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Independent optimization of oxide and sulfide copper recoveries in mixed copper ore flotation using Box-Behnken response surface methodology

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ABSTRACT

Mixed oxide/sulfide copper ores represent an important but challenging resource for copper production due to the differing flotation behaviors of oxide and sulfide minerals. Conventional studies have primarily focused on overall copper recovery, often overlooking the distinct responses of each copper type. In this study, the Box-Behnken response surface methodology (RSM) was employed to systematically investigate and optimize the effects of collector (potassium amyl xanthate) dosage (100–150 g/t), pulp pH (10–11), and sodium sulfide dosage (1000–2000 g/t) on the recoveries of oxide copper, sulfide copper, and total copper from a low-grade mixed oxide/sulfide copper ore (0.86% total Cu, 66.83% oxidation ratio). Detailed mineralogical characterization using X-ray diffraction (XRD), scanning electron microscopy-energy dispersive spectroscopy (SEM-EDS), electron probe micro-analyzer (EPMA), and size-by-size analysis revealed that the primary copper minerals are chalcocite, bornite, and malachite, predominantly associated with silicate- and clay-rich gangue. Quadratic predictive models were developed for each response, demonstrating high statistical adequacy as confirmed by residual diagnostics and analysis of variance (ANOVA). Pulp pH exerted the strongest influence, with higher values favoring oxide copper flotation and lower values benefiting sulfide copper recovery. Optimal conditions yielded predicted recoveries of 76.49% for oxide copper (100 g/t collector, pH 11.0, 1000 g/t Na_2S), 99.99% for sulfide copper (100 g/t collector, pH 10.0, 1252 g/t Na_2S), and 81.40% for total copper (100 g/t collector, pH 11.0, 1707 g/t Na_2S). The results highlight the necessity of separate optimization strategies for oxide and sulfide fractions in mixed ores and provide practical predictive tools for improving metallurgical performance. This work contributes to a more precise understanding of sulfidization-flotation dynamics and provides a useful basis and guiding framework for the processing of similar mixed copper ores.

KEYWORDS

Mixed oxide-sulfide copper ore, sulfidization-flotation, Box-Behnken design, response surface methodology, optimization, oxide and sulfide copper recovery

I. INTRODUCTION

Copper is one of the most important base metals, widely used in electrical, electronic, construction, and industrial applications. With the progressive depletion of high-grade sulfide copper ores, the exploitation of mixed oxide/sulfide copper deposits has become increasingly important. However, the processing of such ores presents significant metallurgical challenges. Acid leaching, the conventional method for treating oxide copper ores, often suffers from high acid consumption and low dissolution efficiency when applied to mixed ores, particularly due to the poor solubility of copper sulfide minerals under typical leaching conditions (Ghorbani et al., 2016; Li et al., 2025). Consequently, alternative beneficiation routes are required for effective recovery from mixed oxide/sulfide copper ores.

The sulfidization-flotation process has emerged as a promising technology for the beneficiation of oxide and mixed copper ores (Azizi et al., 2020; Jena et al., 2022). This approach involves converting the surfaces of oxide minerals into sulfide-like species using sulfidizing agents (commonly sodium sulfide or sodium hydrosulfide), thereby enabling their flotation with conventional sulfide collectors such as xanthates. The process was first industrially applied to oxide Pb-Zn ores in Australia and has since been investigated for various copper-bearing materials. Recent comprehensive reviews have highlighted various sulfidation reconstruction methods, including surface sulfidation, mechanochemical

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sulfidation, and sulfidation roasting, to enhance the floatability of copper oxide minerals such as malachite, chrysocolla, and cuprite (Wei et al., 2026).

Several studies have explored the sulfidization-flotation of copper ores. Newell et al. (2007) investigated the sulfidization and flotation of oxidized chalcopyrite, pyrrhotite, and pentlandite, demonstrating that optimal sulfidization occurs at a potential of approximately –650 mV, with significantly higher flotation recovery for chalcopyrite than for the other minerals. Cao et al. (2009) achieved a concentrate grading 19.01% Cu with 35.02% recovery from a combined oxide-sulfide ore using butyl xanthate and sodium sulfide.

Further contributions include the work of Phetla and Muzenda (2010), who produced a concentrate containing 13% Cu (after desliming) from a low-grade oxide copper-cobalt ore through multi-stage high-temperature sulfidization, attaining 48% Cu and 50% Co recoveries.

Wen et al. (2012) reported a concentrate grading 21.65% Cu with 76% recovery in closed-circuit tests on a refractory oxide copper concentrate using a combination of ethylenediamine phosphate, ammonium bicarbonate, sodium sulfide, and isoamyl xanthate. Similarly, Ge et al. (2013) achieved 18.06% Cu at 80.01% recovery from an ore containing 0.77% Cu (45.5% oxidation ratio) using optimized dosages of butyl xanthate, pine oil, sodium sulfide, and lime.

Xiong and Zheng (2013) determined the stability constants of sulfidization reactions for key oxide minerals and found that cuprite is considerably more difficult to sulfidize than malachite and chrysocolla. Mao et al. (2014) obtained a concentrate assaying 3.93% Cu (64.36% recovery) from a refractory oxide ore in rougher flotation and improved the results to 10.85% Cu at 57.41% recovery in a closed-circuit configuration. More recent studies have employed response surface methodology to optimize overall copper recovery in sulfidized mixed ores. For instance, Azizi et al. (2020) applied RSM to investigate the simultaneous effects of collector, depressant, frother, pH, and agitation rate on the rougher flotation of a sulfidized mixed copper ore, achieving a maximum recovery of 92.75%. Maleki et al. (2023) also used sequential sulfidization and RSM to optimize flotation of a high-grade oxidized copper ore, obtaining 90.51% recovery.

In a previous study by the present authors (Behnamfard et al., 2026), the influence of operational parameters on the performance of the sulfidization-flotation process for a mixed oxide/sulfide copper ore was investigated using a one-factor-at-a-time approach. That work identified collector dosage, activator dosage, and pulp pH as the most influential parameters affecting process performance.

Despite these valuable contributions and recent advances in sulfidation techniques, previous research has primarily focused on overall copper recovery and concentrate grade. To date, no published study has separately optimized the flotation recovery of oxide copper, sulfide copper, and total copper in a mixed oxide/sulfide ore using Response Surface Methodology (RSM), specifically the Box-Behnken design. Such an approach is essential because oxide and sulfide copper minerals exhibit different surface chemistries and respond differently to sulfidization conditions, collectors, and flotation parameters. Independent optimization of these three responses can provide deeper insight into process behavior, reveal the complex interactions affecting each copper type, and lead to more efficient flowsheet design.

The present study therefore aims to optimize the sulfidization-flotation process for a mixed oxide/sulfide copper ore using the Box-Behnken Response Surface Methodology. The effects of key operational variables on the individual recoveries of oxide copper, sulfide copper, and total copper are systematically investigated, and predictive mathematical models are developed. This work addresses a critical research gap and is expected to contribute to a more precise understanding and improved metallurgical performance in the processing of complex mixed copper ores.

II. Materials and Methods

A. Sampling and Sample Preparation

The mixed oxide/sulfide copper ore was obtained from the active mining operation. Run-of-mine ore with a particle size distribution of 0–300 mm was transported by truck to the plant feed stockpile and subsequently fed into the primary crushing circuit. Primary crushing was performed using a jaw crusher. The crusher product was then discharged onto a double-deck vibrating screen with aperture sizes of 25 mm (upper deck) and 10 mm (lower deck). The undersize fraction (–10 mm) was conveyed directly to the fine ore bin, while the oversize fraction was fed to a hydrocone crusher operating in closed circuit with the screen. The final crushed product (–10 mm) was stored in the fine ore bin and served as feed to the grinding-flotation circuit.

For the preparation of representative samples for laboratory flotation experiments, samples of approximately 0.5 kg were collected from the conveyor belt feeding the grinding-flotation circuit at one-hour intervals over several consecutive days. These samples were thoroughly mixed to form a composite sample, which was then dried at ambient temperature. The dried sample was homogenized and divided into representative sub-samples of 350 g using a riffle splitter. The prepared subsamples were used for flotation experiments as well as characterization tests.

B. Analytical Methods

The mineralogical composition of the ore sample was determined using X-ray diffraction (XRD) analysis on a PHILIPS X'Pert Pro diffractometer (Netherlands).

Total copper grade in samples was measured by complete digestion of the samples in aqua regia, followed by determination of copper concentration in the resulting solution using a Varian SpectrAA 880Z Atomic Absorption Spectrometer (AAS, Australia). Oxide copper grade was determined by selective leaching of the samples with dilute sulfuric acid solution, after which the dissolved copper was quantified using the same atomic absorption spectrometer. Sulfide copper grade was then calculated by subtracting the oxide copper grade from the total copper grade.

For more precise quantitative chemical analysis of individual mineral phases and fine inclusions, an Electron Probe Micro-Analyzer (EPMA, SX100, Cameca, France) was employed at the Mineral Processing Research Center. Polished sections were analyzed at focused spots (beam diameter <1 μm) to obtain accurate elemental compositions, complementing the EDS results and enabling reliable identification and characterization of mineral phases.

Morphological and mineral liberation characteristics were investigated using a LEO 1450 VP scanning electron microscope (SEM, Zeiss, Oberkochen, Germany) in backscattered electron (BSE) mode. BSE imaging provides excellent contrast based on differences in average atomic number, with phases containing higher atomic number elements appearing brighter. The SEM was equipped with an Oxford Instruments X-Act energy-dispersive X-ray spectrometer (EDS) with a 10 mm^2 silicon drift detector. EDS microanalyses were performed on selected points and areas using AZtec Energy software to determine the elemental composition of individual mineral grains.

C. Flotation Experimental Procedure

Prior to flotation tests, the 350 g sub-samples were ground to the target particle size of d_80= 65 μm in a laboratory ball mill. Grinding was performed using a charge consisting of 65 steel balls (30 balls of 60 mm and 35 balls of 45 mm diameter) at 65% solids by weight for 15 minutes.

The ground pulp was transferred to a 1000 mL mechanical laboratory flotation cell at a pulp density of 30% solids. Lime was first added to adjust the pulp pH to the desired value, and the pulp was conditioned for 1 minute. Sodium sulfide, as the sulfidizing agent and activator, was then added, and the pulp was further conditioned for 5 minutes. Potassium amyl xanthate (Z6, chemical formula $C_6H_{11}KOS_2$) was subsequently introduced as the collector, followed by 2 minutes of conditioning. Finally, methyl isobutyl carbinol (MIBC, $C_6H_{14}O$) was added as the frother at a dosage of 0.25 mL (equivalent to 35 g/t) and the pulp was conditioned for an additional 1 minute.

No dispersant (e.g., sodium silicate or sodium hexametaphosphate) was added in this study. The gangue is dominated by feldspars and quartz, with clay minerals (smectite and chlorite) identified as minor phases by XRD (Section 3.1), and the experiments were designed to isolate the individual effects of the collector, pulp pH, and sodium sulfide dosages. In addition, the alkaline pH (10–11) and the sulfide/hydrosulfide ions introduced by sodium sulfide can increase the negative surface charge of silicate and clay particles and thereby limit their aggregation and slime coating (Phiri et al., 2019). The potential benefit of an additional dispersant is nevertheless acknowledged and is recommended for future investigation. Potassium amyl xanthate was used as the sole collector because it is a strong and widely used collector for sulfidized oxide copper minerals and for secondary copper sulfides such as chalcocite and bornite, and its use as a single reagent allowed the effect of collector dosage to be interpreted without the confounding influence of a second collector. A mixed collector system (e.g., xanthate combined with dithiophosphate) may provide improved selectivity and flotation kinetics for the copper sulfide minerals present, and its evaluation is recommended for future work.

Froth collection was carried out manually for a total flotation time of 4 minutes or until the froth became barren (white). The concentrate and tailings were collected separately, dried in an oven at 85°C, weighed with an analytical balance (precision ±0.01 g), pulverized, and submitted for chemical analysis to determine total copper, oxide copper, and sulfide copper grades.

The recovery of total copper (R_{total}), oxide copper (R_{oxide}), and sulfide copper ($R_{sulfide}$) was calculated using the following equations:

$$R_{\text{total}}(\%) = \frac{C \cdot c}{C \cdot c + T \cdot t} \times 100 \quad (1)$$

$$R_{\text{oxide}}(\%) = \frac{C \cdot c_{\text{oxide}}}{C \cdot c_{\text{oxide}} + T \cdot t_{\text{oxide}}} \times 100 \quad (2)$$

$$R_{\text{sulfide}}(\%) = \frac{C \cdot c_{\text{sulfide}}}{C \cdot c_{\text{sulfide}} + T \cdot t_{\text{sulfide}}} \times 100 \quad (3)$$

where:

C= weight of dry concentrate (g)

T= weight of dry tailings (g)

c, c_{oxide}, c_{sulfide}= grade of total copper, oxide copper, and sulfide copper in the concentrate (%), respectively

t, t_{oxide}, t_{sulfide}= grade of total copper, oxide copper, and sulfide copper in the tailings (%), respectively.

D. Flotation Experimental Design

Response Surface Methodology (RSM) based on the Box-Behnken design was employed to design the experiments and optimize the sulfidization-flotation process. The experimental design and statistical analysis were performed using Minitab 18 statistical software.

Three independent variables were investigated at three levels each: collector (potassium amyl xanthate) dosage (100, 125, and 150 g/t), activator (sodium sulfide) dosage (1000, 1500, and 2000 g/t), and pulp pH (10, 10.5, and 11). These variables were selected based on preliminary tests and literature review.

The Box-Behnken design resulted in a total of 15 experimental runs, including three center point replicates to evaluate experimental error and process stability. The complete design matrix with the actual levels of the independent variables is presented in Table 1.

The Box-Behnken design (BBD) was preferred over a central composite design (CCD) for the following reasons. First, for three factors the BBD requires only 15 runs (12 edge-midpoint runs plus three center points). In contrast, a CCD with the same number of center points requires 17 runs; this reduces the number of flotation tests, each of which involves grinding and several chemical analyses. Second, the BBD uses only three levels per factor and has no axial (star) points outside the selected ranges, whereas a CCD would require levels beyond the investigated region (e.g., pH below 10 or above 11, and sodium sulfide dosages outside 1000–2000 g/t) that may be impractical or of no interest for the process. Third, the BBD contains no runs in which all factors are simultaneously at their extreme levels, which avoids unrealistic reagent combinations in which excessive depression could be expected. Despite the smaller number of runs, the BBD is adequate for fitting full second-order models with linear, quadratic, and two-factor interaction terms, as required in this work.

This design allowed for the evaluation of linear, quadratic, and interaction effects of the process variables on the recoveries of oxide copper, sulfide copper, and total copper. All experiments were conducted in a randomized order to minimize the effect of uncontrolled factors.

Table 1. Box-Behnken experimental design for the sulfidization-flotation of mixed oxide/sulfide copper ore.

Run No.	Collector dosage (g/t)	pH	Activator dosage (g/t)
1	100	11	1500
2	100	10.5	2000
3	150	11	1500
4	100	10.5	1000
5	100	10	1500
6	125	10.5	1500
7	125	10	1000
8	125	11	1000
9	150	10	1500
10	125	10	2000
11	150	10.5	1000
12	125	11	2000
13	125	10.5	1500
14	125	10.5	1500
15	150	10.5	2000

III. Results and Discussion

A. *Mineralogical Characterization*

A representative sample of the mixed oxide/sulfide copper ore was submitted for X-ray diffraction (XRD) analysis to identify the major and minor mineral phases present in the feed material. The XRD pattern of the ore sample is shown in Fig. 1. The characteristic diffraction peaks of the identified phases are labeled in the figure.

The results indicate that the major mineral phase is sanidine ($(Na,K)(Si_3Al)O_8$), a high-temperature potassium feldspar. The minor phases include albite ($(Na,Ca)(Si,Al)_4O_8$), quartz (SiO_2), smectite group minerals ($Ca_{0.2}(Al,Mg)_2Si_4O_{10}(OH)_2{\cdot}xH_2O$), chlorite ($(Mg,Fe)_6(Si,Al)_4O_{10}(OH)_8$), hematite ($Fe_2O_3$), and vermiculite ($(Mg,Al)_3(Si,Al)_4O_{10}(OH)_2{\cdot}4H_2O$).

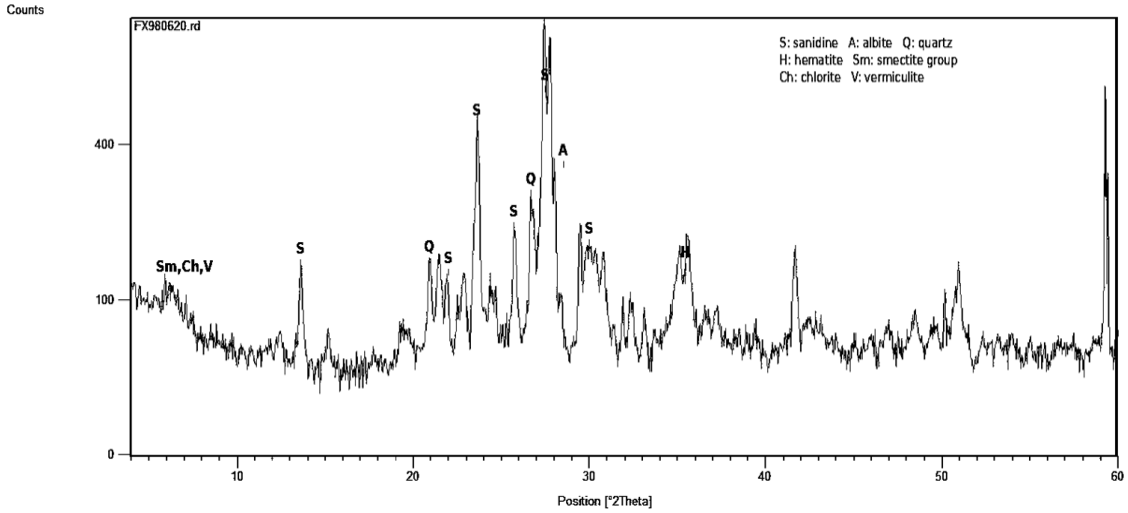
Consequently, the gangue minerals are predominantly silicates, with a significant content of clay minerals (smectite group and chlorite). Copper-bearing minerals were not detected in the XRD pattern. This is expected, as their concentration is below the typical detection limit of powder XRD (approximately 2–3 *wt.*%).

The silicate- and clay-rich gangue assemblage observed in this study is characteristic of many low-grade mixed copper ores, particularly those hosted in volcanic or volcaniclastic rocks. The presence of smectite and chlorite is of particular importance, as these phyllosilicates can cause significant challenges in flotation circuits through increased pulp viscosity, slime coating on valuable minerals, and higher reagent consumption (Lee et al., 2009; Sun et al., 2026).

This mineralogical profile provides a fundamental basis for understanding the flotation behavior of the ore and justifies the application of sulfidization-flotation with careful control of slime-related issues.

Backscattered electron (BSE) imaging and quantitative chemical analysis using an Electron Probe Micro-Analyzer (EPMA) were performed to accurately identify and characterize the copper-bearing minerals and their associations with gangue phases. Representative BSE images and EPMA results are presented in Fig. 2.

The analyses confirmed that the sulfide copper minerals in the ore consist primarily of chalcocite (Cu_2S) and bornite (Cu_5FeS_4), with only minor amounts of chalcopyrite ($CuFeS_2$). Pyrite (FeS_2) was also observed in association with the copper sulfides. The dominant copper oxide mineral was identified as malachite ($Cu_2(CO_3)(OH)_2$). Gangue minerals observed include plagioclase (Pl), alkali feldspar (Afd — consistent with sanidine), amphibole (Am), and iron oxides (Fe-Ox).



Sample:	Major Phase(s)	Minor Phase(s)	Minor Phase(s)
KV=40	Sanidine ($(Na,K)(Si_3Al)O_8$)	Albite ($(Na,Ca)(Si,Al)_4O_8$)	Hematite (Fe_2O_3)
mA=30		Quartz (SiO_2)	Vermiculite ($(Mg,Al)_3(Si,Al)_4O_{10}(OH)_2,4H_2O$)
Ka=Cu		Smectite Group ($Ca0.2(Al,Mg)_2Si_4O_{10}(OH)_2,xH_2O$)	
Fil=Ni		Chlorite ($(Mg,Fe)_6(Si,Al)_4O_{10}(OH)_8$)	

Fig. 1. X-ray diffraction pattern of the mixed oxide/sulfide copper ore sample showing the identified mineral phases (S: sanidine; A: albite; Q: quartz; H: hematite; Sm: smectite group; Ch: chlorite; V: vermiculite)

These results indicate that the ore contains a mixture of secondary copper sulfides and oxides, typical of supergene-enriched zones. The predominance of chalcocite and bornite is advantageous for the sulfidization-flotation process, as these minerals respond well to controlled sulfidization using sodium sulfide (Newell et al., 2007; Xiong and Zheng, 2013). Malachite, as the main oxide phase, can also be effectively activated under appropriate pH and sulfidizing conditions (Fu et al., 2026; Feng et al., 2024). However, the association of copper minerals with iron oxides and silicate gangue may require optimized reagent dosages to achieve selective flotation.

Scanning electron microscopy in backscattered electron (BSE) mode combined with energy-dispersive X-ray spectroscopy (EDS) was used to examine the texture, mineral associations, and liberation characteristics of the copper minerals in the flotation feed. A representative BSE image of the ore at 600× magnification, along with EDS point analyses, is shown in Fig. 3.

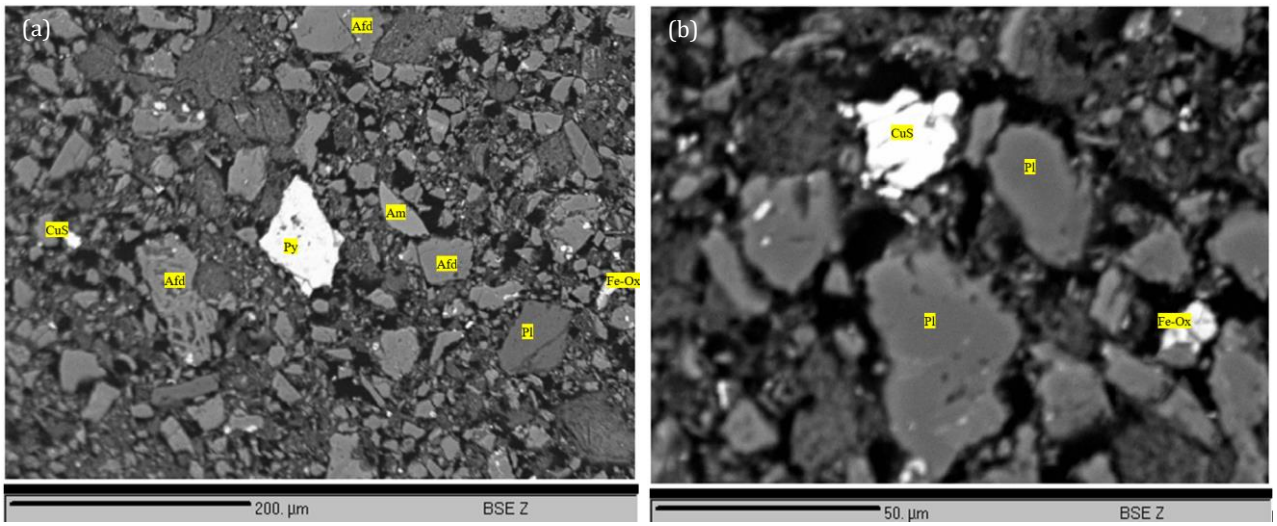


Fig. 2. Backscattered electron (BSE) images and EPMA analysis of the mixed oxide/sulfide copper ore showing mineral associations (CuS: chalcocite; Py: pyrite; Pl: plagioclase; Fe-Ox: iron oxide; Afd: alkali feldspar; Am: amphibole)

The BSE image clearly illustrates a heterogeneous mineral matrix. Point analysis at EDS 1 identified a chalcocite (Cu_2S) grain with an approximate size of 40 µm. This copper sulfide particle is enclosed within a larger grain of sanidine, as confirmed by the EDS spectrum at EDS 2, which shows dominant peaks of silicon (Si), aluminum (Al), and potassium (K), characteristic of alkali feldspar.

This textural relationship — secondary copper sulfide (chalcocite) occurring as inclusions within feldspar grains — indicates partial locking of valuable minerals within the silicate gangue. Although most copper minerals appear liberated at the target grind size (d_80 = 65 µm), such associations suggest that a portion of the copper may remain locked, potentially affecting overall flotation recovery.

The observed texture is consistent with supergene alteration processes commonly occurring in mixed copper deposits, where secondary copper sulfides such as chalcocite form through the replacement of primary minerals or precipitate along fractures and grain boundaries of host silicates (William and Chavez, 2021; Verhaert et al., 2017). These findings highlight the importance of adequate liberation and controlled sulfidization to maximize the recovery of both liberated and partially locked copper minerals during the flotation process.

To determine the total and oxide copper content of the ore and to evaluate the distribution of copper across different particle size fractions, a 360 g sub-sample was ground in a laboratory ball mill for 10 minutes to produce a representative flotation feed. The ground material was then wet-screened into various size fractions, and the total copper and oxide copper contents of each fraction were determined through aqua regia digestion and sulfuric acid leaching, respectively, followed by Atomic Absorption Spectrometry (AAS).

The results of the size-by-size analysis are presented in Table 2. The head sample contained an average of 0.86% total Cu and 0.57% oxide Cu, corresponding to an oxidation ratio of 66.83%. This indicates that the ore is a predominantly oxide-rich mixed copper ore.

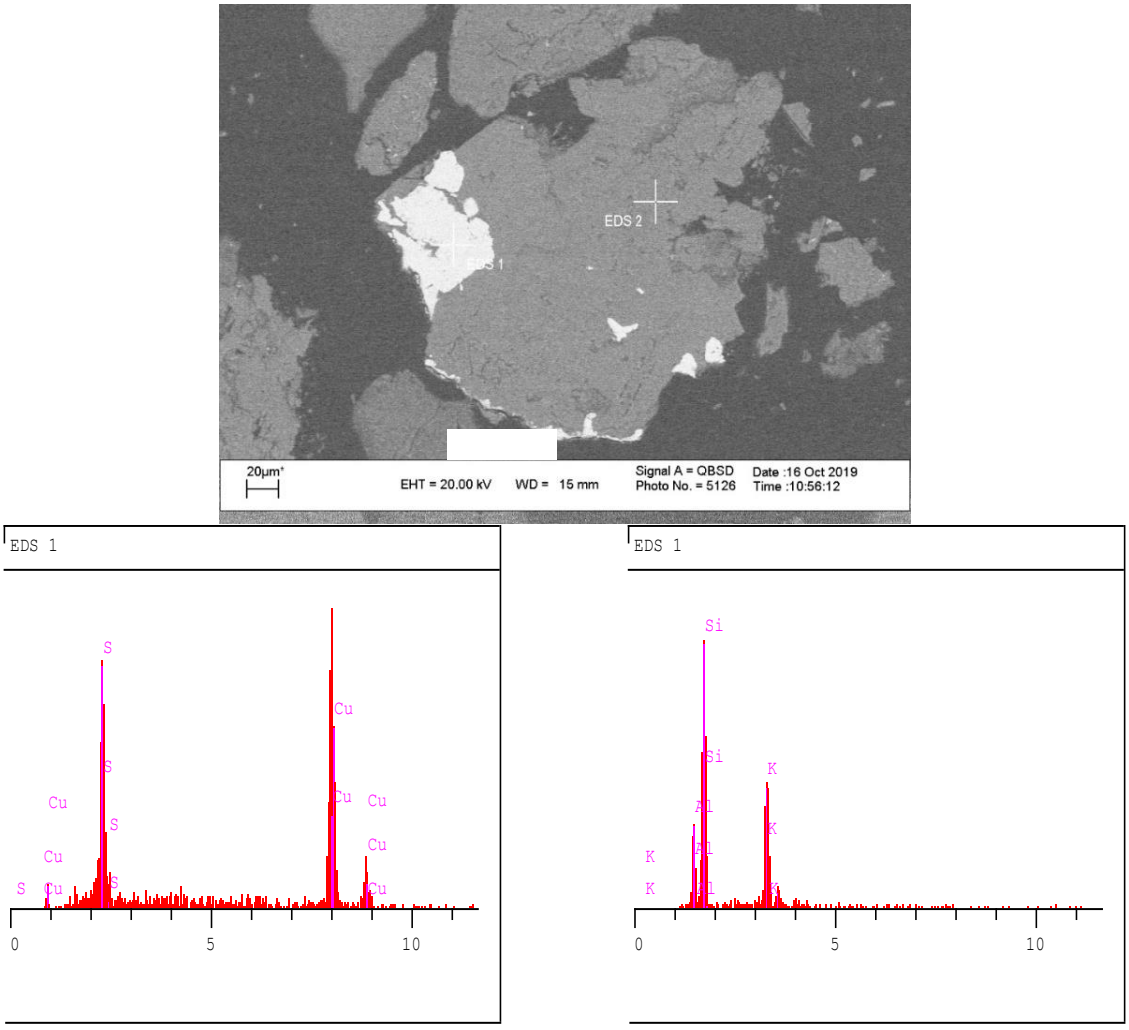


Fig. 3. Backscattered electron (BSE) image of the mixed oxide/sulfide copper flotation feed (600× magnification) with corresponding EDS spectra at two analysis points (EDS 1: chalcocite; EDS 2: sanidine).

Table 2. Mass distribution and copper assay by particle size fraction for the mixed oxide/sulfide copper ore.

Particle Size (μm)	Mass (g)	Wt.%	Cu Grade (%)		Cu Distribution (%)		Oxide Cu / Total Cu in Fraction (%)
			Oxide	Total	Oxide	Total	
+750	10.5	2.94	0.769	0.851	3.94	2.45	90.36
750–125	34	9.53	0.543	0.667	9.01	6.23	81.41
125–75	52.5	14.72	0.532	0.690	13.64	9.95	77.10
75–53	61.8	17.33	0.565	0.774	17.06	13.13	73.11
53–37	30.9	8.67	0.588	0.787	8.87	6.68	74.76
–37	166.9	46.80	0.583	0.699	47.48	45.72	58.40
Head (Calculated)	356.6	100	0.57	0.86	100	100	66.83

The data reveal that a significant proportion of both total copper (45.72%) and oxide copper (47.48%) is concentrated in the finest fraction (–37 μm). Conversely, the coarsest fraction (+750 μm) exhibits the highest copper grade and is almost entirely oxidized (90.36% oxide copper). In all size fractions, more than 58% of the copper is present in oxide form.

This size-dependent distribution is typical of many mixed oxide/sulfide copper ores, particularly those subjected to supergene alteration, where oxide minerals tend to concentrate in both the coarse and fine fractions. The high proportion of copper in the –37 μm fraction is a common challenge in flotation of mixed ores, as fine particles are prone to entrainment, slime coating, and reduced collision efficiency with air bubbles (Phiri et al., 2019; Asghari et al., 2019).

B. Optimization of Oxide Copper Recovery

The sulfidization mechanism plays a critical role in the activation of oxide copper minerals. Upon addition of sodium sulfide (Na_2S) to the pulp, the following equilibria occur depending on pH and Eh:

$$Na_2S + 2\,H_2O \leftrightarrow 2\,NaOH + H_2S \quad (4)$$

$$H_2S \leftrightarrow HS^- + H^+ \quad (5)$$

$$H_2S \leftrightarrow S^{2-} + H^+ \quad (6)$$

$$2HS^- + O_2 \leftrightarrow 2S^o + 2OH^- \quad (7)$$

$$2S^{2-} + 4O_2 \leftrightarrow 2SO_4^{2-} \quad (8)$$

The dominant sulfur species (H_2S, HS^-, or S^{2-}) is strongly pH-dependent. In the alkaline region (pH 7–12.5) under reducing conditions, the hydrosulfide ion (HS^-) is the primary species responsible for sulfidization. For malachite (a major oxide mineral in this ore), HS^- reacts with surface copper ions to form a thin, stable copper sulfide layer (mainly covellite, CuS), as shown in the following reaction (Xiong and Zheng, 2013):

$$(CuCO_3)_2.Cu(OH)_2 + 3HS^- = 3CuS + 2CO_2 + H_2O + 3OH^- \quad K = 2.65\times10^{8} \quad (9)$$

This surface transformation enables the adsorption of xanthate collectors. However, excessive sodium sulfide can depress both sulfidized oxide and sulfide minerals, necessitating precise dosage control.

The effects of collector dosage (potassium amyl xanthate, 100–150 g/t), pulp pH (10–11), and activator (sodium sulfide) dosage (1000–2000 g/t) on oxide copper recovery were investigated using the Box-Behnken response surface design. The main effects of these variables are illustrated in Fig. 4.

Collector dosage showed a quadratic effect. Oxide copper recovery initially decreased as collector dosage increased from 100 to around 125 g/t, then increased at higher dosages up to 150 g/t. This non-monotonic behavior can be attributed to the dual role of xanthate: at low concentrations, insufficient collector coverage occurs on the sulfidized surfaces, while moderate overdosing may enhance hydrophobicity. However, excessive collector can lead to non-selective adsorption or micelle formation.

Pulp pH had the most pronounced effect. Recovery increased significantly when pH rose from 10.0 to 10.5, but declined at pH 11.0. This trend is consistent with the optimal speciation of sulfur ions for sulfidization. The hydrosulfide ion (HS^-), which is the primary activating species for oxide copper minerals, reaches its maximum activity in the pH range of approximately 9.5–10.5. At higher pH values (≥11), the dominance of S^{2-} and excessive hydroxyl ions can lead to over-sulfidization or surface depression (Prokkola et al., 2020).

Sodium sulfide dosage exhibited a negligible overall effect on oxide copper recovery within the studied range, suggesting that even the lowest dosage (1000 g/t) was sufficient for effective sulfidization under the tested conditions.

The influence of sodium sulfide on the gangue minerals must also be considered. It should be noted that gangue recovery and concentrate grade were not measured in this work; the following interpretation is therefore based on the known chemistry of the system rather than on direct measurements. The gangue of this ore consists mainly of feldspars (sanidine and albite) and quartz, together with minor hematite and phyllosilicates (Section 3.1). Silicate minerals are not sulfidized by sodium sulfide and are therefore not expected to adsorb xanthate through a sulfidization mechanism; their recovery is more likely to occur through entrainment of fine particles in the froth, which depends on pulp pH and ionic strength. At pH 10–11, hydroxyl and hydrosulfide ions increase the negative surface charge of silicate and clay particles, which tends to disperse them, whereas at

high sodium sulfide dosages the increased ionic strength may compress the electrical double layer and promote coagulation and entrainment of fines. In contrast, oxide gangue minerals such as hematite may be partially sulfidized and interact with xanthate, which could reduce selectivity. Moreover, sulfide ions can precipitate dissolved Fe and Cu species (Prokkola et al., 2020) and be consumed by oxidation and by reactions with gangue surfaces, thereby lowering the amount of sulfidizing agent available for malachite (Newell et al., 2007; Corin et al., 2017; Wei et al., 2026). The negligible effect of sodium sulfide dosage on oxide copper recovery over 1000–2000 g/t (Fig. 4) suggests that, within this range, such consumption did not limit the sulfidization of malachite. A detailed assessment of the effect of sodium sulfide on the gangue minerals (e.g., through concentrate grade, mass pull, and zeta-potential measurements) is recommended for future work.

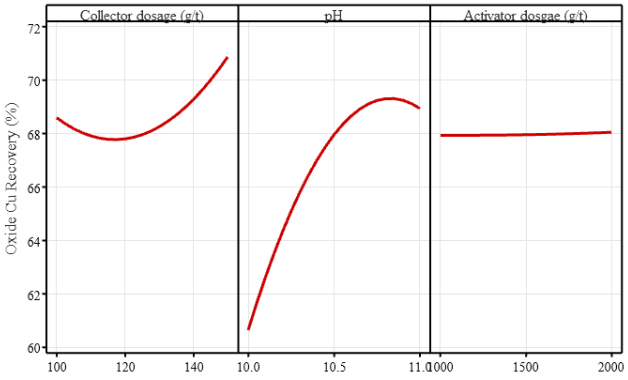


Fig. 4. Main effects plots showing the influence of collector dosage, pulp pH, and activator (Na_2S) dosage on oxide copper recovery in the sulfidization-flotation process (fixed conditions: 30% solids, d_80= 65 µm, potassium amyl xanthate as collector).

The interaction effects between variables are presented in Fig. 5. Significant interactions were observed between pH and activator dosage (Fig. 5a). At pH 10.0, increasing sodium sulfide dosage improved recovery by approximately 10%, likely due to enhanced sulfidization. In contrast, at pH 11.0, higher activator dosage slightly reduced recovery, probably due to the formation of excess sulfide species that act as depressants.

A strong interaction also existed between pH and collector dosage (Fig. 5b). At pH 10.0, higher collector dosage enhanced recovery, whereas at pH 11.0, increasing collector concentration had a negative effect. This highlights the sensitivity of the sulfidized surface to pulp chemistry and collector adsorption. No significant interaction was detected between collector and activator dosages (Fig. 5c).

These observations align with established sulfidization chemistry. The reaction forming a stable CuS layer on malachite (Reaction 9) is highly pH-dependent, with optimal performance in mildly alkaline conditions where HS^- predominates.

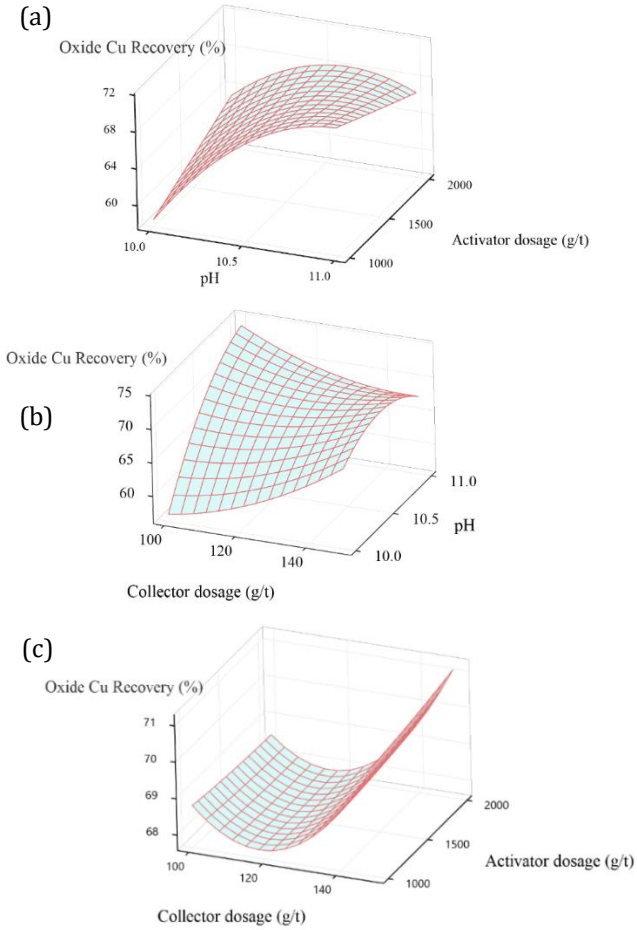


Fig. 5. Two-factor interaction plots for oxide copper recovery: (a) pH vs. activator dosage (collector = 125 g/t), (b) pH vs. collector dosage (activator = 1500 g/t), and (c) collector dosage vs. activator dosage (pH = 10.5)

Using the Response Optimizer tool in Minitab 18, the optimal conditions for maximum oxide copper recovery were determined to be 100 g/t potassium amyl xanthate, pH 11.0, and 1000 g/t sodium sulfide, yielding a predicted maximum oxide copper recovery of 76.49% (Fig. 6).

These findings are consistent with previous studies on the sulfidization-flotation of mixed copper ores, which emphasize the importance of controlled pH and moderate sulfidizing agent dosages to achieve effective surface activation while avoiding depression effects (Lee et al., 2009; Phetla and Muzenda, 2010; Corin et al., 2017; Wei et al., 2026).

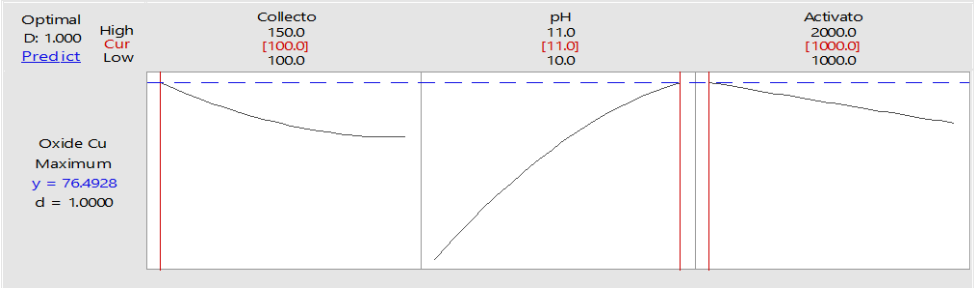


Fig. 6. Response optimizer plot showing the optimal conditions and predicted maximum oxide copper recovery (76.49%) in the sulfidization-flotation process (fixed conditions: 30% solids, d_{80}= 65 µm)

C. Optimization of Sulfide Copper Recovery

The effects of collector dosage (potassium amyl xanthate, 100–150 g/t), pulp pH (10–11), and sodium sulfide dosage (1000–2000 g/t) on sulfide copper recovery were analyzed using the Box-Behnken response surface methodology.

The main effects of the independent variables on sulfide copper recovery are presented in Fig. 7. Collector dosage (potassium amyl xanthate) exhibited a quadratic effect, with recovery decreasing as the dosage increased from 100 to approximately 125 g/t, followed by an improvement at higher dosages up to 150 g/t. This non-monotonic response was more pronounced than that observed for oxide copper. Pulp pH exerted the most significant negative effect, with sulfide copper recovery decreasing markedly and almost linearly as pH increased from 10.0 to 11.0. Activator (sodium sulfide) dosage showed a quadratic influence, whereby recovery increased from 1000 to about 1500 g/t before declining at higher concentrations.

These trends reflect fundamental differences in the surface chemistry and flotation behavior of sulfide versus oxide copper minerals. The quadratic response to collector dosage indicates the existence of an optimal adsorption density; insufficient collector leads to inadequate hydrophobization of the mineral surfaces, while moderate increases enhance floatability. The strong adverse effect of increasing pH is attributed to the reduced stability of the collector-mineral interaction and possible formation of hydrophilic metal hydroxyl species on sulfide surfaces at higher alkalinity. In addition, elevated pH shifts the sulfur speciation toward sulfide ions (S^{2-}), which can depress sulfide minerals. The quadratic effect of sodium sulfide dosage is characteristic of the narrow operational window for sulfidizing agents in mixed systems — moderate concentrations help activate any tarnished sulfide surfaces, whereas excess reagent results in over-sulfidization and depression (Lee et al., 2009; Phetla and Muzenda, 2010; Corin et al., 2017).

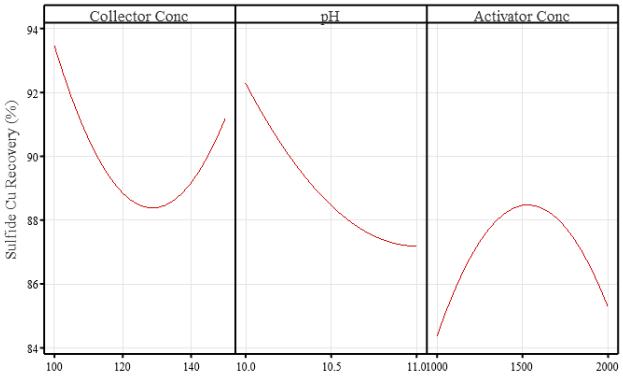


Fig. 7. Main effects plots showing the influence of collector dosage, pulp pH, and activator (Na_2S) dosage on sulfide copper recovery in the sulfidization-flotation process (fixed conditions: 30% solids, d_80= 65 µm)

The two-factor interaction effects on sulfide copper recovery are illustrated in Fig. 8. A significant interaction was observed between pulp pH and activator (sodium sulfide) dosage (Fig. 8a). At the low activator dosage of 1000 g/t, increasing pH from 10.0 to 11.0 resulted in a substantial decline in recovery (from approximately 92% to 82%). In contrast, at the high activator dosage of 2000 g/t, the effect of pH was more complex, with an initial decrease followed by a slight recovery at pH 11.0. Similarly, the interaction between pH and collector dosage (Fig. 8b) revealed contrasting behaviors: at 100 g/t collector, increasing pH caused a sharp drop in recovery (from nearly 100% to 88%), whereas at 140 g/t collector, higher pH led to a modest improvement in recovery. The interaction plot between collector dosage and activator (sodium sulfide) dosage on sulfide copper recovery (Fig. 8c) indicates that there is no significant interaction between these two variables within the studied range. The lines in the plot are nearly parallel, showing that the effect of collector dosage on recovery is largely independent of the activator dosage, and vice versa. This lack of interaction suggests that, under the experimental conditions tested, the mechanisms by which potassium amyl xanthate and sodium sulfide influence the flotation of sulfide copper minerals operate relatively independently. Sodium sulfide primarily acts as a sulfidizing agent and redox controller, while the collector is responsible for rendering the mineral surface

hydrophobic. The absence of a strong synergistic or antagonistic effect implies that within the dosage ranges of 100–150 g/t collector and 1000–2000 g/t Na_2S, increasing one reagent does not substantially alter the effectiveness of the other for sulfide copper recovery.

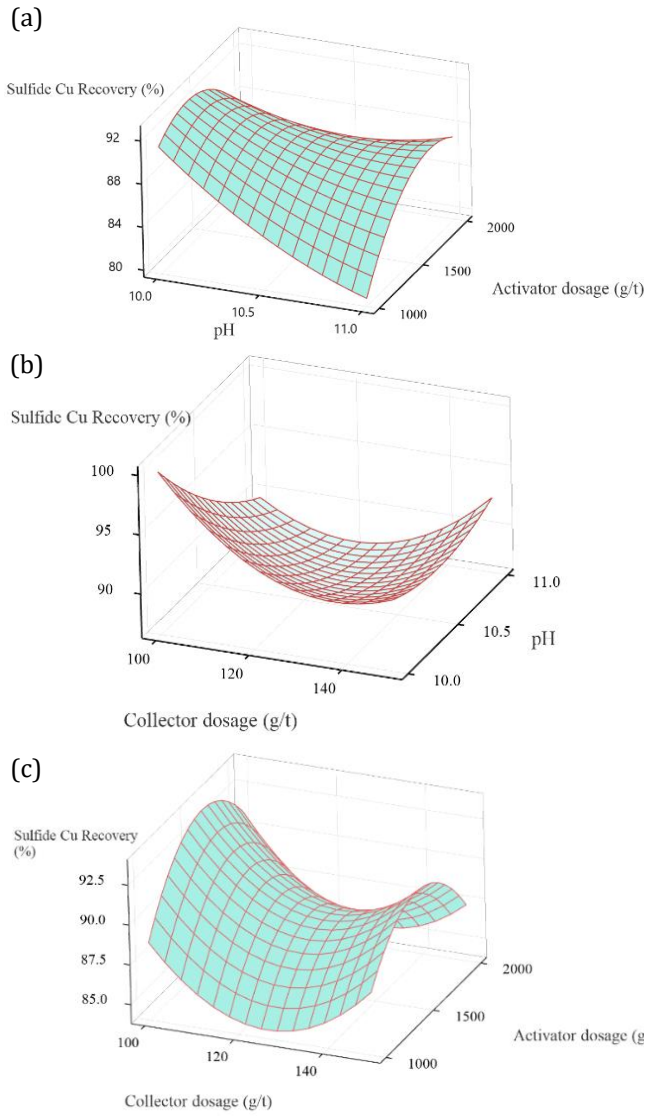


Fig. 8. Two-factor interaction plots for sulfide copper recovery: (a) pH vs. activator dosage (collector = 125 g/t), (b) pH vs. collector dosage (activator = 1500 g/t), and (c) collector dosage vs. activator dosage (pH = 10.5)

These interactions underscore the high sensitivity of sulfide copper flotation to pulp chemistry. The pronounced negative effect of increasing pH at low activator and collector dosages can be attributed to insufficient sulfidization and collector coverage, which fails to counteract the formation of hydrophilic species on the mineral surface under more alkaline conditions. At higher reagent dosages, the system appears to tolerate elevated pH better, likely due to enhanced surface activation and collector adsorption. The lack of interaction between collector and activator dosages suggests that their individual effects on sulfide mineral flotation are largely independent within the studied range. Such complex interactions are commonly reported in the flotation of mixed copper ores, where the balance between sulfidization and collector adsorption is critical and highly pH-dependent (Lee et al., 2009; Phetla and Muzenda, 2010; Corin et al., 2017; Wei et al., 2026).

Using the Response Optimizer in Minitab 18, the optimal conditions for maximum sulfide copper recovery were determined to be 100 g/t potassium amyl xanthate, pH 10.0, and 1252 g/t sodium sulfide, yielding a predicted maximum recovery of 99.99% (Fig. 9).

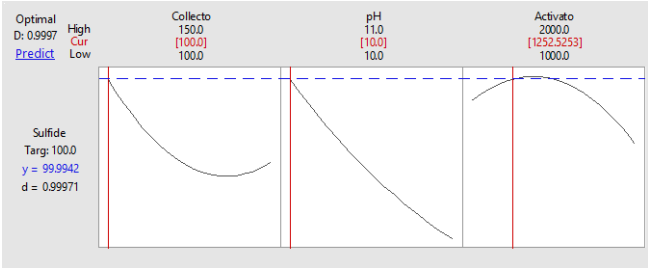


Fig. 9. Response optimizer plot showing the optimal conditions and predicted maximum sulfide copper recovery (99.99%) in the sulfidization-flotation process (fixed conditions: 30% solids, d_{80}= 65 µm).

The high recovery of sulfide copper compared to oxide copper is consistent with the inherent floatability of sulfide minerals and has been reported in numerous studies on mixed copper ores. The differing optimal pH for sulfide (pH 10) versus oxide copper (pH 11) highlights the challenge of simultaneous flotation of both mineral types and justifies separate optimization of responses (Lee et al., 2009; Phetla and Muzenda, 2010; Corin et al., 2017; Wei et al., 2026).

D. Optimization of Total Copper Recovery

The effects of collector dosage (potassium amyl xanthate, 100–150 g/t), pulp pH (10–11), and sodium sulfide dosage (1000–2000 g/t) on total copper recovery were examined using the Box-Behnken response surface methodology. The main effects plots are shown in Fig. 10.

Collector dosage exhibited a quadratic effect, with total copper recovery initially decreasing by about 2% as the potassium amyl xanthate concentration increased from 100 to approximately 125 g/t, followed by an increase at higher dosages up to 150 g/t. This pattern is similar to that observed for both oxide and sulfide copper recoveries, although the magnitude of the initial decrease was smaller than that for sulfide copper. Pulp pH showed a positive overall effect on total copper recovery, with recovery increasing as pH rose from 10.0 to 11.0. This trend is primarily driven by the beneficial effect of higher pH on oxide copper recovery, which outweighs the negative impact of elevated pH on sulfide copper recovery, given the predominance of oxide copper in the ore (approximately 67% of total copper).

Sodium sulfide dosage displayed a quadratic influence on total recovery, increasing from 1000 to around 1500 g/t before declining at higher levels. This behavior reflects the combined response of both copper fractions: moderate activator dosages enhance oxide copper sulfidization, while excessive dosages depress both oxide and sulfide minerals.

In summary, total copper recovery represents a compromise between the differing optimal conditions for the oxide and sulfide fractions. While higher pH favors oxide copper flotation, it is detrimental to sulfide copper recovery. Similarly, the quadratic responses to collector and activator dosages indicate the existence of an optimal operating window that balances the activation of oxide minerals with the avoidance of depression of sulfide minerals.

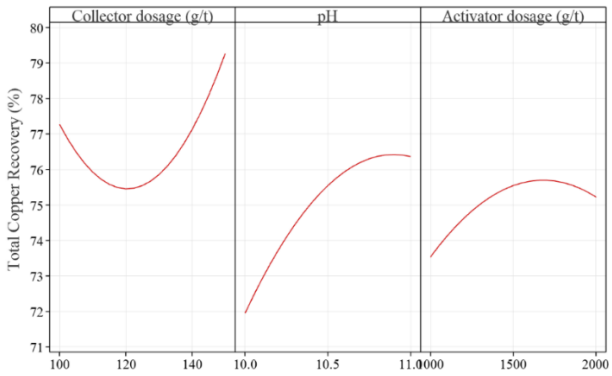


Fig. 10. Main effects plots showing the influence of collector dosage, pulp pH, and activator (Na_2S) dosage on total copper recovery in the sulfidization-flotation process (fixed conditions: 30% solids, d_{80}= 65 µm)

The two-factor interaction effects on total copper recovery are illustrated in Fig. 11. A significant interaction was observed between pulp pH and collector dosage (Fig. 11a). At pH 10.0, increasing collector dosage enhanced total copper recovery. In contrast, at pH 11.0, higher collector concentrations resulted in reduced recovery. This interaction arises from the opposing responses of the two copper fractions: higher collector dosage at lower pH benefits sulfide copper flotation, while at pH 11.0 the positive effect on oxide copper is outweighed by the depression of sulfide minerals.

No significant interaction was detected between collector and activator dosages (Fig. 11b). Increasing activator dosage generally improved total copper recovery across the studied collector range. This additive effect is mainly attributed to enhanced sulfidization and activation of the dominant oxide copper fraction, with minimal adverse impact on sulfide copper minerals within the tested dosages.

Similarly, the interaction between pH and activator dosage (Fig. 11c) was not significant. Higher sodium sulfide concentrations tended to increase total copper recovery at both pH levels, primarily due to improved flotation of oxide copper minerals, despite the potential depression of the sulfide fraction at very high activator levels.

Overall, total copper recovery is dominated by the behavior of the oxide copper fraction. Consequently, the interactions observed for total recovery more closely resemble those of oxide copper, while being moderated by the opposing trends of the sulfide copper fraction.

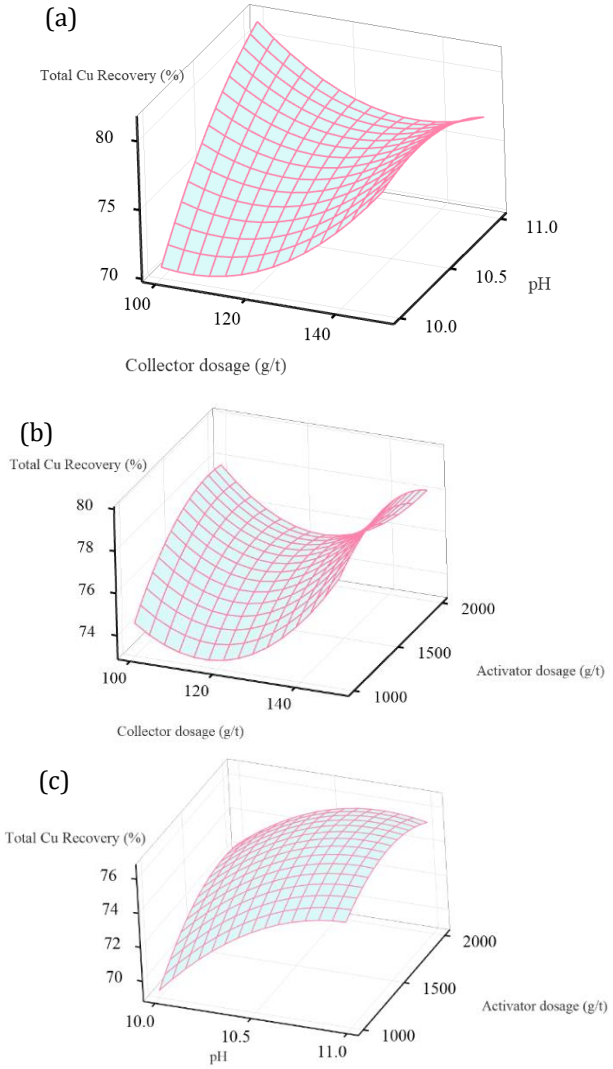


Fig. 11. Two-factor interaction plots for total copper recovery: (a) pH vs. collector dosage (activator = 1500 g/t), (b) collector dosage vs. activator dosage (pH = 10.5), and (c) pH vs. activator dosage (collector = 125 g/t)

The optimal conditions for maximum total copper recovery were determined using the Response Optimizer tool in Minitab 18 (Fig. 12). The predicted maximum total copper recovery of 81.40% was achieved at 100 g/t potassium amyl xanthate, pH 11.0, and 1707 g/t sodium sulfide.

At this optimum, the selected pH of 11.0 is favorable for oxide copper recovery (which constitutes the majority of the copper in the ore). At the same time, the relatively low collector dosage (100 g/t) helps minimize

depression of the sulfide copper minerals. The activator dosage of 1707 g/t provides sufficient sulfidization for the oxide fraction without causing significant depression of the sulfide minerals.

Consequently, the optimum for total copper recovery is shifted toward conditions that prioritize oxide copper flotation, reflecting the higher proportion of oxide copper in the feed and highlighting the challenge of simultaneously optimizing the flotation of both mineral types in mixed oxide/sulfide ores.

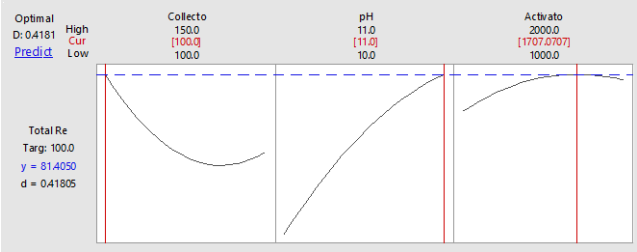


Fig. 12. Response optimizer plot showing the optimal conditions and predicted maximum total copper recovery (81.40%) in the sulfidization-flotation process (fixed conditions: 30% solids, d_{80}= 65 µm).

E. Development of Predictive Recovery Models

Following the execution of the Box-Behnken experimental design, quadratic response surface models were developed to predict the recoveries of oxide copper, sulfide copper, and total copper as functions of the key process variables: collector (potassium amyl xanthate) dosage (A, g/t), pulp pH (B), and activator (sodium sulfide) dosage (C, g/t). The fitted second-order polynomial models are presented in Equations (10)–(12):

$$\text{Oxide Cu Recovery (\%)} = -1985 + 2.98A + 333B + 0.100C + 0.00284A^2 - 12.7B^2 + 0.000000C^2 - 0.348AB + 0.000013AC - 0.0097BC \quad (10)$$

$$\text{Sulfide Cu Recovery (\%)} = 1231 - 3.772A - 156.8B - 0.0733C + 0.00617A^2 + 5.09B^2 - 0.000015C^2 + 0.2157AB - 0.000055AC + 0.01188BC \quad (11)$$

$$\text{Total Cu Recovery (\%)} = -914 + 1.66A + 156B + 0.0530C + 0.00437A^2 - 5.55B^2 - 0.000005C^2 - 0.248AB - 0.000066AC - 0.00276BC \quad (12)$$

These models incorporate linear, quadratic, and two-way interaction terms, consistent with the Box-Behnken design's capability to capture curvature and interactions in flotation systems.

F. Model Validation

After performing the flotation experiments according to the Box-Behnken response surface design, the validity and adequacy of the quadratic models for oxide copper, sulfide copper, and total copper recoveries were thoroughly assessed through residual diagnostic plots and Analysis of Variance (ANOVA). In response surface methodology, the residuals—defined as the differences between the experimentally observed values and those predicted by the model—must conform to several critical statistical assumptions. Specifically, the residuals should be normally distributed with a mean of zero and constant variance, and they should exhibit independence with no systematic patterns related to the order of experimentation. Satisfaction of these assumptions confirms the reliability of the experimental procedure and the predictive capability of the models. Any significant violation would necessitate repetition of the experiments or refinement of the model.

The four-in-one residual diagnostic plots for the total copper recovery model, which is representative of the overall model performance, are shown in Fig. 13. The normal probability plot (Fig. 13a) reveals that the residuals lie closely along the straight reference line, indicating a normal distribution of errors. This observation is supported by the histogram of residuals (Fig. 13b), which displays a roughly symmetric distribution centered around zero. The plot of residuals versus fitted values (Fig. 13c) demonstrates a random scatter of points around the horizontal axis without discernible patterns such as funnels or curves, thereby confirming the assumption of constant variance (homoscedasticity) across the range of predicted responses. Finally, the residuals versus observation order plot (Fig. 13d) shows no systematic time-dependent trends or autocorrelation, with residuals fluctuating randomly on both sides of the zero line and confirming the independence of the observations. Similar diagnostic patterns were obtained for the oxide and sulfide copper recovery models.

Collectively, these residual analyses demonstrate that the developed quadratic models adequately satisfy the underlying assumptions of regression analysis and provide a reliable representation of the flotation behavior within the experimental domain. This conclusion is further reinforced by the ANOVA results (Table 3), in which the overall model is highly significant (p = 0.003) while the lack-of-fit test is non-significant (p = 0.961), indicating no evidence of model misspecification. Therefore, the predictive equations (Equations 10–12) can be confidently employed for process optimization and prediction of copper recoveries in the sulfidization-flotation of the studied mixed oxide/sulfide ore.

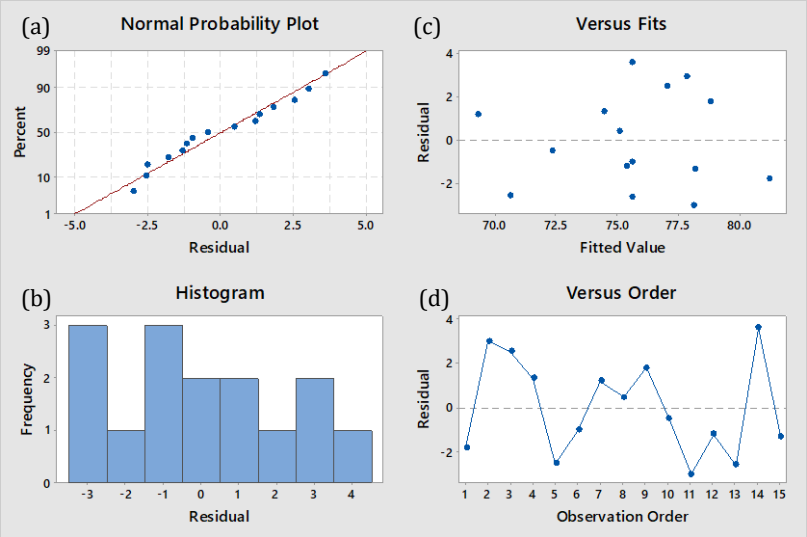


Fig. 13. Residual diagnostic plots for the total copper recovery model in the sulfidization-flotation process using Box-Behnken response surface methodology: (a) normal probability plot, (b) histogram of residuals, (c) residuals versus fitted values, and (d) residuals versus observation order (fixed conditions: 30% solids; activator: Na_2S; feed d_{80} = 65 µm)

Table 3. Analysis of Variance (ANOVA) for the copper recovery model using Box-Behnken design.

Analysis of Variance							
Source			DF	Adj SS	Adj MS	F-Value	P-Value
Model			9	248.933	27.6592	16.34	0.003
	Linear		3	64.378	21.4594	12.68	0.009
		Collector dosage	1	10.527	10.5274	6.22	0.055
		pH	1	52.028	52.0285	30.74	0.003
		Activator dosage	1	1.822	1.8223	1.08	0.347
	Square		3	118.277	39.4256	23.29	0.002
		Collector dosage * Collector dosage	1	54.981	54.9810	32.49	0.002
		pH * pH	1	5.979	5.9794	3.53	0.119
		Activator dosage * Activator dosage	1	48.770	48.7699	28.82	0.003
	2-Way Interaction		3	66.278	22.0926	13.05	0.008
		Collector dosage * pH	1	29.090	29.0900	17.19	0.009
		Collector dosage * Activator dosage	1	1.897	1.8974	1.12	0.338
		pH * Activator dosage	1	35.290	35.2904	20.85	0.006
Error			5	8.462			
	Lack-of-Fit		3	0.966	1.6925	0.09	0.961
	Pure Error		2	7.497	0.3219		
Total			14	257.395	3.7484		

IV. CONCLUSION

This study successfully applied the Box-Behnken response surface methodology to optimize the sulfidization-flotation process for a mixed oxide/sulfide copper ore containing approximately 0.86% total Cu (66.83% oxidation ratio). Detailed mineralogical characterization revealed that the ore is dominated by secondary copper minerals, primarily chalcocite, bornite, and malachite, hosted within a silicate- and clay-rich gangue consisting of sanidine, albite, quartz, smectite, and chlorite. Such associations highlight the challenges posed by slime formation and partial locking, underscoring the importance of controlled grinding (d_{80} = 65 µm) and reagent addition.

Quadratic predictive models were developed for the individual recoveries of oxide copper, sulfide copper, and total copper as functions of collector (potassium amyl xanthate) dosage, pulp pH, and sodium sulfide dosage. The models exhibited high statistical adequacy, as confirmed by residual diagnostics and ANOVA, with significant linear, quadratic, and interaction effects. Pulp pH emerged as the most influential variable, exerting opposing effects on oxide and sulfide copper flotation due to differences in surface chemistry and sulfur speciation. Collector dosage displayed consistent quadratic behavior across all responses, while sodium sulfide dosage required careful control to achieve effective sulfidization without depression.

Optimal conditions differed for each copper fraction, reflecting their distinct mineralogical responses. Maximum oxide copper recovery (predicted 76.49%) was achieved at 100 g/t collector, pH 11.0, and 1000 g/t Na_2S. Sulfide copper recovery reached nearly complete levels (predicted 99.99%) at 100 g/t collector, pH 10.0, and 1252 g/t Na_2S. For total copper recovery, which represents the practical industrial target, the optimum (predicted 81.40%) occurred at 100 g/t collector, pH

11.0, and 1707 g/t Na_2S — conditions that prioritize the dominant oxide fraction while maintaining acceptable sulfide performance.

These findings provide valuable insights into the independent optimization of oxide and sulfide copper recoveries in mixed ores, where simultaneous maximization is inherently challenging due to conflicting parameter requirements. Because the results depend on the specific ore type and mineralogical composition studied here, the developed models and optimized conditions are valid within the investigated ranges and cannot be directly generalized to all mixed oxide/sulfide copper ores; rather, they offer a guiding framework for the processing of similar ore types. Future work should focus on scale-up validation, multi-stage sulfidization strategies, the use of dispersants to mitigate slime effects, the evaluation of mixed collector systems (e.g., xanthate combined with dithiophosphate), a detailed assessment of the effect of sodium sulfide on gangue minerals, and the integration of additional techniques such as controlled potential sulfidization or combined gravity-flotation flowsheets to further enhance selectivity and overall recovery while mitigating the deleterious effects of clay minerals.

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