Heliyon 10 (2024) e26614

Contents lists available at ScienceDirect

Heliyon

journal homepage: www.cell.com/heliyon

Research article

Reclamation of iron and copper from BCL slag in Botswana

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ARTICLE INFO

Keywords:
Copper slag
Froth flotation
DoE
RSM
Iron and copper beneficiation

ABSTRACT

High-grade copper ores have been depleted over the years, making it a challenge in the mining industry. This investigation focused on a methodology to recover iron (Fe) and copper (Cu) from a copper/nickel slag obtained from the Bamangwato Concession Limited (BCL) mine in Botswana. In this modified flotation approach, the Response Surface Methodology (RSM) was used in conjunction with the Central Composite Design (CCD) and Analysis of Variance (ANOVA) to obtain the best optimal flotation conditions for the recovery of iron and copper. Using the RSM – CCD methodology, the optimal predicted responses were illustrated by a coefficient of determination $R^2 = 0.9839$ for recovery for Fe and 0.9655 for recovery for Cu. The recovery of copper increased with the increasing dosage of Na_2S and collector dosage, while the increase of pH, had a decrease in recovery of copper due to the decline in the stability of the froth, which led to the resistance to form stable bubbles for efficient recovery of copper. Selective flotation of copper and iron was achieved by varying the Na_2S dosage to achieve maximum recovery. Under these flotation conditions of PAX (800 g/t), pH (8), −75 μm, sulfurizing agent (Na_2S, 1000 g/t), flotation time of 8 min, pH regulator of NaOH and H_2SO_4 and Methyl Isobutyl Carbinol (MIBC) from the experimental runs merited a grade upgrade of Cu in froth concentrate from 0.581 mass% to 0.884 mass%. An enrichment ratio of 2 was realised, with the recovery of Cu being 62%, whereas Fe in the froth concentrate increased from 69.8 mass% to 71.8 mass%. The main aim was to upgrade the grade and recovery of copper and iron to enhance the recovery for copper and iron in the next experimental stage of leaching.

1. Introduction

The current global economic system is characterised by economic crises and uncertainties in the world market, including a market for diamonds, Botswana's dominant export commodity (Botswana Vision 2036). The global agenda for sustainable development is based on the notion that any effort that promotes sustainable development should be embraced. One is the beneficiation of secondary sources of mineral resources, such as metallurgical slags, to exploit the entire mineral value chain viably. Due to the increase in population and industrialisation, minerals such as copper and iron are in high demand. Over time, exploiting primary sources of these minerals has led to the depletion of high-grade ores, resulting in the processing of only low-grade and complex ores. Modern-day

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https://doi.org/10.1016/j.heliyon.2024.e26614
Received 30 October 2023; Received in revised form 15 February 2024; Accepted 16 February 2024
Available online 18 February 2024

research efforts and technological advancements have shifted their attention to the development and innovative methods to either process low-grade primary mineral resources or higher-grade secondary resources, such as slag, to alleviate the high demand for metals [1].

Base metals such as copper are known to be extracted using pyrometallurgical process routes to produce copper concentrate and a by-product of copper slag (smelter slag), regarded as waste. The world is moving towards a sustainable environmental consideration as well as economic compliance and is looking for ways to process environmentally hazardous waste, such as copper slag, which poses a problem to the environment [2–5].

Copper slag is considered waste, but some old mining operations used ancient and inefficient processing technologies that led to significant amounts of copper being reported to the waste stream (copper slag) instead of the matte stream. Copper slag poses environmental issues because it is stored on vacant land and occupies space that can be used for other uses. Also, heavy metals can leach into the ground, affect the soil structure, and contaminate underground water resources [5,6]. This poses an environmental risk to the stakeholders of the mines and people residing in the vicinity of the mines. Due to this, many researchers have sought to address these problems through flotation, hydrometallurgy, and pyrometallurgy. Finding ways to utilise the copper slag has many benefits, which include rehabilitating the land and eradicating the risks brought by copper slag. Still, most importantly, they provide secondary resource of base metals.

Froth flotation is a physio-chemical process that exploits natural or induced differences in the surface properties of minerals to achieve separation [7]. A modified flotation approach was employed in this study, as it is used primarily for reprocessing ores/slag by employing surficial reactants. True flotation is the main principle that makes flotation possible, which means the different particles (desired minerals and gangue), with one selectively attached to the air bubbles. The second mechanism involves aggregation, which alludes to the physical attribute of entrainment between the particles inside the froth attached to the air bubble generated during flotation Click or tap here to enter text [8]**.** True flotation is vital because that is where the main recovery takes place; hence, conditions are adjusted to enhance the true flotation of the desired (valuable) mineral. Flotation's effectiveness is dependent on the hydrophobic and hydrophilic properties of the subject sample being treated [9,10]. The froth concentrate from the flotation process can further be processed by leaching. There are different types of leaching, but high-pressure leaching provides a better opportunity as it will be financially viable. But first, the flotation process is vital to upgrade the copper recovery and grade to maximize recovery fully at the second phase of high-pressure leaching.

The primary objectives of this study are to evaluate an effective method for beneficiating copper and iron, and upgrade the grade and recovery of the product sample. The parameters influencing flotation to beneficiate copper and iron were investigated to achieve this goal. Response Surface Methodology is incorporated to design the experimental test work, in to achieve the primary objective. There are different mathematical models, which can be used in conjunction with Response Surface Methodology, to investigate parameters to achieve a maximum outcome in this case recovery and grade [11,12]. A statistical approach concerning the design of experiments is used to investigate possible flotation conditions required to maximize high recovery and to upgrade grade to further recover copper and iron in the next phase of experimental set up and previous researchers agree with the statistical approach [13,14].

In addition, this study is vital for Botswana's economy as it continues to find solutions to the depletedand low-content copper ores for recovery. This study investigates copper slag as a secondary source of copper, in this way, it will aid in utilising Bamangwato Concession Limited (BCL) copper slag, which has accumulated over the years since its inception in the 1970's. Some of the heavy metals have the capability of contaminating groundwater and soil structure. Runoff water from rainfall aids in the leaching of these metals. This will help tackle the environmental issues caused by the copper slag, hence leading Botswana in the right direction in considering some of the United Nations Sustainable Development Goals (SDGs) related to the responsible adhesion and eventually promoting Botswana to reach a circular economy. This will be achieved by innovative techniques to recover copper and iron, which will aid in growth in the sustainable industry and improve the infrastructure of the mining town.

2. Materials and methods

2.1. Materials

The feed material (copper slag) used in this study was sourced from a mineral processing plant called BCL in Botswana's Northern region. The copper slag was sampled at different points at the slag dump, and tests were done on it in the laboratory to upgrade the copper mineral for the next phase of experimental test works [15].

All flotation reagents used were chemically pure and supplied by different suppliers to the Extractive Metallurgy Department at the University of Johannesburg, South Africa.

2.2. Characterisation of the slag

The raw copper slag was prepared and homogenised before being sent for different characterisation analyses.

2.2.1. Characterisation and experimental equipment

Experiments were conducted using the Denver D – 1/D-12 Clone flotation cell machine. A Sanxin SX711 pH/mV meter was used to determine the pH of the pulp before, during and after the flotation. A Buchner funnel, filter paper and vacuum pump were used to filter the solids from the liquid for both concentrates and tailings after flotation. The elemental composition of the raw sample, flotation concentrate, and tailings were analysed using an X-ray fluorescence (XRF) coupled with software. The concentration of the metallic elements were measured. The Scanning electron microscope-energy dispersive X-ray spectroscopy (SEM–EDS) was used to measure the imaging of the samples (raw copper slag, concentrate and tailings) [15–17].

In contrast, the X-ray diffractometer (XRD) was used to determine the mineral composition of the samples (raw copper slag and treated copper slag). Fourier Transform Infrared Spectroscopy (FTIR) was used to analyse the inorganic constituents in the samples, and the particle size distribution was studied by a Malvern Mastersizer. The **Rigaku RINT – 2200V** model was used, and Figs. 7 and 13 show the patterns produced by the XRD equipment [17,18].

2.2.2. Flotation reagents

The flotation reagents used in this study were potassium amyl xanthate (PAX), Sodium Isobutyl Xanthate (SIBX), Sodium normal propyl xanthate (SNPX) and Sodium ethyl xanthate (SEX). The frother used is Methyl isobutyl carbinol (MIBC), Sodium hydroxide (NaOH) and sulphuric acid (H_2SO_4) were the pH regulators. Sodium sulfide (Na_2S) was used as a Sulphidizing reagent and shown in (Table 1).

2.3. Flotation experiments

2.3.1. Milling experimental procedure

A laboratory rod mill was used for the experimental wet milling runs on the copper slag sample. A laboratory Buchner funnel inserted with a filter paper and a vacuum pump were used for filtration of solids from the liquid. A 1000 ml measuring cylinder was used to measure water as wet milling was employed, and for measuring water used for flotation. The rod mill used steel rods, and it was a steel vessel. An oven was used for drying the filtered solids after milling, and an analytical balance was used to measure the mass of the dried samples from the oven. Sieves used for this work include 53 μm, 75 μm, 106 μm, 150 μm, 212 μm, 300 μm, 425 μm and 600 μm to determine size distribution, aided with a vibratory shaker.

2.3.2. Milling

Wet milling was done by measuring 1 kg of the slag sample and adding 580 ml of water to produce a slurry. The samples were then milled at different times of 50, 100, 150, and 200 min. After completing milling, the slurry was washed out of the milling vessel and from the grinding steel rods through the wire mesh, which kept the steel rods while the slurry was filtered, and the solids were dried in an oven at 70 °C for 2 h. This was an appropriate time that was monitored to be enough to remove the moisture. A Microtract analyser was used to determine the particle size distribution at each time frame of milling test work. This was done to generate a milling curve. Using the milling curve, it was found that 2 h 30 min (150 min) was enough to generate 80% less than 75 μm from the raw sample for the flotation experimental runs.

2.3.3. Flotation experimental procedure

The investigative experiments and results were based on the methodological approach shown in Fig. 1, which is based on four stages. The first stage covered defining the input parameters being investigated against the value limits of those parameters. These inputs were based on the problem being studied, with the limits of the parameters guided by previous researchers and tailored into this study with specifics of characterisation of the feed material and the problem to be solved (copper and iron upgrade) Click or tap here to enter text [19]. The experimental runs were generated using Design Expert 12 software, using Central Composite Design (CCD) performed through State–Ease Inc., USA. The factor parameters used in the software include pH, collector type, collector dosage and sulfurizing dosage. The output outcomes were Copper grade, copper recovery, iron grade and iron recovery. Equation 1shows a model hich is used to govern the input parameter to evaluate the output.

Experimental runs were generated according to the input parameters. A Denver Flotation machine (D – 1/D-12 Clone) was used for the flotation experimental runs. The liberated copper slag was dewatered and introduced into a flotation cell, and water was added to the solid sample to produce 60% solids and 40% liquid. The flotation experiments were each prepared out at different run specifics, using H_2SO_4 and NaOH to regulate the pulp pH while adding the specific collectors (PAX) and conditioning time of 5 min, and 1-min conditioning after adding the frother, MIBC. The agitation speed was set at 1000 rpm, and air was bubbled into the pulp at a rate of 2.5 l/min. Froth concentrate was collected for 8 min for each run. The concentrate, as well as the tailings, was collected and filtered using a Buchner funnel and pump. They were both dried at 70 °C for 3 h. The experimental test runs were specific and corresponded to the DoE to investigate the individual flotation-specific response of the factors being studied (Table 2) to achieve upgrading the copper and iron for the next phase of recovery (High-pressure leaching).

Table 1
Flotation reagents used for experimental runs.

Frother	Collectors	pH regulators	Sulphidizing agent
MIBC	PAX	NaOH	Na_2S
	SIBX	H_2SO_4	
	SEX		
	SNPX		

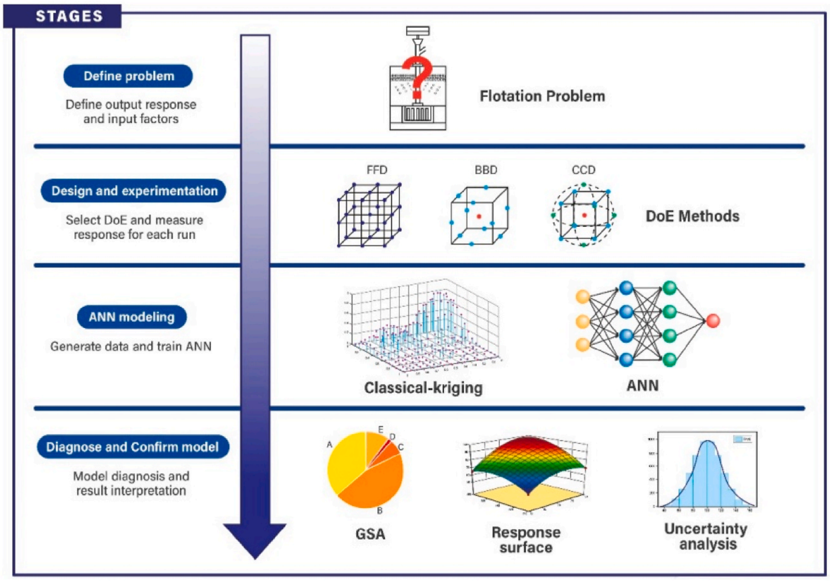


Fig. 1. Proposed methodology for this investigative work [17].

Table 2
Experimental flotation conditions.

Flotation Factors	Symbol	Coded levels			
		1	2	3	4
Activator (Na_2S) dosage (g/t)	A	100	300	500	1000
Collector dosage (g/t)	B	300	500	500	800
pH	C	6	8	10	12
Collector type	D	SIBX	SEX	SNPX	PAX

$$y = \beta_o + \sum_{i=1}^{n} \beta_i X_i + \sum_{i=1}^{n} B_{ii} X_i^2 + \sum_{i=1}^{n} \sum_{j=i+1}^{n} B_{ij} X_i X_j + \epsilon \quad (1)$$

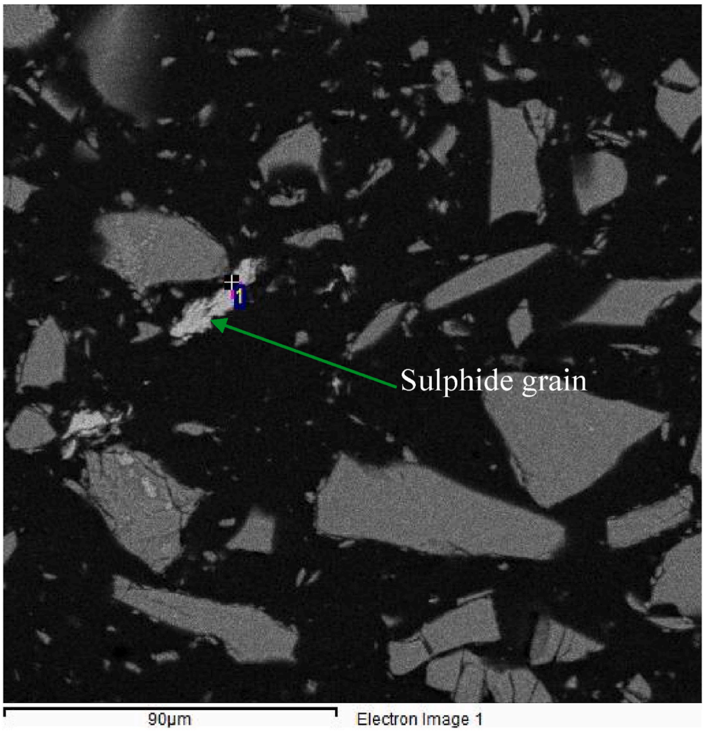
3. Results and discussions

3.1. Scanning electron microscope (SEM-EDS) analysis

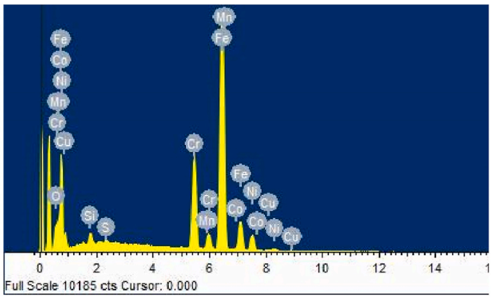
Figs. 2, 3 and 4 illustrate the scanning electron images gained from SEM analysis of the BCL raw copper slag. Fig. 2 shows spot analyses at point 1, which shows the different mapping concentrations of the minerals in that area. It indicates that iron is dominant in all the elements analysed, with the target elements Fe and Cu being available at significant levels. It clearly shows fayalite grains with fractions of Fe – Cu – Ni Sulphide compound, which is supported by Figs. 3 and 4. Fig. 3 shows the spread of Fe across the glass host and can be illustrated by the concentration on the magnitude of the spots on the micrographs. The Fe – Cu – Ni sulphide compound is shown in Fig. 3 and is found in the raw copper slag. This is in agreement with other researchers in this field, which indicate the high concentration of Fe present in the glass host with highlights of bright spots of Fe – Cu – Ni sulphide [17,19].

The flotation concentrate obtained from the experimental test works, from the best flotation conditions, was subjected to characterisation (microstructural) analyses (XRD, SEM–EDS) to correlate the efficacy of the process.

Fig. 5 shows spot analysis at point 1, having mapping of all the elements shown (Cu, Fe, Ni, K, Ti, S and Co). This area is rich in Cu and Ni as it is sulphide grain exposed and entrapped, the goal would be to unlock these minerals of interest for flotation. The analysis point indicates that from the froth concentrate, there has been an upgrade from the raw copper slag, having Cu increased from 0.55% to 18.24% after applying optimal flotation conditions, which will be vital for the next phase of the study (atmospheric and high-pressure leaching). It also shows that iron decreased or escaped mostly to the flotation tailings as it decreased from 69.78% to 17.76%, which will also be used as a raw material in the next phase of the study. It illustrates that the reagents used in the experimental runs were absorbed significantly at the surface of the mineral's particle to facilitate the appropriate operation of the reagents and allow upgrading of the copper and iron particles, which can be observed in the images [16,19]. are in agreement with current study results, indicating an upgrade in the grade of the froth concentrate for both iron and copper, as well as a significant recovery of Cu and Fe. This



Element	Weight %	Atomic %
Cu	0.55	0.45
Fe	69.78	64.86
Cr	17.03	17.01
S	0.16	0.26
Co	0.29	0.25
Ni	7.59	6.71



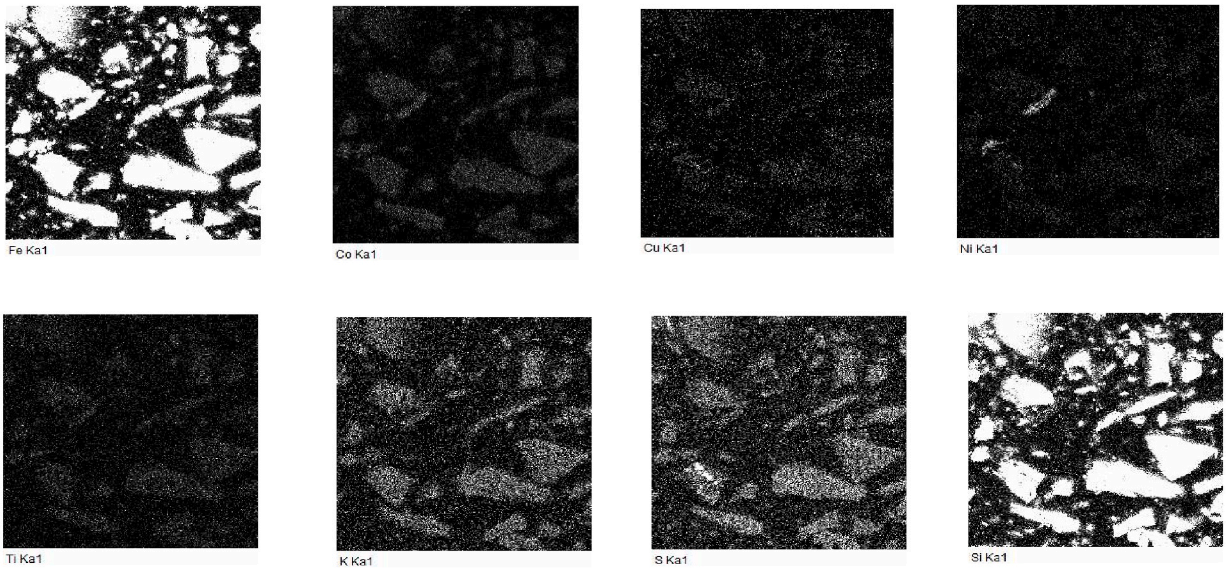


Fig. 2. SEM-EDS surface scan analyses of untreated raw copper slag.

is also in agreement with previous work, as it is essential for the next phase of experimental runs for leaching out copper and iron.

Fig. 6 shows a spot analysis of point two, where a significant amount of minerals of interest are concentrated after the flotation upgrade.

Fig. 2. Shows the fayalite grain at different size resolutions in the glass host, and this is supported by the XRD scans indicating that the Fayalite and chromite are the primary copper and iron mineral bearing in the copper slag. The XRD scans reveal Fayalite as the dominant waste phase, which is also confirmed by the SEM micro images in the copper slag.

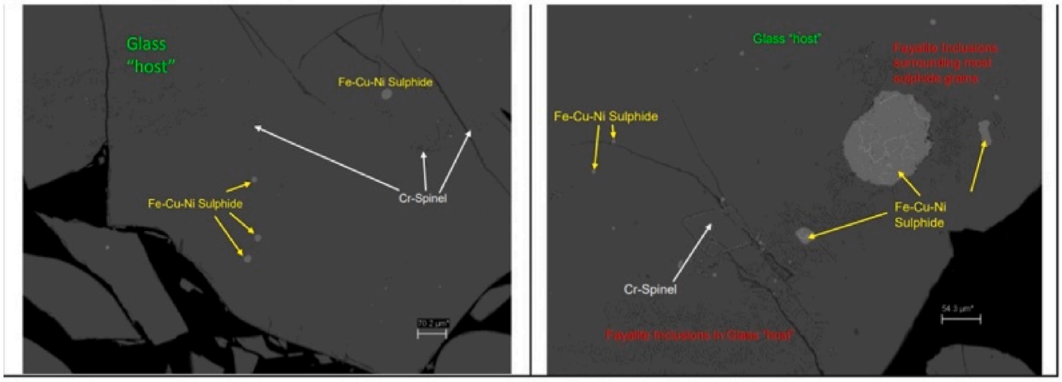


Fig. 3. SEM imaging of raw copper slag [20].

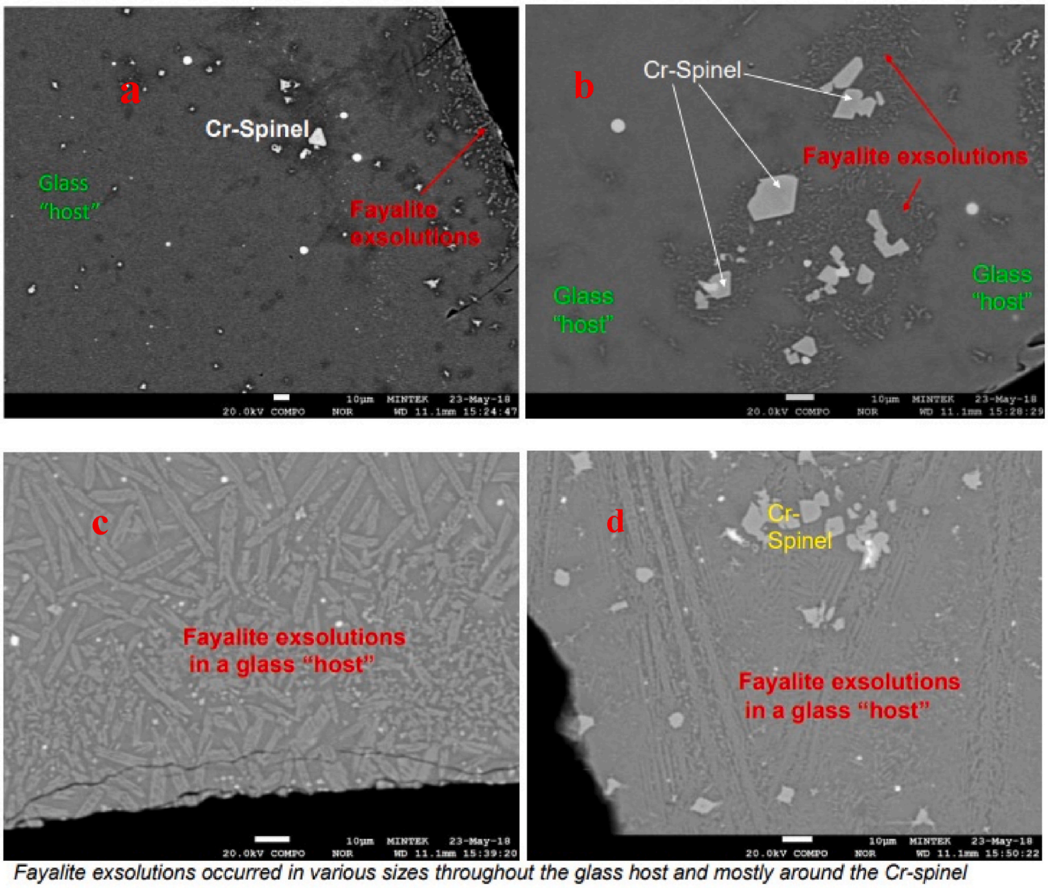
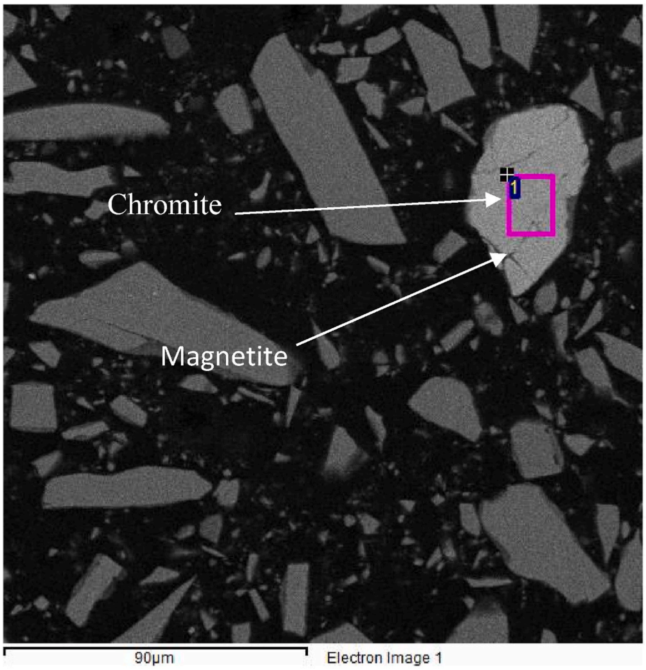


Fig. 4. EMPA imaging shows the occurrence of detected crystalline fayalite phases within the glass host [20].

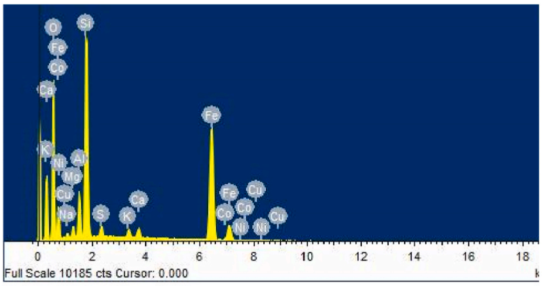
3.2. Mineralogical analyses

The raw copper slag sample was analysed using XRD, and Fig. 7 shows the mineral composition dominating as Fayalite, followed by chromite. The sample is mainly amorphous, illustrated by the high background emitted by the XRD patterns. This is common among copper slags due to the way they were cooled down, and it can also be attributed to others having similar amorphous erections Click or tap here to enter text [15,17,21].

Table 3 also shows that Fayalite is the dominant mineral, followed by chromite. Fayalite contains a lot of Fe as the main process stream of the operating mine was not interested in the recovery of iron but only Cu and Ni. The Cu was not fully detected by XRD;



Element	Weight (%)	Atomic%
Cu	18.24	7.34
Fe	17.76	8.14
Al	0.51	0.48
S	21.14	16.87
Co	0.06	0.03
Ni	0.43	0.19



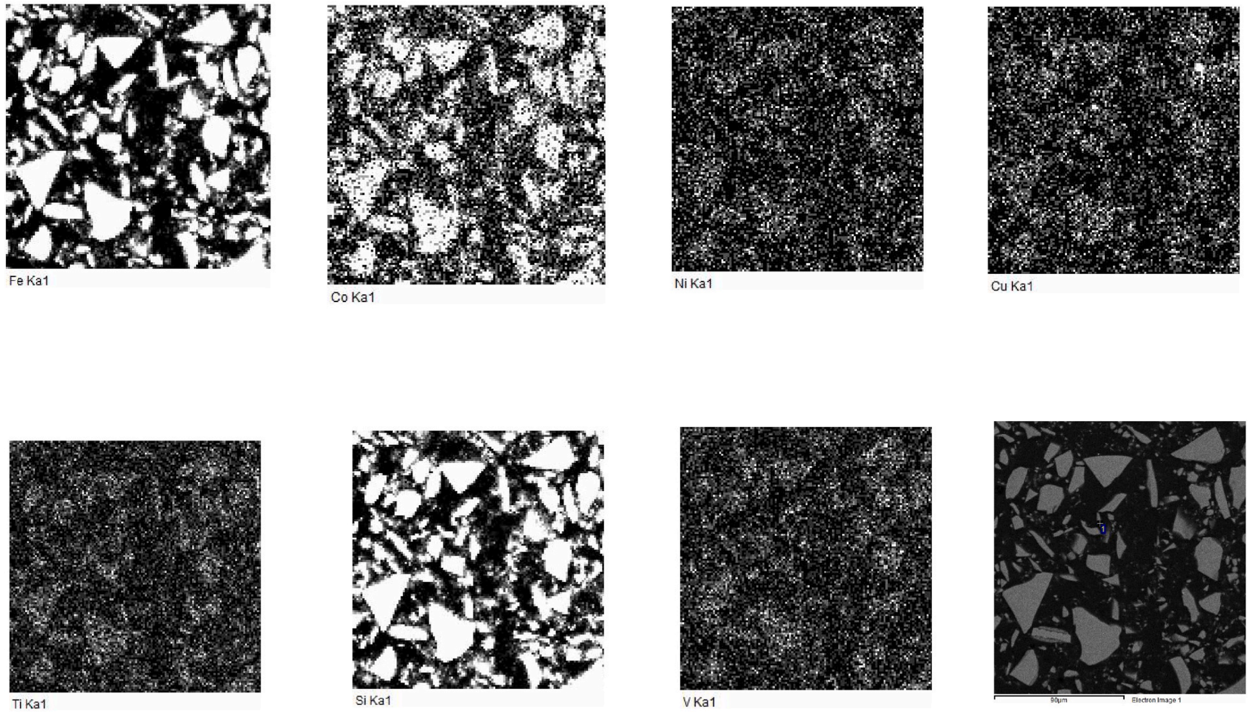


Fig. 5. SEM-EDS surface scan analyses of froth concentrate under optimal conditions.

hence, more tests were done with SEM–EDS. This is in agreement with various slags, which are formed in the same way, having a dominant fayalite phase and chromite, followed by traces of sulphides containing Cu, Ni and Co and oxides of the same metals as well Click or tap here to enter text [15,21,22].

Table 4 shows the elemental analysis of the raw copper slag being used for the flotation test works, with the initial concentration of copper being 0.581 %wt. and Fe being 72.81 %wt.

Table 5 shows the density and pH of the copper slag and flotation concentrate, respectively, which offers a slight change in the density of raw copper slag. This can be accounted for by the fact that most of the iron had been captured into the flotation tailings while Cu was accounted for in the concentrate.

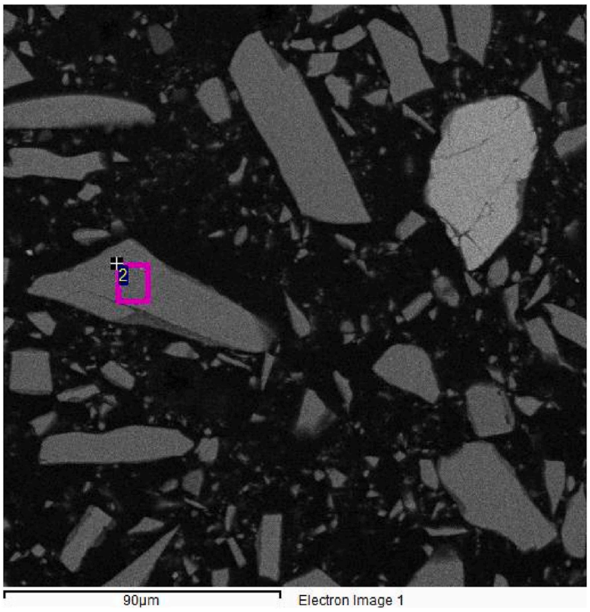


Fig. 6. SEM-EDS surface analysis.

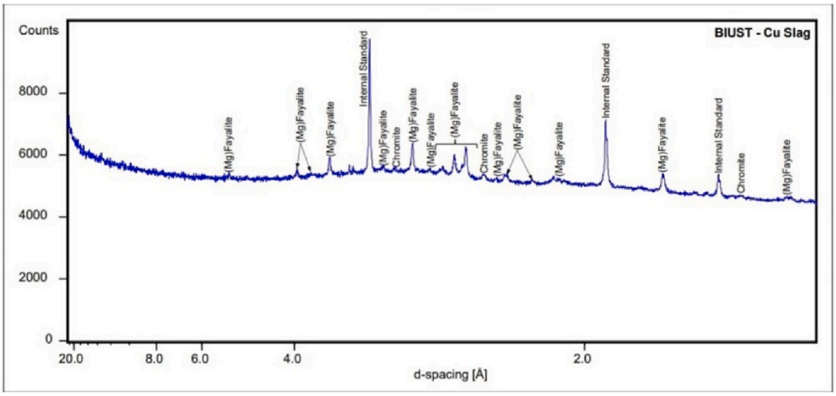


Fig. 7. XRD pattern of the copper slag sample [20].

Table 3
Mineral phases in the copper slag, determined by XRD [20].

Mineral	Chemical Formula	Approximate Abundance (%)
Fayalite	$(Mg,Fe)_2SiO_4$	10–20 %
Chromite	$(Co,Fe)CrO_4$	<3 %
Amorphous content		80%

* The sulphide content was below the detection limit of the XRD analysis.

Table 4
Chemical compositions of copper slag sample.

Sample name	Elements Grade (mass%)			
	Cu	Fe	Al	Ni
Raw CS	0.5810	73.4522	3.3785	0.2923

The comparison between raw copper slag and flotation concentrate indicates the successful upgradation and effectiveness of the flotation route as visualised. Based on the experimental test works carried out and characterisation analyses, it can be concluded that direct flotation is a feasible route using the proposed reagent regimes making it significant enough for the upgradation of copper slag

Table 5
Density and pH of raw copper slag and froth concentrate.

Sample	Density (g/cm^3)	pH
Raw CS	3.7682	7.59
Flotation Concentrate	3.6360	8.68

(low grade), which is supported by the above analyses from characterisation studies. Fig. 12 shows the peaks of the flotation concentrate, and a substantial agreement can be drawn between the applications, assessment, and justification of the flotation parameters concerning the established output process. Using Na_2S in the flotation experimental runs encouraged bubble stability during flotation to enhance the flotation of copper, hence improving the recovery of copper and iron, and this is in agreement with 23]- [24].

3.3. *FTIR analysis*

Fourier Transform Infrared Spectroscopy evaluated the samples. Fig. 10 shows the FTIR spectrum of the raw copper slag sample, and it can be seen that the Fayalite is dominant due to the spectra. For the raw copper slag, it can be seen that the peaks in the ranges of 1750–1455 cm^{-1}, which are associated with Si – O asymmetric stretching vibrations of the v_3 mode in SiO_4[17]. This shows the slag exhibits vibrations, which are mainly associated with Si – O bonds [25].

3.4. *Elemental analysis*

Spot analyses (Fig. 3) on the sulphide blebs revealed that elements occurred in varying compositions (22–32%) S, (13–33%) Fe, (21–46%) Ni and (3–46%) Cu. The darker areas on the sulphide grains were consistent in high content, while the lighter regions showed consistency in high Cu content.

Fig. 4 elaborates on the presence of Fayalite within the glass host of the slag, with the bright red colour suggesting the presence of sulphide. The phenomena are well within research results, with [23] having similar results, with a concentration between the glass and Cr – -spinel. Fig. 4 shows EMPA images revealing the nature of the occurrence of the detected crystalline fayalite phases within the glass host [20].

3.5. *Milling experiments*

Fig. 8 illustrates the particle size class distribution of the BCL copper slag and the response after milling the sample at different times. $-75+53\mu m$ percentage passing was used as the function of the milling time at 50-min intervals up to 3 h 18 min. The goal was to find how long it took to achieve the target of 80% passing 75 μm, which is the best size for flotation runs to be carried out. Fig. 8 shows that achieving this goal takes 2.67 h (160 min).

Elemental analysis was performed on different size distribution classes of the milled copper slag to determine the Cu deportment at each size class. This is shown in Fig. 9, which indicates copper in high amounts at -53 μm and $-75+53$ μm, which is in the range of the sample size used for the flotation test runs. This analysis is in agreement with that of [27]who reported something similar with copper deportment with respect to size classes. It also shows that as the particle size decreases, the copper content increases in the slag.

3.6. *Flotation experiments*

Flotation experiments were carried out using different flotation reagents, mainly used for copper flotation at the BCL copper and nickel mines. The flotation reagents used were varied through the flotation investigation to identify the best reagent suitable to float the copper and iron from the copper slag. MIBC (frother) and NaOH/H_2SO_4 were used in all tests as the primary frother and pH controllers. To improve the floatability of the mineral of interest (Cu and Fe), various primary collectors (PAX, SIBX, SEX and SNPX)

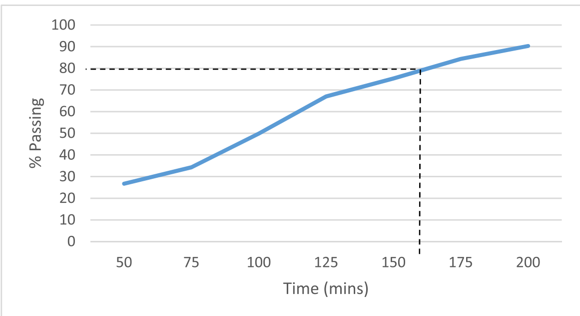


Fig. 8. Milling curve of copper slag liberated at different times.

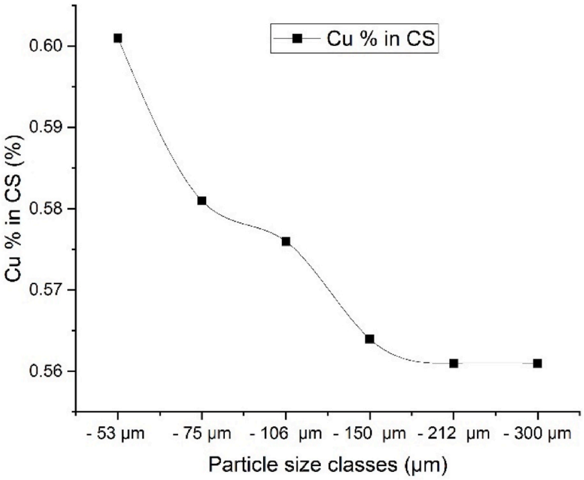


Fig. 9. Assay of copper at different size classes of the BCL copper slag.

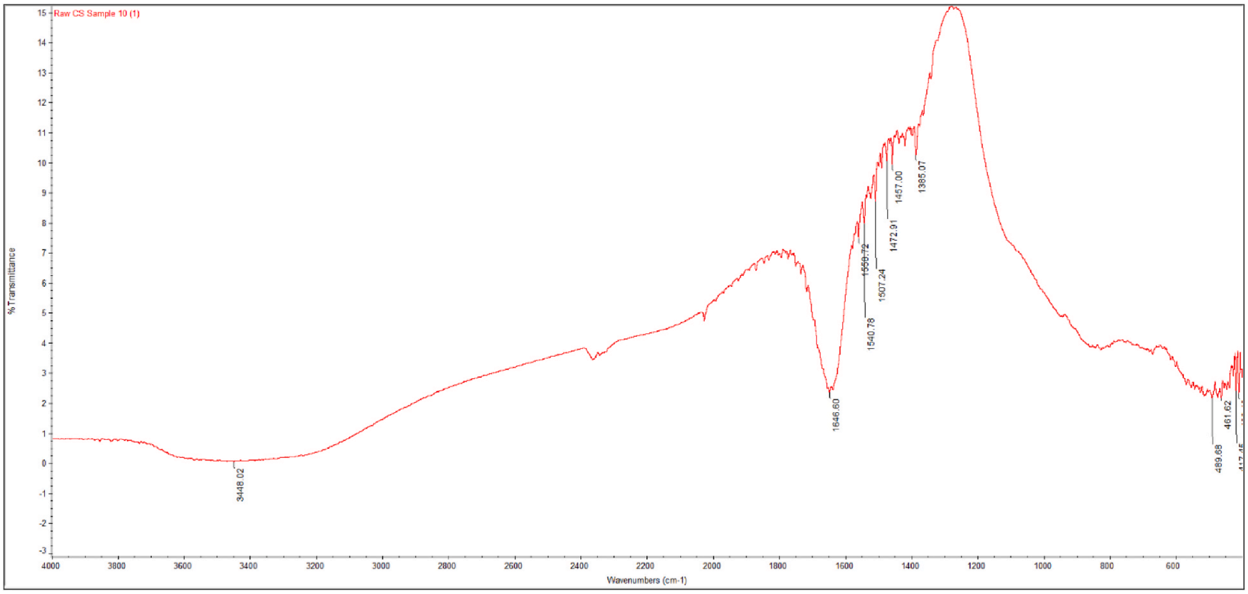


Fig. 10. Shows the FTIR spectrum of the raw copper slag.

and Sulphidizing agents (Na_2S) were used to exploit this. Table 8 shows multiple reagents used in this investigation.

Table 7 illustrates the assortment of flotation experimental runs executed, the collector type, the collector dosage, the flotation pH and the Sulphidizing dosage. This was used to evaluate the best operational conditions to upgrade the copper and iron for the next phase of experimental test works.

The recovery (R) of each metal from the copper slag and enrichment ratio (I) were evaluated by Eqs. (2) and (3).

Table 6
Bulk Modal Mineralogy for copper slag [20].

Mineral	Approximate Formula	Approximate Abundance
Glass Phase	$(Al - Ca - Cu - Fe - K - Mg - Na - O - S - Si)$	85.19
Fayalite	$(Mg, Fe)_2SiO_4$	12.86
Chromite	$FeCr_2O_4$	0.90
Sulphide	Cu_xS_y	1.05
Total		100.00

Table 7
Central composite design Experimental test runs and response using RSM.

	Input Factors				Output Factors	
Exp Runs	F_{Na2S}	F_{pH}	F_c	F_{CD}	Cu_R	Fe_R
1	4	4	4	1	55	66
2	4	1	1	1	44	51
3	4	1	4	4	36	50
4	1	4	4	4	31	63
5	1	1	4	4	27	54
6	4	1	1	4	62	44
7	−0.5	2	3	2	25	60
8	3	3	3	2	40	57
9	3	3	2	−0.5	51	64
10	4	4	4	4	41	60
11	3	3	2	2	37	56
12	3	2	3	2	38	56
13	1	4	1	1	37	63
14	3	2	5.5	3	45	61
15	2	5.5	3	3	41	66
16	3	3	3	3	38	56
17	4	4	1	1	49	59
18	1	1	1	1	23	57
19	2	2	2	5.5	21	50
20	1	1	1	1	24	53
21	5.5	2	2	2	50	54
22	1	1	1	4	20	46
23	1	1	4	1	39	59
24	4	4	1	4	33	54
25	2	−0.5	2	2	21	48
26	4	1	4	1	53	55
27	1	4	4	1	45	70
28	3	3	3	3	38	58
29	3	3	−0.5	3	30	49
30	2	3	3	3	35	57

Table 8
Flotation reagents were used in the experimental runs.

Frother	Collectors	Sulfurizing reagent	pH regulator
MIBC	PAX	Na_2S	NaOH
	SIBX		H_2SO_4
	SEX		
	SNPX		

$$Recovery : R\ (\%) = \frac{Cc}{Ff} \times 100 \tag{2}$$

$$Enrichment\ ratio : I = \frac{c}{f} \tag{3}$$

where R is the recovery of each metal of interest, and f and c represent the feed, concentrate, and tailings assays. In contrast, C and F represent the mass of concentrate and feed, respectively, and I represent the enrichment ratio.

The recovery of the metals of interest and the enrichment ratio confirmed the response to flotation tests.

3.6.1. Analysis of experimental tests work outcomes and the model

The Experimental parameters of froth flotation comprising of pH, collector type, collector dosage and sodium sulphide dosage were all investigated by Response Surface Methodology (RSM). The Central Composite Design with RSM discloses the connection between the different parameters being investigated and the effect of dependent variables, at the same time optimising the experimental runs and results of the multi–parameters under investigation [28] The CCD with the aid of RSM experimental runs and results are illustrated in Table 7 in conjunction with Table 2. The RSM also provides a predicted model from the model fitting techniques aided by the Design Expert software. Linear regression analysis is employed by equation 3.1 regarding coded parameters. The generated model helps by obtaining a response which illustrates the response as a function of the autonomous parameters and their relations.

Fig. 12 illustrates the froth concentrate; it can be seen that the significant peaks represent the phases of copper oxides and iron oxides, which agrees with the upgradation of copper slag through sulfurizing and pH regulation to increase the grade and recovery of copper and iron.

3.6.2. RSM – CCD plots

Design Expert software was used to create the response surface plots to fully comprehend the interaction of the parameters under investigation to reach optimum response [29]. The shape of the contour plots illustrates the significance drawn from the mutual relations between parameters. The shape of the contour plots has a different meaning; a spherical plot proposes the relationship among the related parameters is negligible, while an elliptical contour plot suggests that the relationship between associated parameters is significant. Figs. 13 and 14 illustrate the influence and relations of different parameters on the recovery of copper and iron, respectively, through 3D response plots. Concerning single parameters, the recovery ratio is significantly influenced by pH, Na_2S and PAX dosage and collector type. The influence of collector dosage towards flotation is less than the other parameters. The flotation ratio increases with the dosage of Na2S and pH but decreases gradually after it reaches its maximum value. The relationship between pH, Na_2S and PAX dosage was significant. This is credited to the fact that since copper slag has a low concentration of copper, it requires a high dosage of collector and PAX in that manner since the parent mine uses PAX to recover copper from its ore, following the process matrix. The pH also plays a significant role due to the copper being in the oxide phase due to being subjected to the smelter before being cooled; hence, process conditions at the flotation stage required the pH conditions to be as low as 8 compared to the traditional ones which range from 10 to 12 [8]/.

Conferring to the 3D plots, the relationships of pH and Na_2S dosage (Figs. 13 a and 14 a), collector dosage and Na_2S dosage (Figs. 13 b and 14 b), and collector type and pH (Figs. 13 c and 14 c). These interactions exist to provide the optimal conditions for an all-out response. Tables 9 and 10 further elaborate on the flotation conditions which have significant influence towards recovery. Collector dosage and Sodium sulphide dosage had the more significance influence compared to collector type and pH, this is showed by the parameter having a value of <0.0001. Table 10 three of the flotation parameters had more significance, collector dosage, pH, collector type and sodium sulphide dosage all had significant influence to floatability of the desired metallic component (Fe and Cu).

Adeq Precision measures the signal-to-noise ratio. A ratio greater than 4 is desirable. Your ratio of 73.541 indicates an adequate signal. This model can be used to navigate the design space.

3.7. Optimum flotation conditions and their application

One of the goals of RSM is to attain optimum operational experimental conditions for an optimal response. To achieve the maximum recovery of copper, pH of 8, PAX as the collector, collector dosage of 800 g/t and sodium sulphide dosage of 1000 g/t should be used. Repetitive experiments were conducted to confirm the validity of experiments of optimal conditions, reaching a recovery of 62% at 0.88 wt%.

Table 11 shows a summary of the optimum conditions evaluated by the RSM analysis. The maximum chemical composition of the froth flotation concentrate attained under the maximum optimum flotation conditions XRD analysis is also shown in Table 6 and Fig. 11, respectively. Table 12 shows the Cu grade in the flotation concentrate increased from 0.581 mass % to 0.884 mass % with an enrichment ratio of 4 from the optimum flotation conditions attained flotation, with the grade of Fe being 69.8% and 72.8%, respectively. The flotation concentrate comprised chalcopyrite ($CuFeS_2$) as the main component and quartz (SiO_2) as the gangue minerals. The flotation concentrate upgrade will be used in the second phase of the study of high-pressure leaching of copper and iron, which is similar to that of [26].

3.8. Limitations to study

The experimental flotation test works were carried out using different laboratorial equipment and chemicals, the following are the limitations which posed towards the investigation.

- Slag recovery technologies such as flotation which was utilized as the first stage of this investigation. Some of the limitations associated with flotation, copper bearing phases such as sulphides are easily recovered by flotation but the slag contains oxide which makes it hard to recover the desired minerals.
- Fine grinding is required to unlock minerals trapped in the locked fayalite phase, and fine grinding is energy intensive which is costly to achieve effective recovery.

Table 9
ANOVA Table for Cu recovery.

Source	Sum of squares	DF	Mean square	F – value	P - value	
Model	16.73	4	4.18	66.78	<0.0001	Significant
A - ***Na_2S dosage***	4.22	1	4.22	67.37	<0.0001	
B – pH	0.1914	1	0.1914	3.06	0.0927	
C – Collector type	3.02	1	3.02	48.21	<0.0001	
D – Collector dosage	8.36	1	8.36	133.58	<0.0001	

N.B: $R^2 = 0.9144, Ad\ R^2 = 0.9814, Pred\ R^2 = 0.9764$

Table 10
ANOVA Table for Fe recovery.

Source	Sum of squares	DF	Mean square	F – value	P - value	
Model	1089.67	4	272.42	383.61	<0.0001	Significant
A - ***Na_2S dosage***	60.17	1	60.17	84.50	<0.0001	
B – pH	560.67	1	560.67	787.45	<0.0001	
C – Collector type	228.17	1	228.17	338.01	<0.0001	
D – Collector dosage	240.67	1	240.67	338.01	<0.0001	

N.B: $R^2 = 0.9839$, $Ad\,R^2 = 0.9814$, $Pred\,R^2 = 0.9764$, The **Predicted** $R^2 = 0.9839$ is in reasonable agreement with the **Adjusted** $\boldsymbol{R^2} = 0.9814$; i.e., the difference is less than 0.2.

Table 11
The optimum flotation conditions.

Factors	Units	Flotation conditions
Flotation time	Minutes	8
Collector type		PAX
Collector dosage	g/t	800
Frother	g/t	MIBC
pH **regulator**	ml	$NaOH/H_2SO_4$
Sulfurizing agent	g/t	Na_2S (1000 g/t)
Particle size	µm	−75 + 54
Agitation Speed	rpm	1000
pH		8

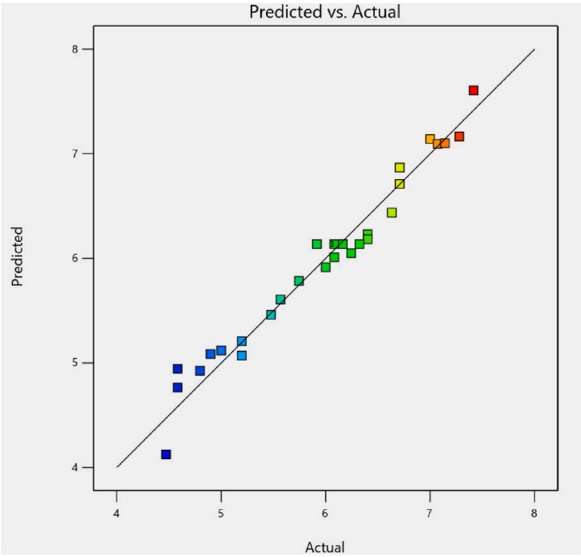


Fig. 11. Experimental value versus predicted values of response.

4. Conclusions

Characterisation analysis illustrated that the copper in the copper slag from BCL is mostly oxides minerals, mainly comprising of Fayalite, chromite and copper-bearing minerals in the form of oxides and traces of sulphides. The preliminary nature of this study was to utilise flotation and the different parameters to attain enhanced/improved grade and recovery of copper and iron. The basis of this nature was achieved and presented in the results and conclusions section.

This study aimed to investigate the use of flotation to upgrade and recover copper and iron from copper slag. The influence of collector dosage, particle size, sulfurizing dosage, pH regulator on copper and iron recovery, and grades, respectively, were discussed. The results showed that the grade of Cu and Fe were upgraded from 0.581 to 0.884 for Cu and Fe from 69.8 to 71.8. The RSM for flotation of the copper slag gives a quadratic model. This model aids in predicting the flotation recoveries as functions of operational parameters. The importance and competence of the model suggested by RSM–CCD are verified by analysis of variance. Copper and Iron recovery are both influenced by the parameters investigated through flotation. The pH and Na_2S dosage relations are pretty

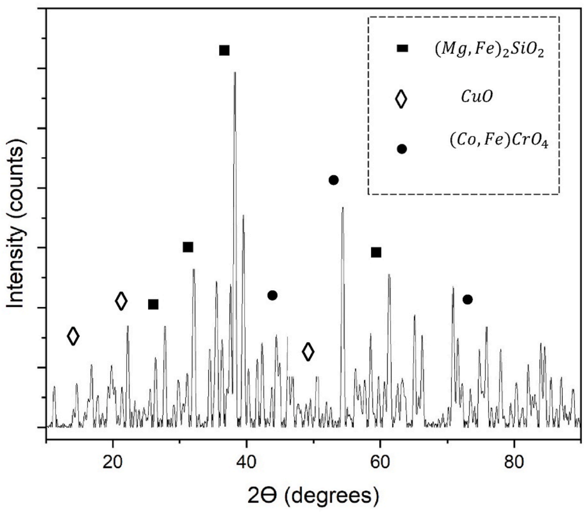


Fig. 12. XRD pattern of flotation concentrate.

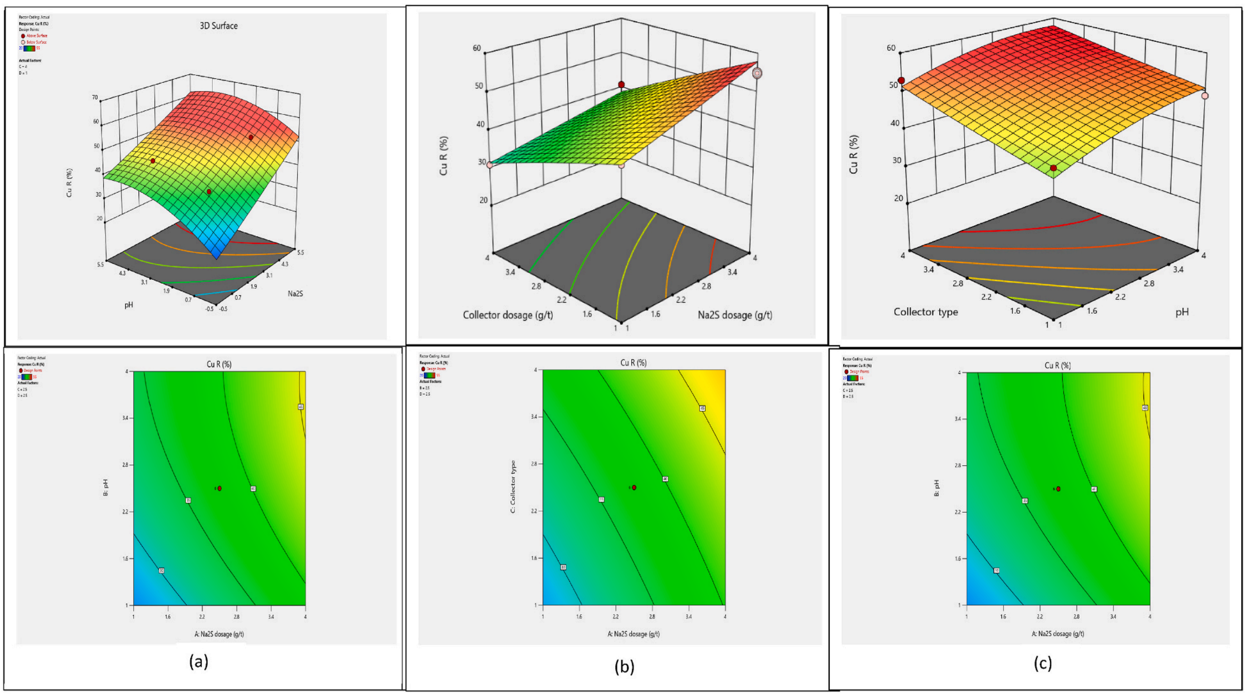


Fig. 13. Response Surface between parameters and Contour plots for Cu Recovery.

substantial. This generated a recovery reaching 62% and 70%, respectively, for Cu and Fe under the following controlled flotation conditions at a pH of 8, sulfurizing reagent (Na_2S) of 1000 g/t, collector (PAX) of 800 g/t, frother (MIBC) of 100 g/t, agitation speed of 1000 RPM and flotation time of 8 min. RSM analysis showed that the Cu increased concerning the addition of sulfurizing reagent, which aids in improving the flotation of Cu Kinetics due to an increment of the froth stability.

4.1. Future work Perspectives

- Addition of specific engineered Sulphidizing agents that could improve the surface area for hydrophobicity to encourage flotation.
- More studies should be performed to ascertain the
- Extra vigorous analytical methods to determine the concentration of sulphur and oxide species to be looked upon.

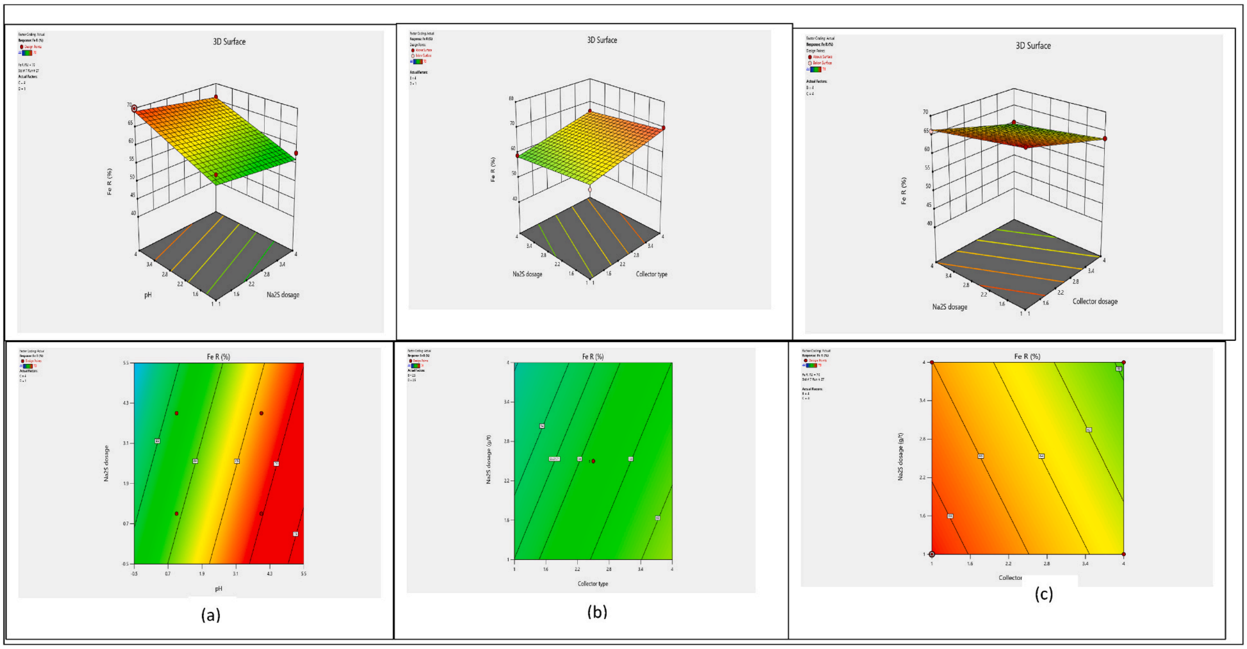


Fig. 14. Response Surface between parameters and Contour plots for Fe Recovery.

Table 12
XRF results for copper slag and froth concentrate.

Element	Fe (%)	Cu (%)	MgO
Cu Slag	69.8	0.581	0.76
Froth concentrate	72.8	0.884	

Funding statement

This research received no specific grant from funding agencies in the public, commercial, or not-for-profit sectors.

Additional information

No additional information is available for this paper.

Data availability statement

Data will be made available on request.

CRediT authorship contribution statement

T. Gabasiane: Writing – original draft, Visualization, Project administration, Methodology, Investigation, Conceptualization. **G. Danha:** Supervision, Methodology. **T. Mashifana:** Supervision, Investigation, Formal analysis. **T. Mamvura:** Validation, Supervision.

Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:Tlotlo Gabasiane reports administrative support, equipment, drugs, or supplies, and statistical analysis were provided by University of Johannesburg. Tlotlo Gabasiane has patent pending to Pending. If there are other authors, they declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgements

This research did not receive any specific grant from funding agencies in the public, commercial, or not–for–profit sectors.

The authors thank the Botswana Concession Limited (BCL) for providing the copper samples. The authors would also like to thank the Botswana International University of Science and Technology and the University of Johannesburg for making their labs available for conducting the experimental work and investigations.

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