Development of an empirical model for copper extraction from chalcocite in chloride media

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Abstract
Multivariate models are a useful tool when studying the effects of independent variables on one or more dependent variables, since this approach allows modeling of the dynamics of complex systems based on simple analytical models with considerable certainty. Due to the decrease in the copper oxide mineral grades, leaching of copper sulfide minerals (secondary sulfides) has positioned itself as a benchmark of operation for the Chilean mining industry. The present work proposes the study of the effects of sulfuric acid, chloride concentration and time on the extraction of copper from sulfuric minerals (chalcocite), considering an experimental design, the surface optimization methodology and the adjustment of a quadratic model. The experimental data were adjusted by multiple regression analysis and were statistically analyzed. A model was developed to represent the copper extraction from the Cu_2S mineral as a function of the statistically significant variables (chloride concentration and time) that contribute to explain the variation of the response variable under the set of parameters sampled.

Keywords: minerals, design of experiments, statistical validation, modeling.

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1. INTRODUCTION

Chile is the main copper exporter in the world [1–3] and it is also the country with the largest copper reserves [4]. However, there is a clear need in the country to generate new initiatives and/or technologies to stop the recession in the growth of this industry in last years. Among the main factors that affect the level of the country's mining are a decrease in mineral grades in the deposits [5,6], increase in treatment costs for new minerals to be processed (secondary and primary sulfides) and aging of mineral deposits [7–10]. In addition, in recent years problems of environmental pollution have had an impact on discontent and social mobilizations. Chile has currently approximately 7,000 mining deposits, considering small, medium, and large mining, where 55 % of them are copper reservoirs [4]. In the past, exploitation of mining worksites in Chile was carried out mainly by the hydrometallurgical route for the treatment of oxidized copper minerals. However, in large copper mining, oxidized minerals are increasingly becoming scarce, where it is projected that they will decrease from approximately 30 % today to 12 % in 2027 [11].

Sulfurized minerals correspond to 69 % of the Chile's copper production [12]. These are mainly treated by flotation and pyrometallurgical processes [13–15]. However, these types of processes generate large environmental liabilities, such as tailings dams in mineral concentration processes and smelting plants, sulfur dioxide emissions (SO_2), which together with NO_x and CO_2 can cause major problems such as acid rains and increased local pollution [16,17].

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Most of copper minerals on the planet correspond to sulfur minerals and a minor quantity to oxidized minerals [18]. Among the secondary sulfides, chalcocite is the most abundant mineral [19] and requires relatively simple forms of treatment compared to the other sulfides [20]. Chilean mining companies operate in extremely arid areas, which has considerably increased the use of seawater in mineral processing [21]. In recent years, various studies have been developed for the use of seawater in extractive metallurgy processes, either for flotation and thickening [22–24] and leaching [25–27] processes. When working in chlorinated media, the oxidative dissolution of chalcocite occurs in two stages [28,29], where the first stage is faster while the second can be accelerated with the increase in temperature [17,19,30,31]. This is problematic, because of long leaching times that imply long waits in cash flows in companies and the alternative of increasing temperature is not viable for conventional processes [8,32–34].

The leaching process has been studied by several authors [35–42] and the leaching of chalcocite in chlorinated media has been previously studied [20,28,43]. Toro *et al.* [17], studied the effect of chloride ions from seawater and wastewater from desalination plants for leaching of a pure chalco-mineral and discovered that the second option provided better results due to higher concentration of chloride ions. In addition, it was found that sulfuric acid had little relevance in Cu extraction. However, in this study [17], the authors evaluated the effect of H_2SO_4 in very narrow ranges, without considering cases in which the concentration of chloride in the system is low.

In the present investigation, an empirical model is proposed that validates the experimental design of copper extraction from sulfide minerals. Generation of this model type contributes to investigating the effects of input variables or factors on one or more response variables by intentional changes, which are used to identify the conditions in the process and its components that affect the extraction behavior, in order to identify the optimal configuration of factors.

2. MATERIALS AND METHODS

2. 1. Chalcocite

The mineral used is from the Michilla Mine, located in northern Chile. It was selectively taken from a vein inside the mine, seeking that the chalcocite ore was as pure as possible. Subsequently, the material was reduced to the size range 150 ± 106 µm using a porcelain mortar. It was then chemically analyzed by atomic emission spectrometry *via* induction-coupled plasma (ICP-AES), developed in the Applied Geochemistry Laboratory of the Department of Geological Sciences of the Universidad Católica del Norte. It was determined that the mineral has a chemical composition of 79.83 % Cu, 8.49 % Fe and 11.68 % of S. Copper Sulfide, PDF 83-1462, corresponding to the ICDD database PDF-2 (2004). Also, a mineralogical study was conducted by using a Bruker brand X-ray diffractometer, automatic and computerized model D8 (FEI Company, Brisbane, Australia). Figure 1 presents results of the analysis, which shows that the chalcocite mineral is 99.90 % (The peaks correspond to the previously described database).

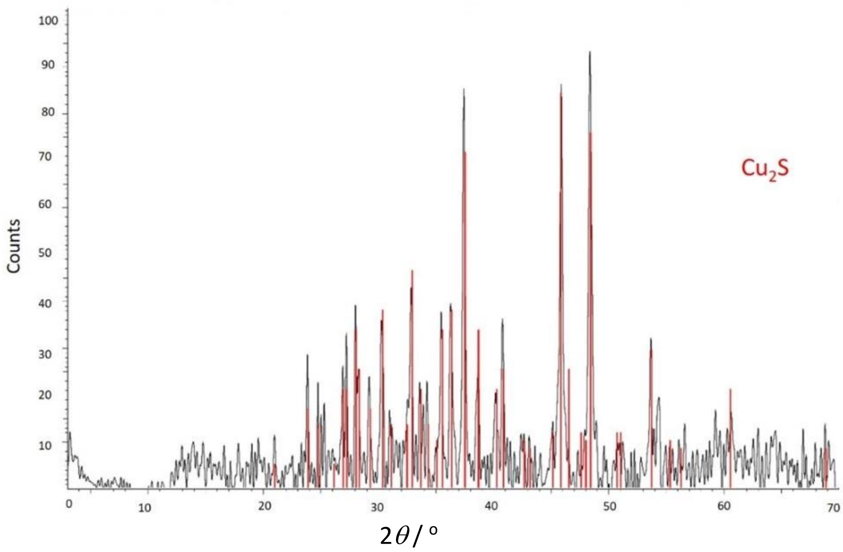

***Figure 1**. X-ray diffractogram for the chalcocite mineral*

2. 2. Leaching tests

Leaching tests were carried out in a 50 mL glass reactor with a 0.01 S/L (solid/liquid) ratio of leaching solution. A total of 200 mg of chalcocite ore was maintained in suspension with the use of a 5-position magnetic stirrer (IKA ROS, CEP 13087-534, Campinas, Brazil) at a speed of 600 rpm. The tests were conducted at room temperature of 25 °C, with variations in sulfuric acid and chloride (NaCl) concentrations as well as the leaching time. The tests were performed in triplicate. Measurements were carried out by analyzing 5 mL undiluted samples by using atomic absorption spectrometry with a coefficient of variation ≤ 5 % and a relative error between 5 to 10 %. Measurements of pH and the oxidation-reduction potential (ORP) of leach solutions were made using a pH-ORP meter (HANNA HI-4222, HANNA instruments, Woonsocket, RI, USA) The solution ORP was measured in an ORP electrode cell containing a platinum working electrode and a saturated Ag/AgCl reference electrode.

2. 3. Experimental design

The effect of independent variables on Cu extraction were studied using the response surface optimization methodology [44,45]. The Composite Central Face (TLC) design and a quadratic model were applied to the experimental design for Cu extraction [46]. The methodology used consisted of copper sulfide leaching experiments, 125 experimental tests in total, to study the effects of H_2SO_4 concentration, chloride concentration and time (see Table 1) on the dependent variable. For the experimental modeling and design, the Minitab 18 software (Minitab LLC, PA) [47,48] was used, allowing to investigate linear effects, interactions and quadratic effects of the independent variables in the copper recovery. The experimental data were fitted by means of multiple regression analysis [49] to a quadratic model, considering only those factors that contribute to explain the variability of the model and that have a high statistical significance.

Table 1. Experimental parameters for the central design of the composite face

Factor/ Level	Lower	Low	Medium	High	Higher
$c_{H_2SO_4}$ / mol dm^{-3}	0.5	0.75	1	1.5	2
c_{Cl^-} / g dm^{-3}	20	35	50	75	100
Time, h	3	4.5	6	7.5	9
Codification	-1	-0.5	0	0.5	1

Then, the general shape of the experimental model is given by Equations 1 and 2 [50]:

$$Y = \text{constant} + \text{linear effects} + \text{interaction effects} \quad (1)$$

$$Y = b_0 + b_1x_1 + b_2x_2 + b_3x_3 + b_{11}x_1^2 + b_{12}x_1x_2 + b_{13}x_1x_3 + b_{22}x_2^2 + b_{23}x_2x_3 + b_{33}x_3^2 \quad (2)$$

where, Y is the response variable, b represents the coefficients of the independent variables x, which are the sample parameters (time, and chloride and H_2SO_4 concentrations).

The goodness of fit of the regression model presented by the Eq. (2) is studied by determining the coefficient of determination, R^2, and p values, which indicate if the model obtained is adequate to describe the mineral extraction under the domain sampled. The coefficient of determination, R^2, measures the proportion of the total variability of the dependent variable with respect to its mean that is explained by the regression model, while p values represent statistical significance, indicating whether there is a statistically significant association between the response variable and the term [51].

3. RESULTS AND DISCUSSION

By analyzing the main effects of linear parameters, interactions and quadratic effects, presented in Figure 2, it is evident that the linear effects of time and chloride concentration (see Fig. 2a), the effects of the interaction of time and chloride (see Fig. 2b), and the quadratic effect of time (see Fig. 2c) have the main effects on the experimental design of the model. Additionally, the contour plot showed in Figure 3 indicates that copper extraction increases at high levels of time and chloride concentration.

Based on the information obtained by the ANOVA analysis (Table 2), effects of the interactions {chloride and H_2SO_4 concentration} and {time, H_2SO_4 concentration} on copper extraction are not significant (p> 0.05). Only the interaction

{Time, Chloride} and the linear and quadratic effects of time and chloride concentration contribute to explain the variations in experimental data. Additionally, the linear and curvature effects of the H_2SO_4 concentration variable do not contribute to explain the variation in the response variable.

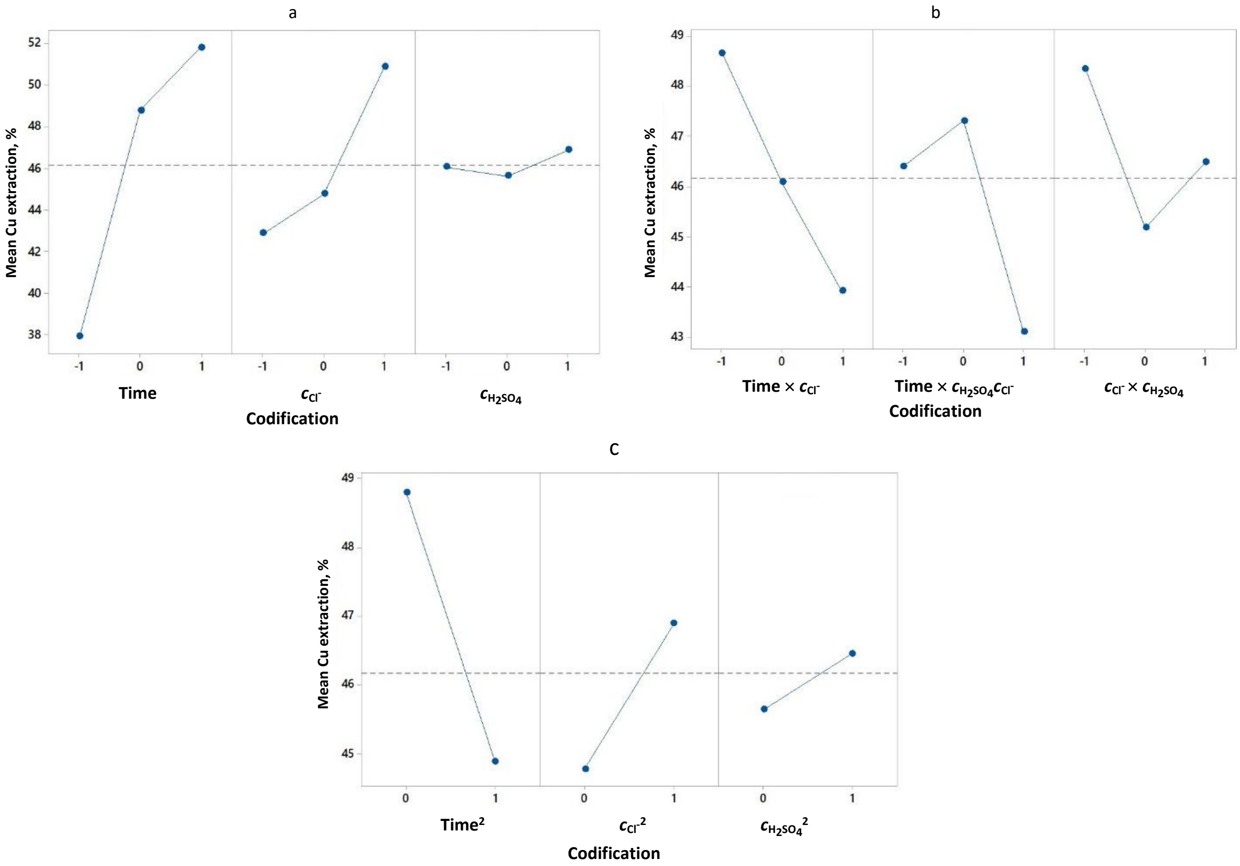

Figure 2. Plots of linear (a), interaction (b) and quadratic (c) effects of independent variables on copper extraction from chalcocite (plots generated in MINITAB 18 software[47])

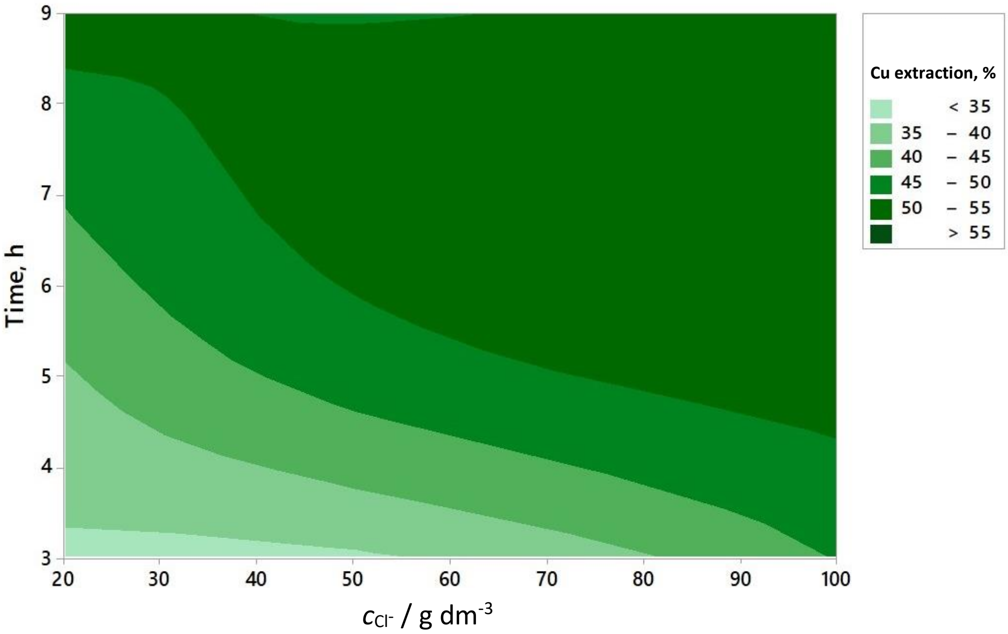

Figure 3. Contour plot of Cu extraction as a function of time and chloride concentration.

Table 2. ANOVA of experimental design

Source	Adj SS	Adj MS	F-Value	*p*-value
Regression	4883.81	976.762	133.37	0.000
Time	759.12	759.121	103.66	0.000
c_{Cl^-}	115.35	115.35	15.75	0.000
Time^2	274.22	274.215	37.44	0.000
$c_{Cl^-}{}^2$	102.84	102.84	12.84	0.000
Time × c_{Cl^-}	194.44	194.441	26.55	0.000

Adj SS - adjusted sums of squares; Adj MS - adjusted mean squares

After deletion of the insignificant coefficients, the model developed to predict mineral extraction over the range of tested experimental conditions is presented by the Eq. (3).

$$\text{Cu extraction} = 100\,(0.4879 + 0.0694x_1 + 0.04x_2 - 0.0237x_1x_2 - 0.0393x_1^2 - 0.0154x_2^2) \quad (3)$$

Here x_1 and x_2 are coded variables that represent time and chloride concentration, respectively. The ANOVA test (Table 2) indicates that the presented quadratic model, Eq. (3), is adequate to represent the Cu extraction under the established parameter range. There is no lack of fit of the model, and the R^2 statistics (see Table 3) and the standard error of the regression (S) validates it. ANOVA analysis shows that the indicated factors influence in Cu extraction, because *F* statistic or $F_{\text{Regression}}$ (133.37) [see Table 2] > $F_{\text{Theorical; 95 \% confidence level}}$ ($F_{5,119}$ = 2.29). Additionally, the *p*-value of the model represented by Eq. (3) indicates that the model is statistically significant.

Table 3. Model summary

S (Cu extraction, %)	R^2, %	Adjusted R^2, %	Predicted R^2, %
2.7062	84.86	84.22	83.38

Finally, in the response surface shown in Figure 4, it is found that copper extraction increases with time and the increase in Cl concentration.

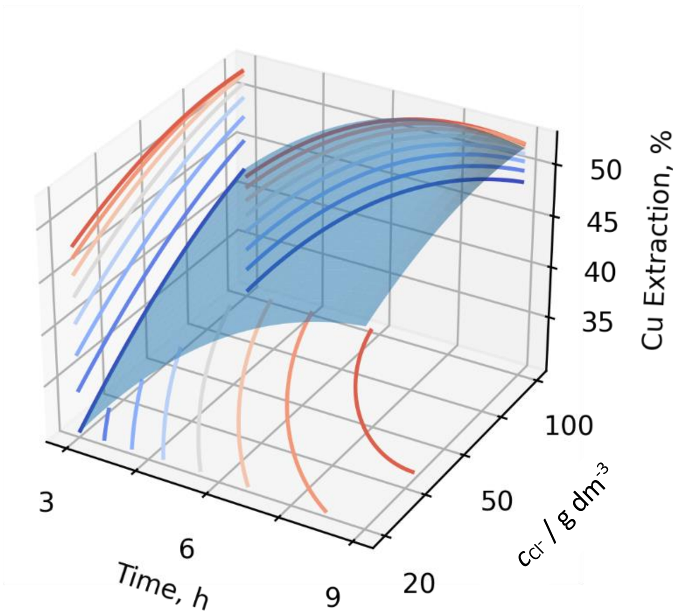

Figure 4. *Response surface of the independent variables Cl concentration and time in copper extraction (plot generated matplotlib library in Python 3.7.0 programming language [52]).*

The methodology used in the present manuscript to evaluate the extraction of copper from chalcocite has also been used for other copper minerals, as for example, in the study carried out by Pérez *et al.* [53] for covellite, and the study by Aguirre *et al.* [54] for chalcopyrite, the world's most abundant copper mineral [27,55,56]. Aguirre *et al.* [54] proposed a statistical model (based on the response surface methodology) to study dynamics of copper extraction from a pure chalcopyrite mineral, evaluating the following working parameters: acid concentration (0, 0.25 and 0.5 M), chloride concentration (0, 50 and 100 g dm^{-3}), temperature (30, 60 and 90 °C) and ionic liquid concentration (20, 40 and 60 % v/v). The results indicated that the concentration of chloride in the system and the increase in temperature have a synergistic effect on the copper extraction, while operational variables such as concentrations of the ionic liquid and sulfuric acid did not influence the dissolution of $CuFeS_2$. Finally, Aguirre *et al.* [54] indicated that the optimal working conditions are: 20 % v/v of the imidazolium-based ionic liquid, 100 g dm^{-3} chloride and the temperature of 90 °C. This is consistent with the results of the present study, where the concentration of sulfuric acid has a low importance in the dissolution of Cu_2S, where the concentration of Cl^- is being more relevant.

4. CONCLUSION

Under normal pressure and temperature conditions, chloride concentration and time have a synergistic effect on the extraction of copper from chalcocite, while the sulfuric acid concentration has little influence on the extraction percentage. The highest extraction yields were obtained at higher chloride concentrations and at the last sampling time tested (9 h).

The experimental results of Cu_2S leaching using NaCl and H_2SO_4 were satisfactorily adjusted by multiple regression analysis to a quadratic model that describes the effects of each studied variable on the Cu extraction. The final model predicts the Cu extraction depending on the chloride concentration and extraction time. Therefore, it can be concluded that it is possible to treat secondary copper sulfides with high contents of chalcocite, using sources with high chloride contents, such as wastewater from drinking water desalination plants, and working at low concentrations of sulfuric acid.

Within the continuation lines of this research work, it is being considered to include the sampling of more factor levels develop models that incorporate a greater number of operational variables, generate simulation models of the dynamics of the chalcocite leaching process, development models based in machine learning algorithms and stochastic modeling.

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SAŽETAK

Razvoj empirijskog modela za ekstrakciju bakra iz halkozina u medijumu koji sadrži hloridne jone

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(Stručni rad)

Višeparametarski modeli su korisno sredstvo za proučavanje efekata nezavisnih promenljivih na jednu ili više zavisnih promenljivih pošto ovaj pristup zasnovan na jednostavnim analitičkim modelima omogućava modelovanje dinamike složenih sistema sa značajnom izvesnošću. Zbog sve manjeg sadržaja bakra u oksidnim rudama bakra, izluživanje bakra iz minerala bakar-sulfida postavljeno je kao novi standard rada čileanske rudarske industrije. Ova studija obuhvata ispitivanje uticaja sumporne kiseline, koncentracije hloridnih jona i vremena na ekstrakciju bakra iz sulfidnog minerala-halkozina, uzimajući u obzir eksperimentalni dizajn, metodologiju odzivne površine i podešavanje modela kvadratne zavisnosti. Na eksperimentalne podatke je primenjena višestruka regresija i rezultati su statistički analizirani. Razvijen je model koji predviđa ekstrakciju bakra iz minerala halkozina (Cu_2S) u funkciji statistički značajnih promenljivih (koncentracija hloridnih jona i vreme) u okviru opsega testiranih parametara.

Ključne reči: sulfidni minerali, eksperimentalni dizajn, statistička validacija, modelovanje