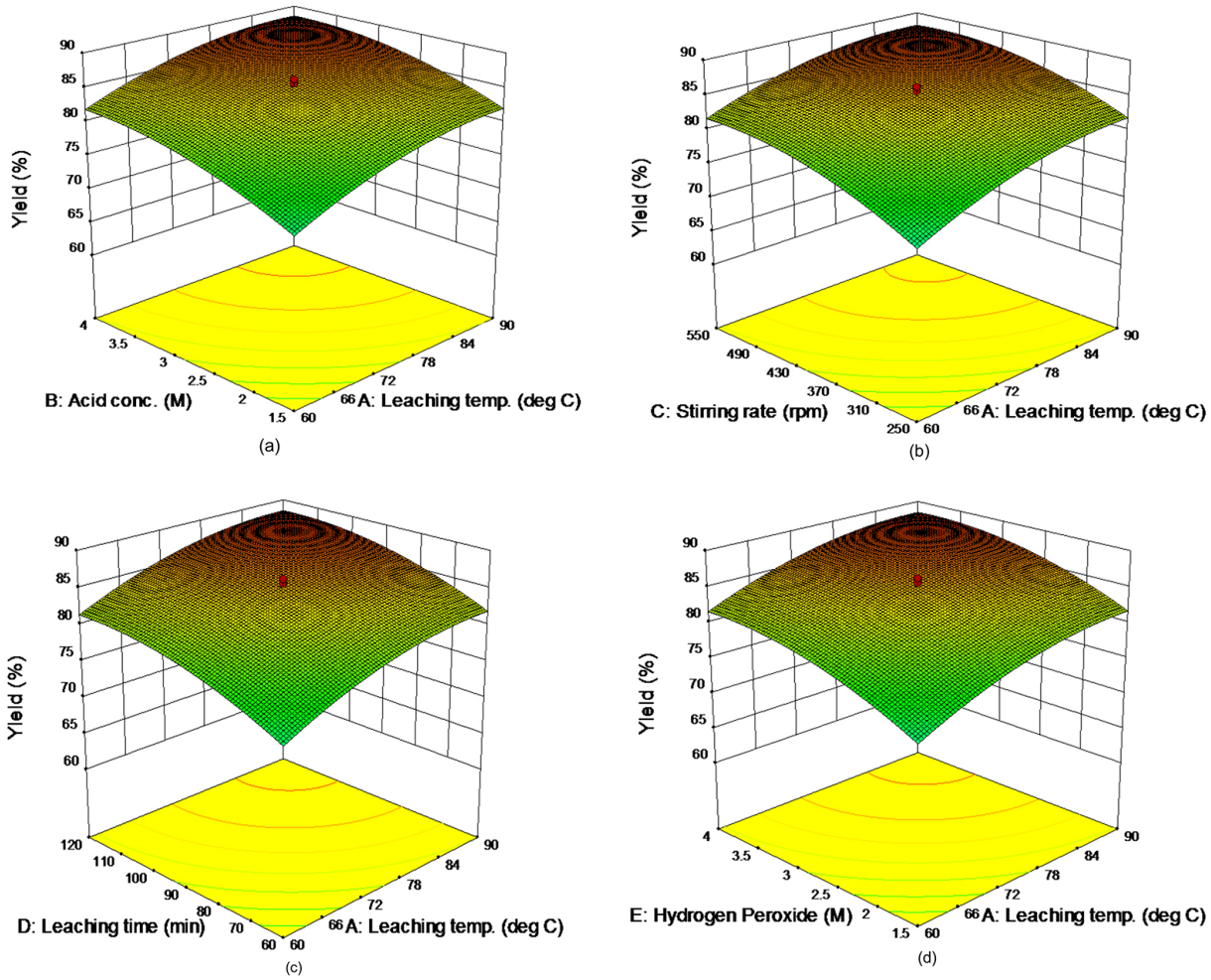

Figure 5—3D response surface plots on effect of process variables on zinc yield

the leaching temperature was increased from 60 to 90°C, the percentage recovery of zinc increased from 82 to 88.5 %, while as the acid concentration was increased from 1.5 to 4 M, the recovery increased from 82.3 to 88.5%. The effect of stirring rate and leaching temperature on the percentage yield of zinc is shown in Figure 5b. As the stirring rate was increased from 250 to 490 r/min, the recovery increased from 81.9 to 88%, while as the leaching temperature was increased from 60 to 90°C, the recovery increased from 81.8 to 88%. The effect of leaching temperature and leaching time on the percentage zinc yield is shown in Figure 5c. As the leaching temperature was increased from 60 to 90°C, the recovery of zinc increased from 81.4 to 88.6%, while as the leaching time was increased from 60 to 120 minutes, the recovery increased from 81.9 to 88.6%. Figure 5d shows the effect of hydrogen peroxide concentration and leaching temperature on the zinc recovery. As the hydrogen peroxide concentration was increased from 1.5 to 4 M, the zinc recovery increased from 81.7 to 88.5%, while as the leaching temperature was increased from 60 to 90°C, the recovery increased from 81.8 to 88.5%. The 3D plots are helpful in understanding the interactive effects of the process parameters within the leaching system.

Optimization of the leaching process using response surface methodology (RSM) and genetic algorithm (GA)

The genetic algorithm tool of Matlab and the optimization tool of Design Expert were employed for the optimization study. Equation [5] was solved for the best solutions to ensure that the responses are maximized within the design space. A conventional method, which involves selecting the most economically viable option, was adopted. In choosing the objective for each of the factors for the numerical optimization, various considerations were taken into account. The significance of each of the variables in terms of the final response was the most vital consideration. The response was set at maximum value; and every other single factor was kept in range except the reaction time, which was set to a minimum target. In view of these considerations, the software predicted optimum reaction conditions with a desirability of 1.00. The ideal conditions for zinc recovery were a temperature of 84.17°C, HCl concentration of 3.14 M, stirring rate of 453.08 r/min, leaching time of 107.55 minutes, and hydrogen peroxide concentration of 3.93 M. The yield of zinc at these optimum conditions was 90.89%, which was validated as 89.65% by conducting three independent experimental replicates.

The GA optimization parameters were obtained by carrying out a number of trials. The best condition was established in which the initial range was [1,100], the selection function was stochastic uniform, the values of the elite count and crossover fraction were respectively equal to 2 and 0.8, while the mutation and crossover function were selected as well as the migration, stopping criteria, output function, and level of display respectively.

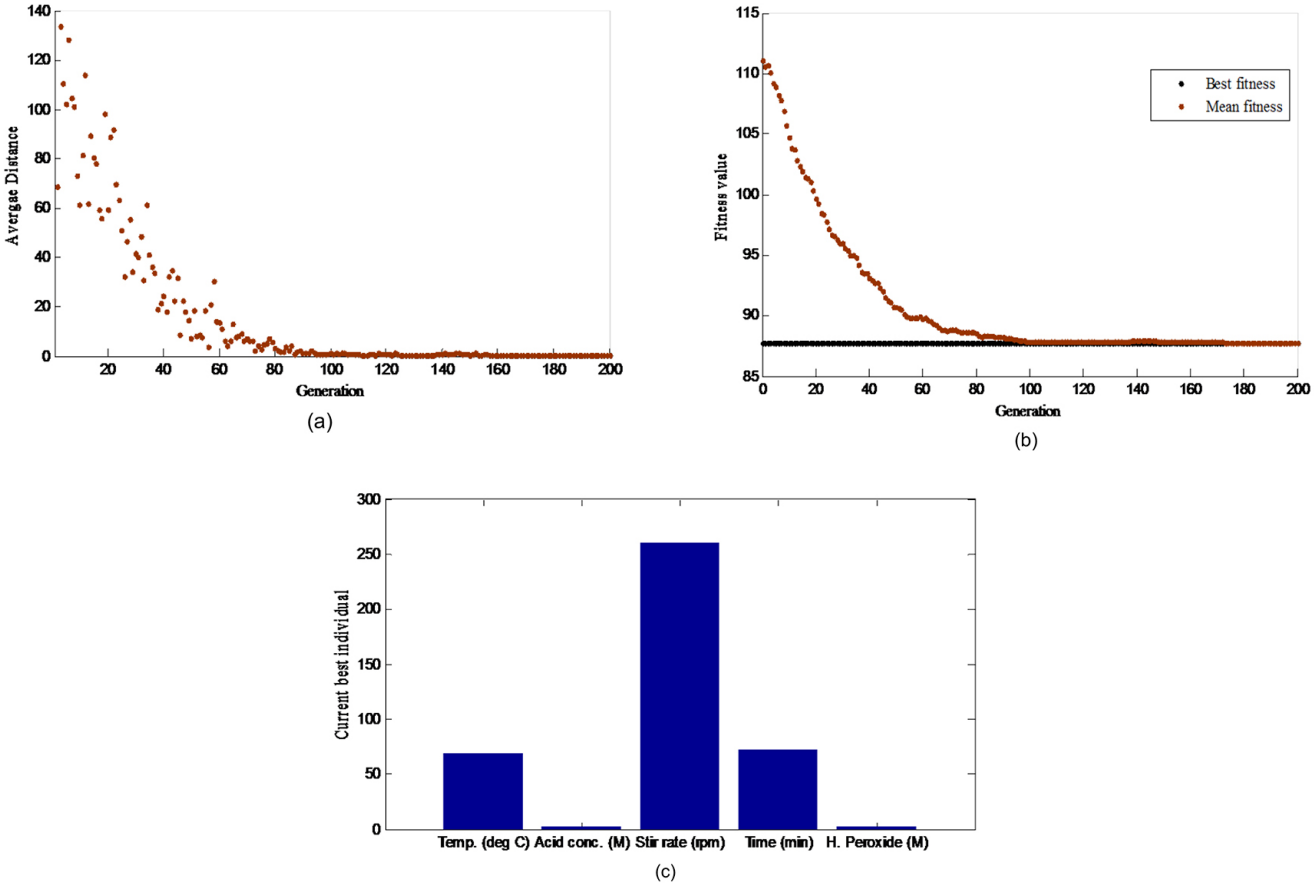

Figure 6—The charts for the points obtained by GA. (a) Average distance between the individuals in each generation, (b) the best value for the fitness function, and (c) the optimal values obtained for the fitness function's independent variables

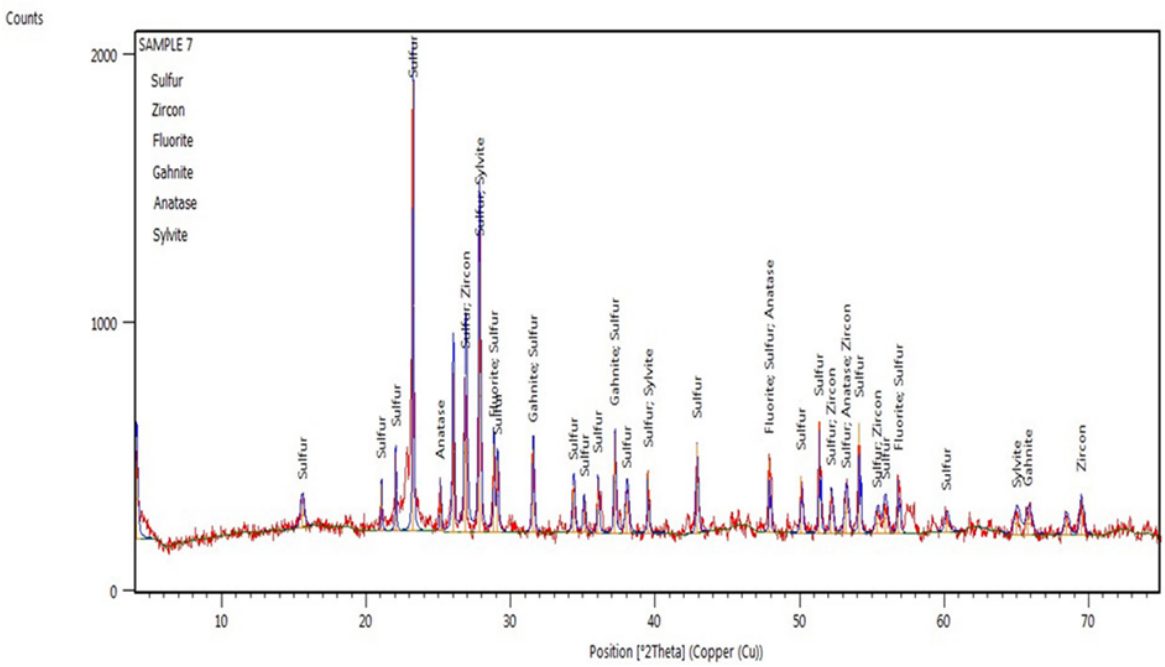

Figure 7—X-ray diffractogram of the post-leach residue

Figure 6a shows the average distance between the individuals for each generation. It can be observed that for this case, the group has good array. Figure 6b shows the best estimation of the fitness function of generation 200, which is 87.73%. The optimal values of the independent variables for the leaching temperature, acid concentration, stirring rate, leaching time, and hydrogen peroxide concentration were equivalent to 69°C, 2.55 M, 260 r/min, 72 minutes, and 2.3 M respectively (Figure 6c). The

zinc yield estimated was validated in triplicate with an average value of 86.70%. This shows that GA is very effective for the optimization exercise.

Analysis of the residue

The residue from sphalerite leached with 3.14 M HCl/3.93 M H_2O_2 at 84.17°C was analysed by XRD. The X-ray diffractogram (Figure 7) showed three principal peaks at 23.24, 27.84, and 26.89° 2θ. The XRD data revealed the presence of sulphur, zircon, fluorite, gahnite, anatase, and sylvite. It is important to note the absence of sphalerite in the residue, which shows that the bulk of the sphalerite must have dissolved.

Conclusion

The optimum conditions for the recovery of zinc from sphalerite with a binary solution of hydrochloric acid and hydrogen peroxide were investigated. Response surface methodology (RSM) and genetic algorithms (GAs) were deployed for the modelling and optimization of process parameters. Analysis of experimental results revealed that the quadratic model gave a good description of the experimental data. The process input variables for the leaching process were optimized by GA and RSM for best zinc yield. The results indicated that GA predicted a zinc yield of 87.73%, while RSM predicted a yield of 90.89%.

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