

An investigation on the potential recovery of copper from a low-grade copper resource containing high silica content

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Abstract

The present research aimed to experimentally peruse the tank leaching of copper from a low-grade ore containing 0.57% Cu, 24.69% Si, and 10.367% Fe. To this end, firstly, the behaviour of important variables influencing the dissolution process was examined using the one factor at a time (OFAT) method and response surface modelling (RSM), and then the leaching kinetics was scrutinized to obtain further recognition of the leaching operations. The findings derived from the OFAT technique demonstrated that the particle size of 0-2 mm was a better choice for the tank leaching relative to other fractions. The presence of clay compounds in the ore led to a high increase in acid consumption. The leaching rate improved with an increase in the solid percentage from 20 to 40% and then decreased with a further increase due to lack of proper mixing. Additionally, the pulp stirring speed had a little effect on the dissolution of copper, confirming that the mixing rate of pulp should be at a level that suspends the particles and prevents them from settling. Then, the RSM was utilized to model and optimize the process and determine the possible interaction between operating parameters. The results showed that copper leaching was significantly dependent on the interaction between the factors. The optimized amounts of parameters were found to be 90 kg/t acid concentration, 35% solid content, 150 rpm pulp mixing rate, 2.5 h leaching time, and 0-2 mm size range. The kinetic analysis showed that the diffusion model through the production layer with an activation energy of 14.15 kJ/mol was the rate-controlling stage of the reaction, and based on that, a new model describing the dissolution kinetics of copper was proposed.

Keywords:

copper oxide, tank leaching, extraction efficiency, optimization, dissolution kinetics

1. Introduction

Copper is one of the most important and widely used industrial metals worldwide after iron and aluminum. Many industries have direct or indirect dependence on this strategic metal due to its unique features, like high thermal and electrical conductivity, alloying ability, ductility, malleability, and resistance to corrosion (Nozari and Azizi, 2020; Hosseinzadeh et al., 2021). The widespread use of copper means that it is directly affected by global economic conditions, and any minor changes in the economic statistics can lead to fluctuations in its price. In nature, copper can combine with almost every element in the periodic table, forming various minerals such as oxides, sulfides, carbonates, silicates, hydroxides, chlorides, sulfates, and phosphates. Copper resources are mainly detected in the form of sulfides and oxides, such as chalcopyrite (CuFeS_2), bornite (Cu_5FeS_4), chalcocite (Cu_2S), covellite (Cu(OH)_2), malachite

(Cu(OH)_2), and azurite ($\text{Cu}_3(\text{CO}_3)_2(\text{OH})_2$) (Habashi, 1999; Azizi et al., 2018; Nozhati and Azizi, 2020). More than 80% of the world's primary copper resources exist in the form of sulfides, which are treated by the flotation process and then concentrated by conventional pyrometallurgical methods (Davenport and King, 2002; Hosseinzadeh et al., 2021). Whereas, the oxide resources are mostly processed by hydrometallurgical techniques, including acid leaching followed by solvent extraction and electrowinning (SX-EW) methods. It is accepted that approximately 20% of copper produced worldwide is processed by hydrometallurgical methods (Hosseinzadeh et al., 2021). Recent surveys show that the trend of the copper production industry is increasingly moving towards hydrometallurgical methods and low-grade reserve processing. Ahmed et al. (2016) reported that zinc (95%) and copper (99%) could be efficiently leached from brass slag by H_2SO_4 . Additionally, the leaching kinetics followed a shrinking core model with surface chemical reaction as the rate-controlling step. Tanda et al. (2017) investigated the leaching behaviour of copper oxide minerals in an alkaline glycine liquor and reported that under optimal conditions after 24 hours, nearly 95.0%, 91.0%, 83.8%, and 17.4% of

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copper were recovered from azurite, malachite, cuprite, and chrysocolla, respectively. **Bai et al. (2018)** perused the optimization and the kinetics of copper leaching from a low-grade cuprite ore. Their findings indicated that approximately 92.5% copper was extracted using 150 g/dm³ sulfuric acid at the particle size of 0.125–0.074 mm, reaction temperature of 353 K, and 180 min leaching time. **Nozari and Azizi (2020)** experimentally examined the dissolution kinetics of copper from a mixed oxide-sulfide copper ore in Iran. The results showed that above 88% copper was recovered at optimal values of operating parameters: 17% H₂SO₄ dosage, 11.3 ml/g solid/liquid ratio, 390 rpm pulp mixing rate, 50°C temperature, and 60 minutes dissolution time. **Apua and Madiba (2021)** investigated the leaching kinetics for the extraction of different elements (copper, cobalt, and iron) from a copper oxide ore in H₂SO₄ solution. Their results demonstrated that approximately over 97% copper could be recovered by sulfuric acid at 70°C, pH 1 after 60 min. Also, the mechanism controlling the leaching kinetics was a mixed model including diffusion and chemical reaction. **Tanaydin et al. (2022)** surveyed the leaching behaviour of malachite in HNO₃ solution in two stages. Optimal leaching conditions were determined using 0.5 M nitric acid concentration, 50°C temperature, a solid-to-liquid ratio of 0.004 g/ml, and an agitation speed of 500 rpm. Under optimal conditions, the leaching rate was 99% after 120 minutes of reaction time. **Mohanraj et al. (2022)** studied the characteristics and recovery of copper from a low-grade copper ore using the hydrometallurgy method, reporting that approximately 95% of copper could be recovered using 0.5 M sulfuric acid and 30% H₂O₂ under conditions of 300 rpm stirring speed, 53–63 microns particle size, a liquid-to-solid ratio of 20:2, and a leaching time of 30 minutes (**Mohanraj et al., 2022**). **Yang et al. (2023)** investigated cuprite leaching with ozone as an oxidant in H₂SO₄ solution and its oxidation dissolution process. Leaching test results showed that the leaching efficiency of cuprite promoted by 43.24% with increasing ozone when temperature, H₂SO₄ concentration, and stirring rate were kept constant at 50°C, 0.014 M, and 800 rpm, respectively. **Sun et al. (2023)** examined the improvement of the leaching operations of copper in sulfuric acid solution using oxygen and ultrasound, showing that with ultrasonic application, the copper dissolution rate increased to 7.52 g/h/L, 1.83 times more than without ultrasound (4.109 g/h/L). Following this, **Pan et al. (2024)** studied the process mineralogy and the leaching of Yulong copper oxide ore in Tibet with sulfuric acid and calcium fluoride. Their findings revealed that the dissolution rate of copper promoted to 60.57% (7.34% more than atmospheric pressure leaching) using 50 g/L H₂SO₄ and CaF₂ with the magnitude of 1% of the ore mass at 0.074 mm (85%) particle size, 1:4 liquid/solid ratio, 30°C temperature, and 4 h dissolution time. Recently, **Hu et al. (2025)** investigated the copper extraction from

a low-grade copper oxide ore containing high silicon and proposed an alkali-dissolving desilication method, followed by ammonia leaching for efficient recovery of copper.

In general, the literature shows that sulfuric acid is the most commonly used leaching reagent due to its economic efficiency and higher dissolution rate compared to other leaching agents. Although numerous studies have been conducted on copper recovery from oxide resources, few studies have focused on the leaching of low-grade ores, especially resources containing high silica and their kinetic modelling. The presence of silicate minerals makes the copper ore relatively resistant to the leaching process. **Toro et al. (2021)** reviewed the role of silicates and clay minerals in a copper heap leaching process and demonstrated that these minerals are a rate-limiting parameter in the leaching processes. The effect of silicate and clay minerals on the overall leaching agent consumption may be even greater than the presence of carbonates in ore. Additionally, the presence of silicate and clay compounds in fine particle size may result in the risk of silica gel formation due to the reaction between leaching lixiviant (sulfuric acid) and silicate owing to the easy polymerization of silica in solution. Thus this study was aimed to assess the facility of dissolving a low-grade copper oxide source with high silica content. On the other hand, given that the optimal conditions for copper recovery vary significantly from one ore to another due to the complexity of mineralogy, the important parameters affecting the leaching process of copper are assessed and optimized. Additionally, the leaching kinetics as a powerful tool and one of the significant aspects of separation techniques was scrutinized to deepen understanding of the dissolution process.

2. Material and methods

2.1. Materials

The studied sample (about 150 kg) was collected from the Kuh Khairy mine, Iran. The received sample was then subjected to preparation operations. The sample was firstly crushed using a jaw crusher (Fritsch 01.703, Fritsch, Idar-Oberstein, Germany), and then by a cone crusher to attain particles smaller than 4 mm in diameter. After mixing and dividing the sample into four parts, half of the sample was coded, packaged, and archived, and then the other half was homogenized and ground in a ball mill so that above 90% of particles were smaller than 2 mm in diameter. After that, the sample was remixed and homogenized and then split by means of cones and riffles to obtain a representative sample. Finally, the chemical composition of the representative sample was identified using x-ray fluorescence spectrometer (Philips PW1480 x-ray spectrometer, Netherlands) as shown in **Table 1**. As can be seen in **Table 1**, the studied sample contains high levels of silicate and

clay components, and the copper content in the sample is about 0.57%. The content of Si ($0.467 \times 52.88 = 24.69$) and Fe ($0.7 \times 14.81 = 10.367$) is also found to be 26.69,

and 10.367%, respectively. In addition, the representative sample (less than 2 mm in diameter) was analyzed by an atomic absorption spectrometer (Thermo Elemental's SOLAAR S Series, Waltham, MA, USA), and the copper content was found to be ~0.54%.

Table 1. Chemical compositions of the studied representative sample

Component	Content (%)	Component	Content (%)
Si	24.69	Mn	0.18
Fe	10.37	Zn	0.03
Al	6.80	Na	0.59
Cu	0.57	K	0.66
Ca	2.65	Cr	0.03
Mg	2.56	Ba	0.02
Ti	0.42	Sr	0.02

2.2. Leaching experiments

Leaching tests were conducted on the prepared representative sample in 1 L beakers using a mechanical stirrer with adjustable stirring speeds. For each leaching test, initially, a sulfuric acid solution was prepared at a predetermined concentration. Then, 200 g of the representative sample along with a specified volume of the sulfuric acid solution based on the desired solid percentage was transferred into the beaker. After adjusting the

Table 2. The experimental conditions for performing the leaching tests using RSM-CCD strategy and the determined values of recovery and the content of copper leached

Factor	Factorial levels			Star point $\alpha = 2.0$		
	Low (-)	Center (0)	High (+)	-2	+2	
(A) Acid concentration (kg/t)	70	80	90	60	100	
(B) Solid percent (%)	25	30	35	20	40	
(C) Leaching time (h)	1.5	2	2.5	1	3	
(D) Stirring rate (rpm)	150	250	350	50	450	
Run	A	B	C	D	Copper content leached (mg/L)	Cu leaching efficiency (%)
1	70	25	1.5	150	1050.5	55.29
2	70	25	2.5	350	1205.78	63.46
3	90	35	1.5	150	2113.58	68.86
4	90	35	1.5	350	1973.84	64.31
5	60	30	2	250	1456.86	59.64
6	90	25	2.5	350	1278.9	67.31
7	90	35	2.5	350	2062.71	67.21
8	90	25	1.5	350	1144.33	60.23
9	70	25	2.5	150	1212.54	63.82
10	80	30	1	250	1480.16	60.59
11	80	30	3	250	1676	68.61
12	70	35	2.5	150	2041.85	66.53
13	80	30	2	250	1614.4	66.09
14	80	30	2	70	1581.5	64.74
15	80	40	2	250	2740.77	72.13
16	80	30	2	250	1651.5	67.61
17	70	35	2.5	350	2004.92	65.32
18	70	25	1.5	350	1111.67	58.51
19	100	30	2	250	1671.96	68.44
20	90	25	2.5	150	1262.25	66.43
21	90	35	2.5	150	2165.02	70.54
22	90	25	1.5	150	1169.04	61.53
23	70	35	1.5	150	1890.1	61.58
24	80	30	2	450	1599.61	65.48
25	80	30	2	250	1624.43	66.50
26	70	35	1.5	350	1957.7	63.78
27	80	20	2	250	895.28	62.83

stirring speed, the solution was stirred for a specific duration. After finishing leaching time, the resulting pulp was filtered, and the leach liquor was analyzed by atomic absorption spectrometer to specify the copper concentration. Ultimately, the leaching efficiency of copper (R) for each experiment was calculated utilizing the following formula:

$$R = \frac{C_f \times V}{C_i \times m} \times 100 \quad (1)$$

in which C_f describes the concentration of copper in the leach liquor or PLS (Pregnant Leach Solution) (g/L), V denotes the volume of the leach solution (L), C_i represents the copper content in the solid sample (%), i.e. here C_i was 0.54/100 based on atomic absorption spectrometer (AAS), and m is the weight of the sample (g).

2.3. Experimental design based on RSM-CCD

Modelling and process optimization were accomplished utilizing response surface methodology (RSM) combined with central composite design (CCD) to obtain more accurate information about the influence of effective parameters on the dissolution process, especially interactive effects between the parameters. RSM-CCD is one of the most important and efficient DOE (design of experiments) techniques used to develop, improve, model, and optimize the processes and assess the relative importance of the operating parameters (Montgomery, 2001; Myers and Montgomery, 2002; Bezerra et al., 2008; Motamedizadeh et al., 2021). Thus, according to the RSM-CCD model and Equation 2, a set of 27 experiments ($N=2^{(5-1)} + 2 \times 4 + 3=27$) consisting of 16 partial factorial runs, 8 axial runs ($\alpha = 2$) and 3 repeated experiments at the middle point was designed and conducted. Table 2 shows the opted factors and the operating conditions for performing leaching tests.

$$N = 2^{(n-p)} + 2n + n_c \quad (2)$$

where N is the total number of experiments, n depicts the number of parameters, n_c exhibits the number of central experiments, and P is a fraction of the number of parameters (here $P=1$) (Motamedizadeh et al., 2021).

2.4. Kinetic study

The reaction kinetics was investigated to identify and understand the mechanism of copper leaching process. The reactions occurring between the ore particles and leach liquor during the leaching process are typically heterogeneous and, in such a system, the expression of the overall reaction rate is complicated by the interactions between physical and chemical processes (Habache et al., 2009; Ghasemi and Azizi, 2018). Based on previous studies, the reaction rate between mineral particles and acidic leach solution mainly follows the shrinking core models (SCMs). SCM models are preferred for the kinetic modelling due to their accurate representation for the actual leaching process (especially

closer approximation of real particles in the different situations) compared to other models (Levenspiel, 1999; Nozari and Azizi, 2020; Apua and Madiba, 2021; Houshmand et al., 2024). In heterogeneous SCM models, the reactant particles are assumed to be nonporous, spherical shape, and their size does not change during reaction. Also, the reaction between the mineral particles and the leaching Lixiviant occurs on the outer surface of the particles (Ekmekyapar et al., 2015; Bai et al., 2018). However, these assumptions may not exactly match with the reality. Therefore, SCM models were utilized for kinetic modelling of copper leaching process. In this regard, the diverse SCM models were applied to the experimental data as shown in Equations 7-9.

Surface chemical reaction

$$1 - (1 - X)^{\frac{1}{3}} = k_{sc} \times t \quad (3)$$

Liquid film diffusion

$$1 - (1 - X)^{\frac{2}{3}} = k_{lf} \times t \quad (4)$$

Diffusion through a product layer

$$1 - 3(1 - X)^{\frac{2}{3}} + 2(1 - X) = k_{pl} \times t \quad (5)$$

In the aforementioned equations, k_{sc} , k_{lf} , and k_{pl} are the kinetic rate constants, individually, for surface chemical reaction, diffusion through liquid film, and product layer diffusion model (min^{-1}), X is the conversion fraction of copper recovered and t implies the dissolution time (min). Meanwhile, the process kinetics is commonly controlled by the slowest stage.

3. Results and Discussion

To realize the possibility of copper recovery from the low-grade source with high silica, the present research was conducted in three operational phases. In the first stage, the behaviour and impact of operating variables such as particle size, sulfuric acid concentration, solid percentage (pulp density), pulp stirring speed, leaching temperature, and leaching time were investigated under fixed conditions with OFAT (one factor at a time) method. In the second phase, the possible interactive effects between operating parameters were evaluated, and the process optimization was performed using statistical experimental design and response surface methodology. In the third step, the leaching kinetics was studied to identify the dissolution mechanism and gain further insight of the copper leaching process.

3.1. Impact of major parameters on copper leaching

Figures 1, and 4-7 display the impact of operating parameters including particle size, sulfuric acid dose,

solid percentage (pulp density), stirring rate, and leaching time on the leaching efficiency and the content of copper in PLS solution. These factors were assessed on the basis of OFAT method when one parameter was changed and the other parameters were fixed at a constant value. The following observations can be made on the impact of the parameters on the leaching yield of copper.

The optimal and desirable particle size has a great impact on the dissolution amount of copper. As can be seen in **Figure 1**, the 0-500 μm particle size has a higher copper content and extraction efficiency. Interestingly, contrary to the expectation that copper dissolution should reduce with increasing particle size, it is observed that for particle sizes of 0–2 mm the dissolution rate increases. It is generally accepted that the leaching rate increases with decreasing particle size due to an improved liberation degree of the mineral, increased contact surface area, and enhanced permeability of the leaching agent (**Janyasuthiwong et al. 2016; Hao et al. 2022; Tanaydin et al. 2022**). However, this behaviour may vary among the size fractions of a given ore, depending on the mineralogy, mineral associations, and the dissemination of various species (**Hansen et al., 2005**).

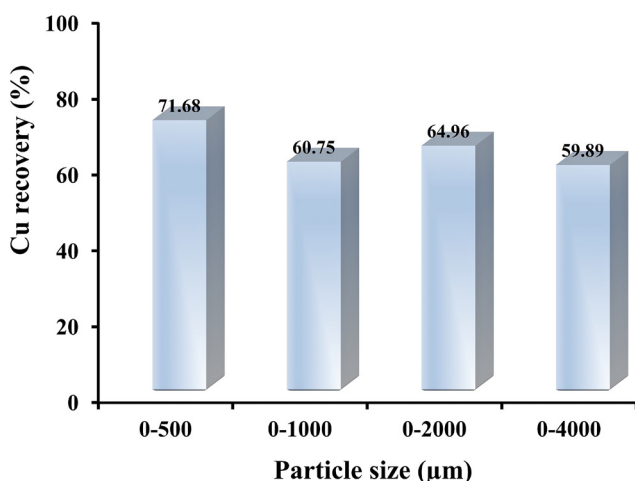
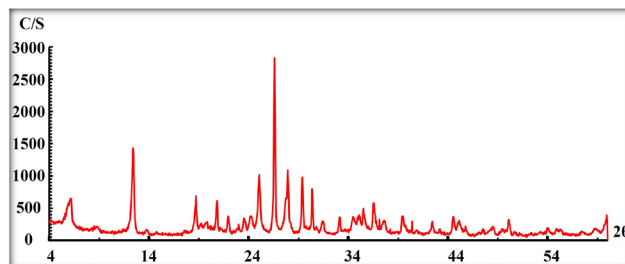


Figure 1. The impact of particle size on the leaching efficiency under conditions: 25°C temperature, 70 kg/t acid concentration, 30% solid percentage, 150 rpm stirring speed, and 90 min leaching time

According to XRF and XRD analyses (see **Figure 2**), the studied sample significantly contains silicate and clay minerals, which affect the copper dissolution performance due to their different and complex structures. The presence of clay and silicate compounds in the sample can influence the consumption of acid (leaching agent), and this is more severe in the fine particles. For instance, some silicate minerals (like mica, and clay) can consume acid generated by the oxidation phenomenon, and/or can quickly adsorb acid (such as kaolinite, and montmorillonite) (**Toro et al., 2021**). In addition, clays and silicates have negative effects on most mineral pro-

Test Method: LW06



Sample:	Major Phase(s)	Minor Phase(s)
01	Chlorite (29-0701)	Muscovite-Illite (26-0911)
Az: 943-1	(Mg,Fe) ₆ (Si,Al) ₄ O ₁₀ (OH) ₈	KAl ₂ Si ₃ AlO ₁₀ (OH) ₂
kV = 40	Quartz (33-1161)	Hematite (33-0664)
mA = 30	SiO ₂	Fe ₂ O ₃
Ka. = Cu	Albite (09-0466)	Orthoclase (31-0966)
Fil. = Ni	NaAlSi ₃ O ₈	KAlSi ₃ O ₈
	Calcite (05-0586)	
	CaCO ₃	

Figure 2. X-ray diffraction (XRD) spectra of the studied sample

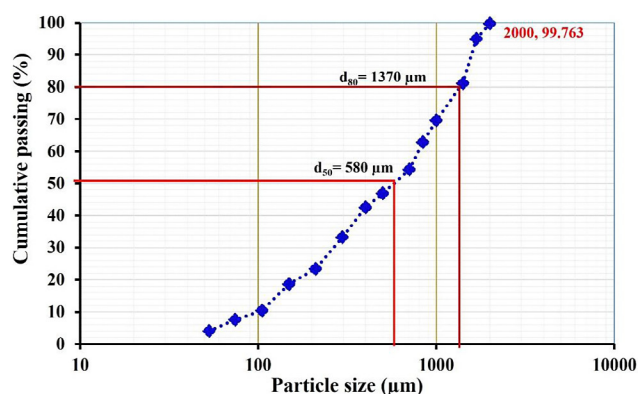


Figure 3. Particle size distribution for the investigated 0-2 mm sample

cessing units owing to their adverse impacts on the pulp rheology and cause many problems in the filtration stage (**Luo et al., 2024**). Thus, regarding the presence of clay and silicate compounds in the sample, as well as the high fines (which creates filtration problems) produced in the smaller size fraction, the fraction of 0-2000 μm was selected for further study. The particle size analysis for the fraction of 0-2000 μm is given in **Figure 3**.

The nature of the leaching operations is based on the penetration of the lixiviant into the very small pores in the ore. Theoretically, an increasing concentration of lixiviant will increase the reaction rate. However, in leaching operations, especially acid leaching, an excessive increase in the concentration of the leaching agent, in addition to the non-optimal consumption of acid, leads to the dissolution of gangue compounds into the PLS solution. **Figure 4** indicates the impact of acid dosage ranging from 50 to 130 kg/t on the leaching performance. It can be seen that the recovery rate and the value

of leached copper increase with an increasing acid concentration from 50 to 80 kg/t, but a further increase in acid concentration has little effect on copper extraction efficiency and remains almost constant. This trend can be attributed to the change in leaching mechanism due to the change in acid concentration, and more importantly, due to the mineralogical composition of the studied sample (presence of silicate and clay compounds in the sample) which increases acid consumption. Thus, considering the copper content of about 1650 ppm in PLS liquor and also from an economic point of view, a concentration of 80 kg/t of acid was selected to continue the investigation.

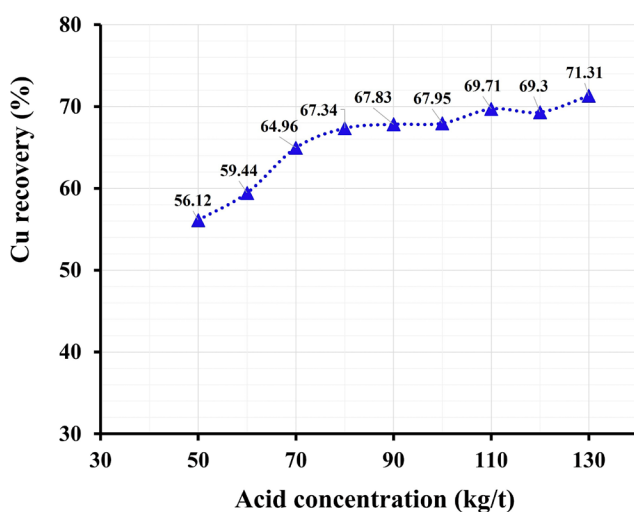


Figure 4. The impact of acid consumption on the leaching efficiency under conditions: 25°C temperature, 0–2000 µm particle size, 30% solid percentage, 150 rpm stirring speed, and 90 min leaching time

The pulp density (solid percentage) depends on the leaching system and its role in the tank leaching is very important. Therefore, six leaching tests were conducted to determine the effect of pulp solids percentage at the values of 20, 25, 30, 35, 40, and 45% under constant operating conditions including ambient temperature, 0–2 mm particle size, pulp mixing rate of 150 rpm, sulfuric acid concentration of 80 kg/t, and leaching time of 90 min. The behaviour of solid percentage on the extraction rate from the studied sample is shown in **Figure 5**. It is observed that as the solid percentage increases from 20 to 40%, the amount of leached copper increases sharply, and with further increase, the leaching efficiency decreases due to lack of proper mixing. The reason for the increase in recovery with increasing solid percentage can be attributed to the better performance of the agitation process due to more and more effective collision of particles in the solution. Therefore, low solid percentages reduce the dissolution efficiency and require a large amount of acid for a small amount of the sample and, on the other hand an excessive increase in the solid percent creates problems for slurry mixing, pumping, and filtration operations. Hence, a solid percentage of 35 (given

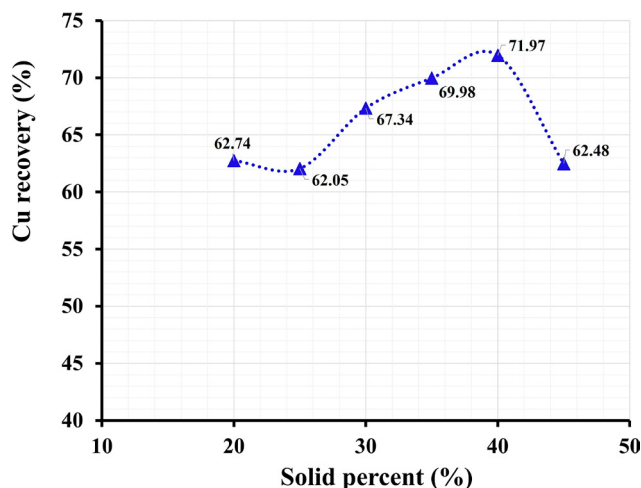


Figure 5. The impact of solid content on the leaching efficiency under conditions: 25°C temperature, 80 kg/t acid concentration, 0–2000 µm particle size, 150 rpm stirring speed, and 90 min leaching time

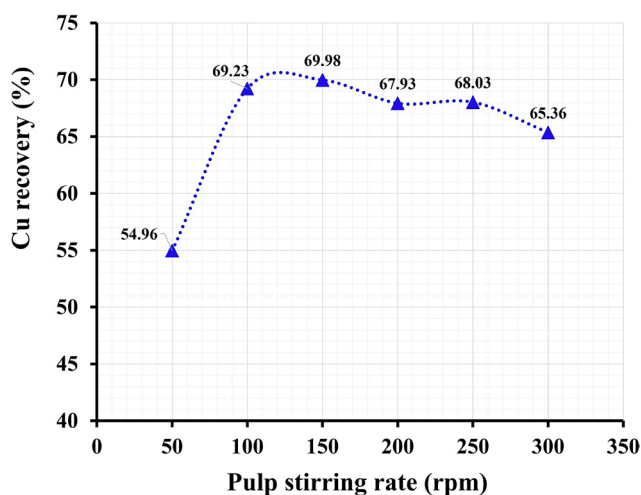


Figure 6. The impact of pulp stirring rate on the leaching efficiency under conditions: 25°C temperature, 80 kg/t acid concentration, 0–2000 mm particle size, 35% solid percentage, and 90 min leaching time

the recovery rate of about 70% and the copper content of about 2.15 g/L in PLS) was selected for further studies.

Figure 6 shows the leaching recovery as a function of pulp agitation rate. As can be considered, the highest amount of copper is leached in a stirring rate of 150 rpm, and high mixing speeds have a negative impact on the dissolution efficiency. In general, the pulp agitation increases the dissolution rate at the beginning of the operation, but has little effect as the operation continues. In many systems, it is sufficient to have an agitation speed that suspends the solid particles and prevents them from settling. Therefore, based on the results received and observations, a stirring speed of 150 rpm was used to continue the study.

Each leaching project has an economic time depending on the concentration of the dissolving agent, particle size, and leaching method, and if the leaching time is

longer than this desired time, the project tends to become uneconomical. Thus, the effect of contact time of the received sample with the leaching agent (sulfuric acid) was studied from 15 to 180 minutes as shown in **Figure 7**. As expected, the extraction efficiency (copper recovery and copper content in the PLS solution) increased with leaching time rising and reached its maximum value (about 71% with a concentration of 2192 mg/L) after 2 h. Thereafter, the copper dissolution rate increased slightly with increasing time, and the changes in the copper leaching rate were very small.

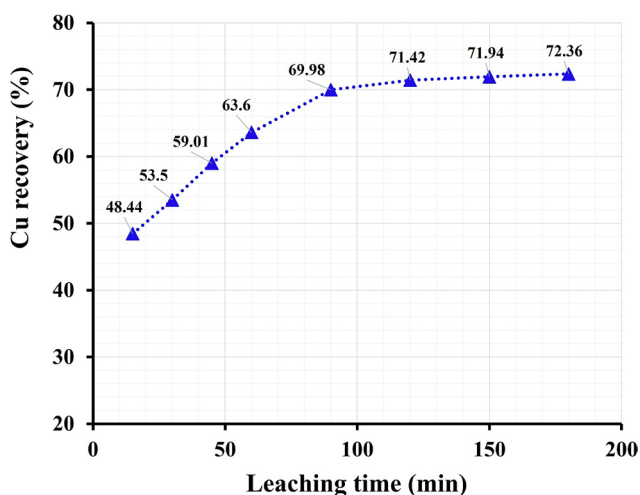


Figure 7. The impact of leaching time on the leaching efficiency under conditions: 25°C temperature, 80 kg/t acid concentration, 0–2000 µm particle size, 35% solid percentage, and 150 rpm pulp stirring rate

3.2. Modelling and process optimization

3.2.1. Modelling and interactive effects between the operating parameters

After designing and carrying out the tests based on RSM-CCD strategy (see **Table 2**), the experimental data was statistically analyzed in the Design Expert software environment. In this regards, a quadratic polynomial model based on **Equation (6)** (Bezerra et al., 2008) was fitted to the data.

$$Y = \theta_0 + \sum_{i=1}^k \theta_i x_i + \sum_{i=1}^k \theta_{ii} x_i^2 + \sum_{1 \leq i < j \leq k} \theta_{ij} x_i x_j + \varepsilon \quad (6)$$

where Y denotes the process responses (leaching recovery of copper (%) or copper concentration leached (mg/L), k is the number of parameters, θ_0 depicts a constant term, θ_i is the coefficients of the linear terms, x_i and x_j display the parameters, θ_{ii} represents the coefficients of the quadratic terms, θ_{ij} is the coefficients of the interactive terms among the parameters, and ε implies the residual error (the difference between the measured and the predicted values).

Ultimately, after removing the very negligible terms (p -value > 0.05), the final regression models developed

to approximate the leaching rate (R_{Cu}) and the content of copper leached were obtained according to **Equations 7** and **8**, respectively.

$$R_{Cu} = +66.69 + 1.91 \times A + 2.09 \times B + 2.19 \times C - 0.12 \times D - 0.76 \times A \times D - 0.9 \times B \times C - 0.58 \times B \times D - 0.88 \times A^2 - 0.74 \times C^2 - 0.61 \times D^2 \quad (7)$$

$$C_{Cu} = +1569.52 + 46.87 \times A + 436.07 \times B + 50.62 \times C - 5.37 \times D + 52.51 \times B^2 \quad (8)$$

In the above **Equations**, A , B , C , and D represent the sulfuric acid concentration, solid percentage, leaching time, and pulp agitation rate, respectively. Also, all parameters are based on the coded units. The coded amounts were used for simplifying the calculations and uniform comparison and determined according to the following formula (Motamedizadeh et al., 2021).

$$X_i = \frac{x_i - x_0}{\Delta x}, \quad i = 1, 2, 3, \dots \quad (9)$$

in which X_i is the value of coded parameter, x_i is the value of real parameter, x_0 is the magnitude of real parameter at the centre point, and (Δx) is the step change in the actual parameter.

The final quadratic polynomial models developed were statistically evaluated employing analysis of variance (ANOVA) at a 95% confidence level (p -values less than 0.05) as presented in **Tables 3** and **4**.

The high F -values and low probability levels ($p < 0.05$) in the ANOVA tables indicate the model proposed and the parameter is statistically significant. As can be seen, the developed models show an excellent relationship between the operational parameters and the process responses with p -values less than 0.0001, and so they can be used to predict the response variable. Meanwhile, the difference between (R^2) and adjusted (R^2) of 0.0379 (The leaching efficiency) and 0.0026 (the copper content leached) indicate a very good correlation between experimental data and the fitted model. Additionally, the larger F -values and smaller p -values indicate which the parameter has a greater influence than the other parameters. It is observed from **Table 3** that the leaching time, solid percentage and acid concentration have the greatest impact on the copper leaching recovery, respectively, while pulp mixing rate does not have much effect on the efficiency of process. It is also seen that the leaching efficiency of copper highly depends on the interactive impacts among the parameters. The significance degree of the parameters on the copper concentration leached is found to be in the order of the quadratic effect of solid percent (B^2) > the linear effect of solid percent (B) > the linear effect of leaching time (C) > the linear effect of acid concentration (A) > the linear effect of stirring rate (D). The perturbation graph (see **Figure 8**) showing the effect of all operating parameters on the process re-

Table 3. The results of ANOVA of the model proposed to approximate the leaching rates of copper and determine the relative importance of the influential operating parameters

Source	Sum of squares	Degree of freedom (DF)	Mean Square	F Value	p value	Remark
Model	362.49	10	35.25	24.77	< 0.0001	significant
Acid concentration (A)	87.13	1	87.13	59.54	< 0.0001	
Solid percentage (B)	104.79	1	104.79	71.61	< 0.0001	
Leaching time (C)	115.15	1	115.15	78.69	< 0.0001	
Stirring speed (D)	0.37	1	0.37	0.25	0.6231	
AD	9.23	1	9.23	6.3	0.0232	
BC	12.94	1	12.94	8.84	0.009	
BD	5.44	1	5.44	3.72	0.0718	
A2	18.41	1	18.41	12.58	0.0027	
C2	13	1	13	8.88	0.0088	
D2	8.88	1	8.88	6.07	0.0255	
Residual	23.41	16	1.46			
Lack of Fit	22.18	14	1.58	2.56	0.3161	not significant
Pure Error	1.24	2	0.62			
Cor. Total	385.9	26				
Summary of statistical analysis of model						
R ²	0.9393					
Adjusted R ²	0.9014					
Adeq Precision	19.4989					

Table 4. The results of ANOVA of the model proposed to approximate the copper concentration leached and determine the relative importance of the influential operating parameters

Source	Sum of squares	Degree of freedom (DF)	Mean Square	F Value	p value	Remark
Model	4.75×106	5	9.5×105	376.91	< 0.0001	significant
Acid concentration (A)	52716.56	1	52716.56	20.91	< 0.0002	
Solid percentage (B)	4.56×106	1	4.56×106	1809.81	< 0.0001	
Leaching time (C)	61498.24	1	61498.24	24.39	< 0.0001	
Stirring speed (D)	691.33	1	691.33	0.27	0.606	
B2	73532.32	1	73532.32	29016	< 0.0001	
Residual	52955.46	21	2521.69			
Lack of Fit	52218.86	19	2748.36	7.46	0.1246	not significant
Pure Error	736.6	2	368.3			
Cor. Total	4.81×106	26				
Summary of statistical analysis of model						
R ²	0.989					
Adjusted R ²	0.9864					
Adeq Precision	73.685					

sponses (the leaching rate and the content of copper leached) also confirms these results. This graph displays the impacts of all the parameters at a special point in the design space. The sharp slope or curve in graph implies the sensitivity of the leaching rate and the content of copper leached to that parameter. As observed in **Figure 3**, all parameters except pulp stirring rate significantly affect the process responses, confirming the results derived from ANOVA tables.

In addition to the above, the three-dimensional (3D) response surface plots were utilized to attain a better understanding of the main and synergistic effects of parameters on the leaching recovery and content of copper leached, as shown in **Figures 7-10**. These graphs are obtained based on the change in two variables when other variables are fixed at their middle level, and display the nature and magnitude of possible interactions between various parameters.

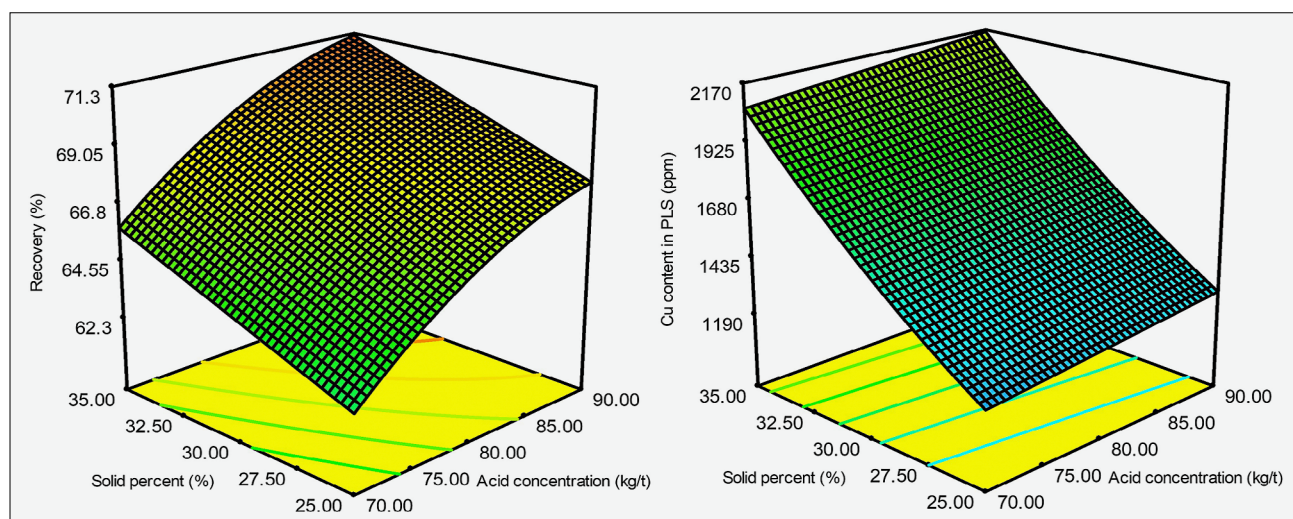
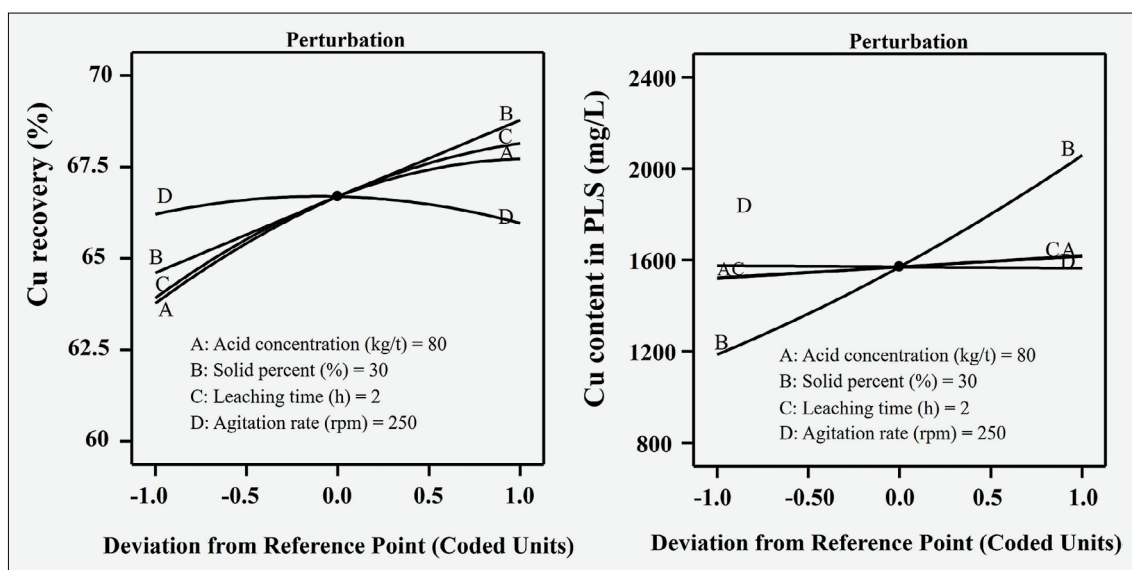


Figure 9 shows the simultaneous effect of acid concentration and solid percentage on the recovery and concentration of copper in the PLS solution. As can be seen, copper recovery is a quadratic function of acid concentration and has a linear relationship with solid percentage. It is obvious that at low and high solids percentages, copper recovery enhances with an increase in the acid concentration, and this increase is more intense at lower solid percentages. The highest copper recovery is observed at a solid percentage of 35 and an acid concentration of 90 kg/t, which, under these conditions, reaches more than 71% with a copper concentration of ~2170 mg/L in PLS solution. It is noteworthy that under these conditions, the mixing rate was 150 rpm and the leaching time was 2.5 hours. Additionally, it was found that the effect of acid concentration on copper recovery is greater

and the effect of solid percentage on the leached copper concentration is higher. At the lower solid percentage, the acid has more surface area and effective contact to react with copper-bearing minerals, leading to higher leaching rate, but only if the acid is not overly concentrated, as it may cause excessive dissolution of other unwanted elements. While, at high solid percentages, the leaching pulp becomes denser, reducing the effectiveness of acid due to less surface area exposure. In this case, the higher acid concentrations may be required to compensate for the reduced contact, though this can increase costs and side reactions. Wang et al. (2019) attributed the increase in copper dissolution with increasing acid concentration to the enhanced activity of hydrogen ions (H^+). They also reported that the upward trend in copper dissolution rate decreases with a further increase.

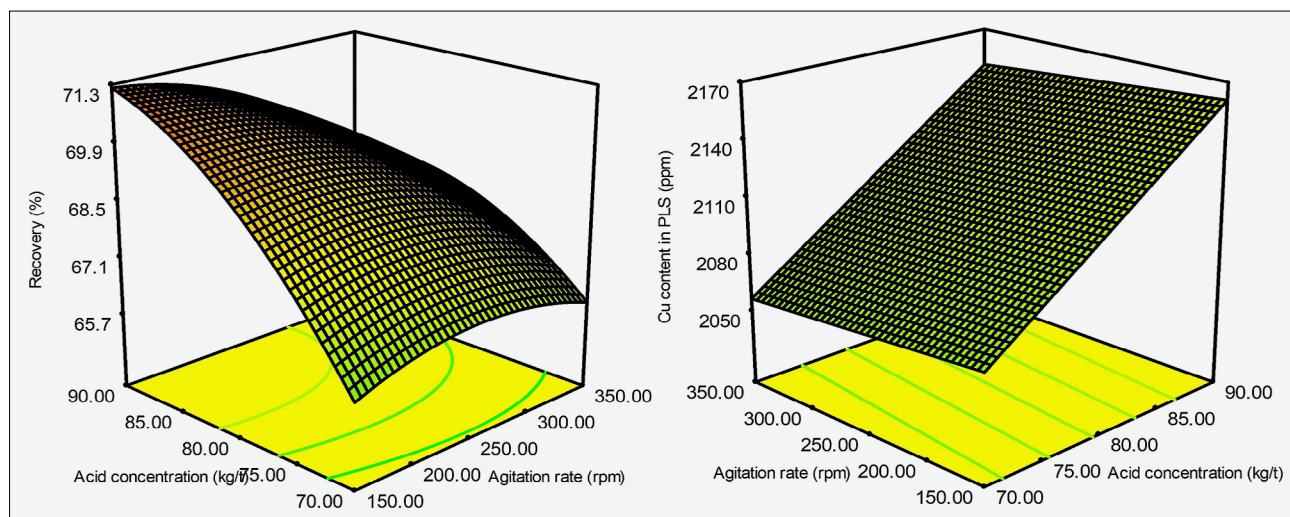


Figure 10. 3D graphs displaying the combined impact of acid concentration and pulp mixing rate on the recovery and concentration of copper leached

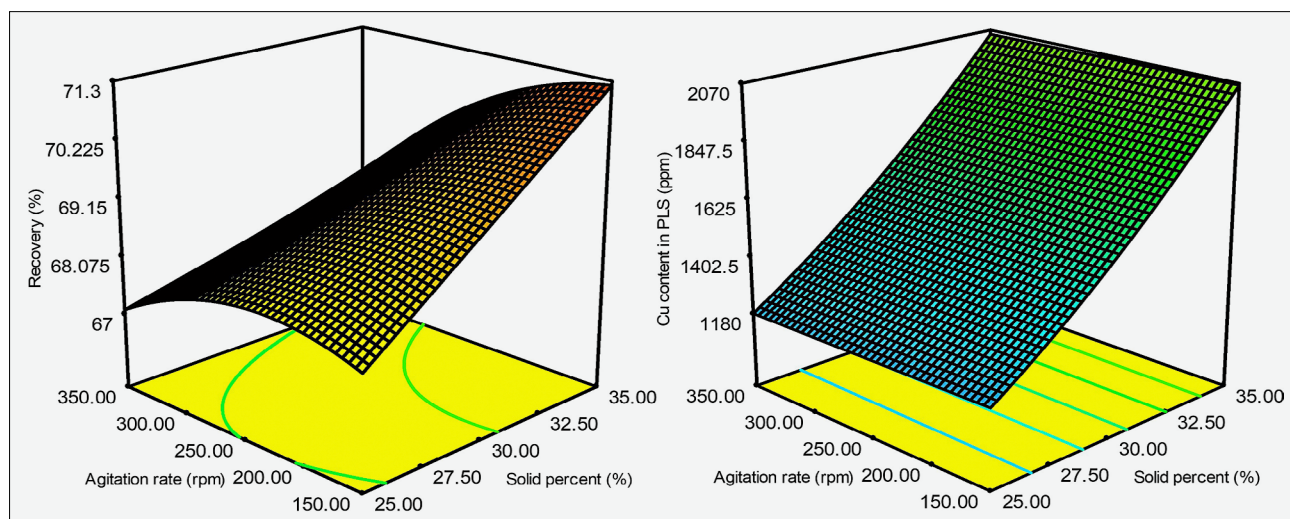


Figure 11. 3D graphs displaying the combined impact of solid percentage and pulp mixing rate on the recovery and concentration of copper leached

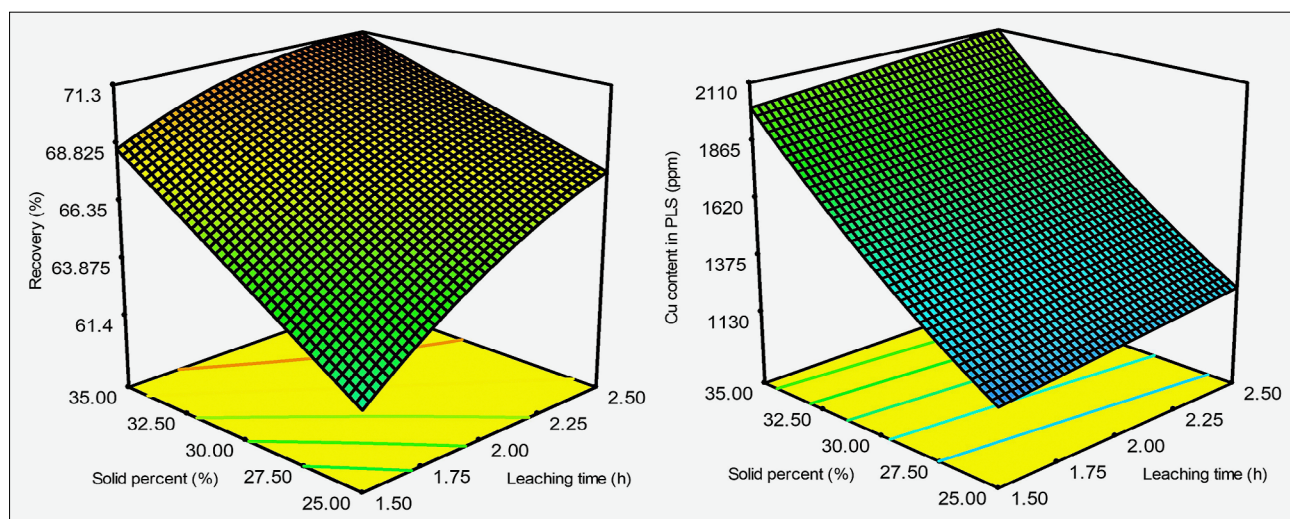


Figure 12. 3D graphs displaying the combined impact of solid percentage and leaching time on the recovery and concentration of copper leached

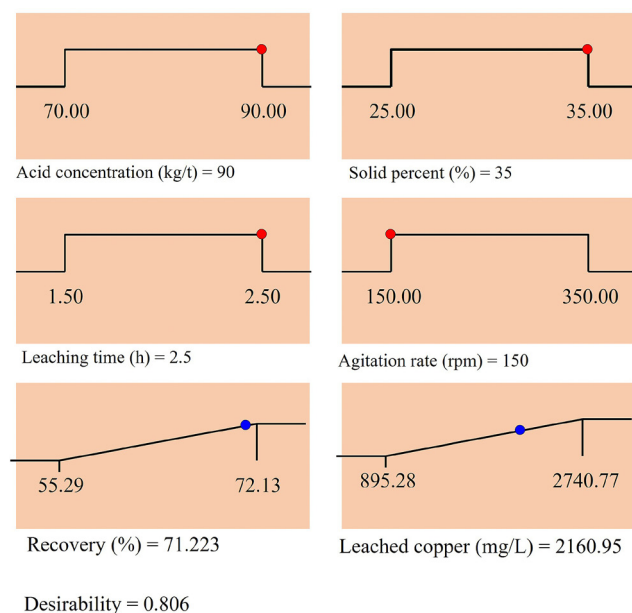


Figure 13. The optimized values of operating parameters to achieve maximum copper dissolution efficiency (recovery and content of copper in PLS solution)

The combined effect of acid concentration and solid percentage with pulp mixing rate are shown in **Figures 10 and 11**, individually. It can be seen that the dissolution rate of copper increases with the pulp mixing speed at the beginning of the operations, and generally the pulp stirring speed had a smaller effect on the dissolution performance compared to the other two parameters, showing that the copper dissolution process is affected by a certain value of pulp mixing rate. Additionally, according to **Figures 10 and 11**, the dissolution efficiency increases greatly by increasing the acid concentration and solid percentage at a lower mixing rate within 2.5 h. At lower mixing rates, high acid concentration compensates for slower mass transfer by offering strong chemical reactivity, leading to increased leaching efficiency. A high solids percentage provides more material for the acid, and increases overall dissolution at low stirring rates. However, an excessive solid percentage will cause the leaching pulp to be denser and reduce the copper recovery. Nevertheless, the combination of these factors can still significantly improve efficiency, even if stirring is not optimal. It is also observed that when the acid concentration and stirring speed are simultaneously low, the recovery decreases. Similar results were found in the work reported by **Maleki et al. (2023)**.

Figure 12 exhibits the interaction between the solid percentage (pulp density) and the contact time of leaching agent with the sample. As can be seen, these two parameters have a synergistic effect in increasing copper leaching efficiency, and this synergistic effect is greater at lower solid percentages, although it reaches its maximum value at 35% solids and a contact time of 2.5 hours. In general, increasing the leaching rate with a decrease in solid/liquid ratio can be due to the greater amount of

acid per unit amount of solid sample and the decrease in mass transfer resistance (**Hosseinzadeh et al., 2021**).

3.2.2. Process optimization

The numerical optimization of leaching process was conducted using Design Expert (DX) software and employing the desirability function procedure. The optimal operating conditions proposed by RSM modelling for achieving the highest leaching efficiency and the leached copper content are illustrated in ramp plots in **Figure 13**. As observed, the optimized values of leaching parameters for -2 mm size fraction as follow: 90 kg/t sulfuric acid concentration, 35% solid content, 150 rpm agitation rate, and leaching time of 2.5 hours. It is found that under these optimal conditions, a PLS solution with a copper concentration of ~2160 mg/L and an efficiency of more than 71% can be achieved. It is noteworthy that in the confirmation experiment conducted under the proposed optimum conditions similar to **Run 21** (see **Table 2**), a recovery of about 70.5% with a copper content of 2165 mg/L in PLS was achieved.

3.3. Leaching kinetic modelling

To model, the left side of **Equations 3-5** was plotted as a function of leaching time for each operational variable, including the concentration of leaching agent, solid percentage (solid/liquid ratio), pulp agitation speed, and leaching temperature. For instance, the results obtained from the temperature parameter are shown in **Figure 14**, in which the slope of the straight lines represents the kinetic rate constant for copper leaching. The values of kinetic constants and correlation coefficients for each parameter are provided in **Table 5**. According to **Figure 14** and **Table 5**, the diffusion equation through a product layer exhibits the best correlation relative to other kinetic equations, demonstrating that this model is the controlling step of the reaction rate.

To better distinguish the reaction mechanism, the activation energy was determined by analyzing the data and applying the Arrhenius equation according to the following equation (**Levenspiel, 1999; Adebayo and Olasehinde, 2015**):

$$k = A \times e^{\frac{-E_a}{R \times T}} \Rightarrow \ln(k) = \ln(A) - \frac{E_a}{R} \cdot \frac{1}{T} \quad (10)$$

where k implies the reaction rate constant (min^{-1}), A illustrates the frequency factor (min^{-1}), E_a denotes the activation energy (J/mol), R depicts the universal gas constant ($8.314 \text{ J} \cdot \text{K}^{-1} \cdot \text{mol}^{-1}$), and T is for the leaching temperature (K).

To measure the activation energy, $\ln(k)$ versus $(1/T)$ for each temperature was plotted, as shown in **Figure 15**. The slope of the straight line in this graph represents $-\frac{E_a}{R}$. Thus, the activation energy of the diffusion model

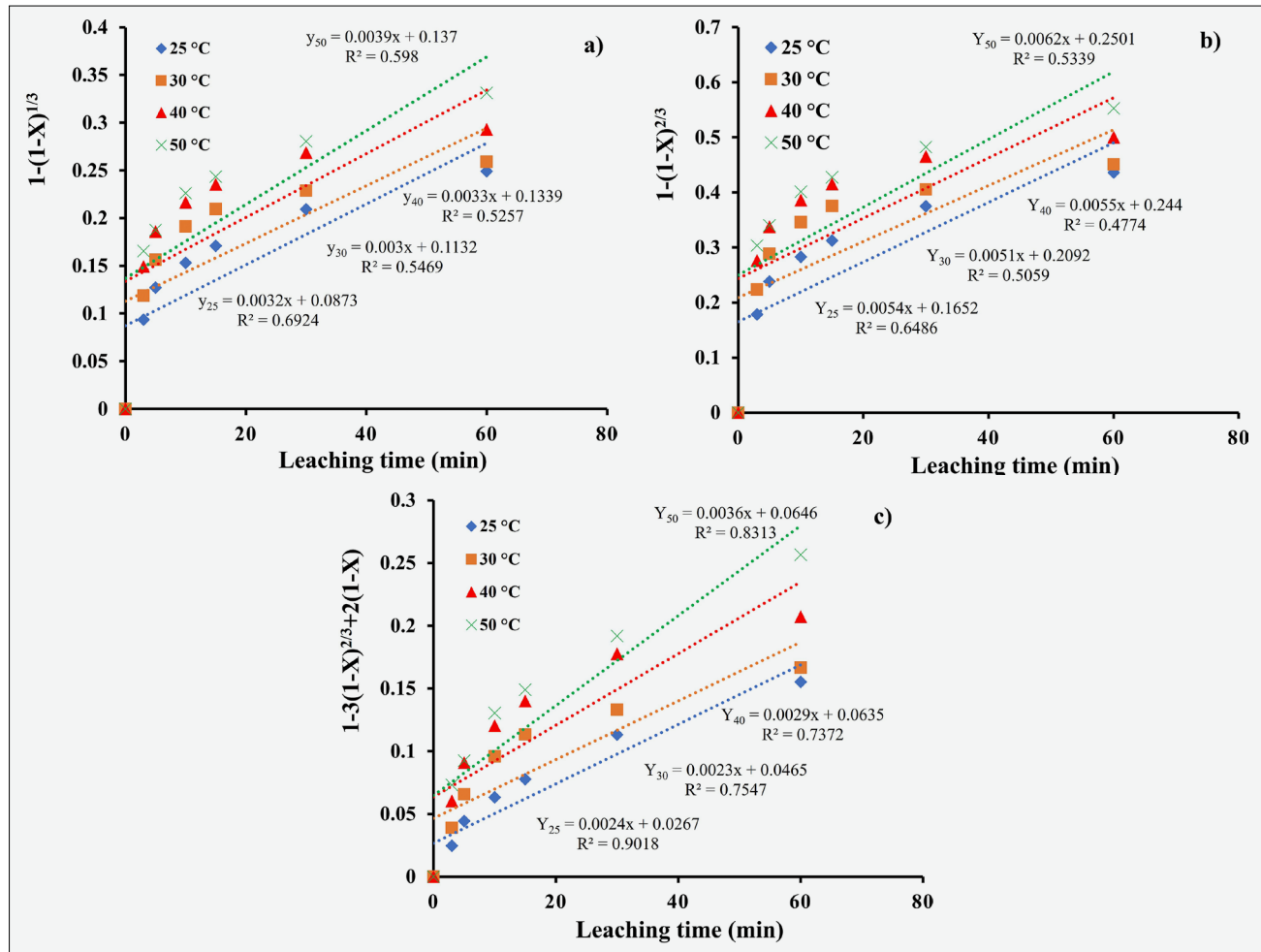


Figure 14. The graph of the SCMs models versus leaching time at different temperatures (25, 30, 40, and 50)

Table 5. The kinetic analysis of the shrinking core models fitted to the experimental data

	Surface chemical reaction		Liquid film diffusion		Diffusion through a product layer	
Factors	k_{sc} (min ⁻¹)	R ²	k_{lf} (min ⁻¹)	R ²	k_{pl} (min ⁻¹)	R ²
Temperature (°C)						
25	0.0032	0.6924	0.0054	0.6486	0.0024	0.9018
30	0.0030	0.5469	0.0051	0.5059	0.0023	0.7547
40	0.0033	0.5257	0.0055	0.4774	0.0029	0.7372
50	0.0039	0.5980	0.0062	0.5339	0.0026	0.7313
Acid concentration (kg/t)						
70	0.0030	0.5574	0.0051	0.5182	0.0023	0.7501
80	0.0031	0.5607	0.0052	0.5168	0.0024	0.7777
90	0.0033	0.5161	0.0054	0.4726	0.0028	0.7060
100	0.0034	0.5320	0.0055	0.4847	0.0027	0.7345
Stirring speed (rpm)						
100	0.0033	0.5655	0.0054	0.5214	0.0027	0.7655
150	0.0031	0.4923	0.0052	0.4515	0.0026	0.6802
250	0.0036	0.5540	0.0059	0.5031	0.0032	0.7541
350	0.0032	0.4980	0.0053	0.4540	0.0027	0.6984
Solid percentage (%)						
25	0.0035	0.6767	0.0059	0.6387	0.0027	0.8441
30	0.0032	0.5538	0.0054	0.5160	0.0025	0.7306
35	0.0036	0.5247	0.0058	0.4814	0.0031	0.6945
40	0.0041	0.5647	0.0065	0.5081	0.0039	0.7508

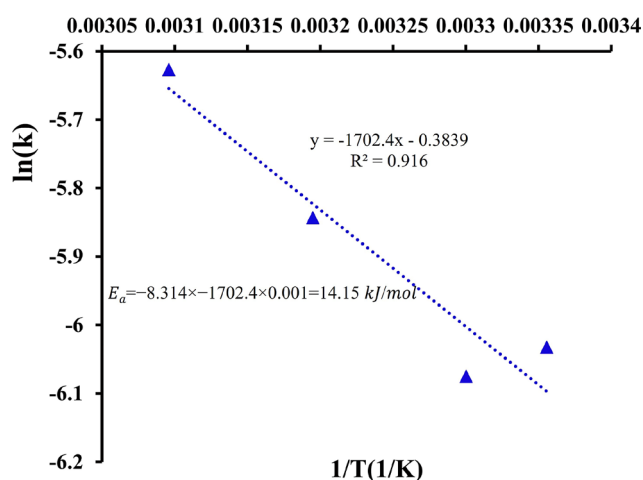


Figure 15. Arrhenius graph for product layer diffusion model to approximate the activation energy

through a product layer was calculated 14.15 kJ/mol ($-\frac{E_a}{R} = -1702.4 \rightarrow E_a = -8.314 \times -1702.4 = 14154 J = 14.15 kJ$). According to **Espiari et al. (2006)** and **Ekmekyapar et al. (2012)**, the values of E_a less than 40 kJ/mol demonstrate that the leaching process is controlled by diffusion phenomena. **Ekmekyapar et al. (2015)** investigated the leaching kinetics of malachite ore in ammonium sulfate solutions and reprotted the diffusion model through the product layer was the rate-controlling step of the dissolution kinetics. **Hosseinzadeh et al. (2021)** performed a similar kinetic study on copper leaching from a low-grade copper oxide source, and reported that the predominant mechanism was a diffusion process with the activation energy of 11.72 kJ/mol. Therefore, by combining **Equations 5** and **10** and ascertaining the influence of parameters on the apparent rate constant given in **Table 5**, the mathematical equation showing the kinetics of copper leaching can be described as follows:

$$1 - 3(1-x)^{\frac{2}{3}} + 2(1-x) = A \times (SA)^s \times (SP)^p \times (SR)^r \times e^{\frac{-E_a}{R \times T}} \times t \quad (11)$$

in which A , SA , SP , and SR represent frequency factor, sulfuric acid concentration, solid percentage, and pulp stirring rate, individually, and the constants s , p , and r depict the reaction order for each the corresponding parameter. The reaction orders (constants) were determined by plotting $\ln(SA)$, $\ln(SP)$, and $\ln(SR)$ against $\ln(kpd)$ and approximating the slope of straight line each curve. The constants s , p , and r were estimated to be 1.3021, 2.1337, and 0.8689, respectively. Ultimately, the ultimate model describing the leaching kinetics of copper was understood as **Equation 12**:

$$1 - 3(1-x)^{\frac{2}{3}} + 2(1-x) = A \times (SA)^{1.3021} \times (SP)^{2.1337} \times (SR)^{0.8689} \times e^{\frac{-14.15 \times 10^3}{8.314 \times T}} \times t \quad (12)$$

4. Conclusions

The presence of silicates and clays in ores creates various problems in the mineral processing industry, such as the formation of gel during sulfuric acid leaching, filtration problems, high consumption of leaching lixiviant (acid), and reduced leaching efficiency, all of which lead to high costs. Thus, the major aim of the present paper was to scrutinize the potential recovery of copper from a low-grade ore consisting of high silica content using tank leaching method. To this end, this study was conducted in three working phases. In the first phase, the behaviour of major parameters consisting of particle size, sulfuric acid concentration, solid percentage (pulp density), stirring rate, and leaching time was assessed with the one factor at a time method. The findings demonstrated that the amount of extracted copper augmented with increasing the acid concentration, leaching time and the solid percent. Meanwhile, 0-2 mm size fraction was a better choice for the tank leaching than other fractions including 0-500 μm , 0-1 mm and 0-4 mm. Also, the amount of acid used for the studied sample was very high, and this was due to the presence of silicate and clay compounds in the sample. Additionally, the stirring speed of pulp had little effect on the leaching process of copper compared to other factors. In the next step, modelling and optimizing the leaching process, and evaluating the interactive effects between the operating parameters were conducted using the statistical design of experiments based on response surface methodology (RSM). The results showed that the effect of leaching time, solid percent, and acid dosage had the greatest efficacy on the dissolution efficiency of copper. In addition, the leaching recovery of copper was remarkably dependent on the interactive impacts among the solid percent and leaching time, and the acid concentration and agitation speed. The influence degree of the operating variables on the copper content leached was distinguished in the order of the quadratic effect of solid percent > the linear effect of solid percent > the linear effect of leaching time > the linear effect of acid concentration > the linear effect of stirring rate. The optimization of leaching process was performed with the desirability function technique and the maximum leaching efficiency (71%) with a PLS solution containing 2160 g/L of copper was obtained at the acid concentration of 90 kg/t, solid percentage of 35, pulp mixing rate of 150 rpm, leaching time of 2.5 h, and size fraction of 0-2 mm. Eventually, the kinetics of copper leaching was analyzed using the shrinking core (SCMs) equations to recognize the mechanism of the process and the controlling step of the reaction rate. It was found that the leaching process was controlled by diffusion model through the product layer with the activation energy of 14.15 kJ/mol. Finally, a kinetic mathematical equation depicting the leaching process of copper from the desired copper oxide ore was proposed.

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SAŽETAK

Istraživanje potencijalne uporabe bakra iz niskokvalitetnih izvora koji sadržavaju visok sadržaj silicijeva dioksida

Cilj ovoga istraživanja bio je eksperimentalno ispitati izluživanje bakra iz rude niske kvalitete koja sadržava 0,57 % bakra, 24,69 % silicija i 10,367 % željeza. Prvo je ispitano ponašanje važnih varijabli koje utječu na proces otapanja korištenjem metode jedan faktor u vremenu (OFAT) i modeliranja odzivne površine (RSM), zatim je detaljno ispitana kinetika izluživanja kako bi se identificiralo naredno djelovanje izluživanja. Rezultati dobiveni OFAT metodom pokazali su da je veličina čestica od 0 do 2 mm bolja opcija za izluživanje u spremniku u odnosu na druge frakcije. Prisutnost glinenih spojeva u rudi dovela je do velikoga povećanja potrošnje kiseline. Brzina izluživanja poboljšala se s povećanjem postotka krutih tvari s 20 na 40 %, a zatim se smanjila s daljnjim povećanjem zbog nedostatka pravilnoga miješanja. Osim toga, brzina miješanja pulpe imala je mali utjecaj na otapanje bakra potvrđujući da brzina miješanja pulpe treba biti na razini koja suspendira čestice i sprječava njihovo taloženje. Zatim je s RSM-om obavljeno modeliranje i optimizacija procesa te određivanje moguće interakcije između radnih parametara. Rezultati su pokazali da je izluživanje bakra znatno ovisilo o interakciji između radnih parametara. Optimizirani parametri bili su koncentracija kiseline od 90 kg/t, sadržaj krutih tvari od 35 %, brzina miješanja pulpe od 150 o/min, vrijeme izluživanja od 2,5 h i raspon veličine čestica od 0 do 2 mm. Kinetička analiza pokazala je da je difuzijski model u proizvodnome nizu s energijom aktivacije od 14,15 kJ/mol mjera koja kontrolira brzinu reakcije, te je na temelju toga predložen novi model koji opisuje kinetiku otapanja bakra.

Ključne riječi:

bakrov oksid, izluživanje u spremniku, učinkovitost ekstrakcije, optimizacija, kinetika otapanja

Author's contribution

Milad Abbassi (M.Sc. student): conceptualization, investigation, data curation, formal analysis, methodology, software, and writing – original draft. **Asghar Azizi** (Associate professor): conceptualization, funding acquisition, investigation, methodology, project administration, software, supervision, validation, visualization, writing – original draft and writing – review & editing.

All authors have read and agreed to the published version of the manuscript.