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Research article

Characterization of copper slag for beneficiation of iron and copper

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ABSTRACT

Before disposal of any metallurgical waste to the environment, it is the responsibility of mining institutes to adhere to the permissible metal content limits. Base metals, especially iron and copper, have adverse effects of reducing the soil pH and excessive concentrations of these in the disposed waste may result in soil pollution and toxicity, with adverse effects on plant growth and animal health. Copper slag is a metallurgical waste that is disposed by way of stockpiling at designated dump sites within a mining site. The observed depletion of high-grade iron ores in Botswana and the environmental hazards associated with disposal of untreated metallurgical waste, presents an opportunity for research on secondary sources of iron and copper. Our characterization results show that this BCL copper slag is a good secondary source of base metals, especially iron and copper. These results reveal that the elemental proportion of iron was around 35.4%. Literature states that an iron grade that is considered viable for economic beneficiation should be at least 25% and this slag has an iron content above this limit, hence poses a serious environmental threat upon disposal. This article presents an investigation into the mineralogy of the copper slag at a plant situated in Selebi Phikwe, a town in the northern part of Botswana. Quantitative evaluation of minerals by scanning electron microscopy (QEMSCAN) quantified that no cobalt – sulphide was detected and strongly indicated that the cobalt within the sample occurs in solid solution in either the fayalite phase or glass phase. Spot analysis from electron probe micro – analyzer (EPMS) images indicated an unusually high content of copper compared to any other metal. We elucidate that, this was due to the inefficient processing techniques employed during operational years of the mine. The relative compositions of Co, Fe, Ni and Cu were 0.14%, 35.4%, 0.28% and 0.29% respectively. This analysis justifies our interest in considering this copper slag as a secondary source of iron for beneficiation purposes.

1. Introduction

The mining and mineral processing sector plays a major role in the economy of Botswana. The depletion of high grade primary sources of metallic ores [1], has led to the closure of most mines e.g. BCL, Tati Nickel, Lerala Mine, African Copper Mines and Discovery Metals, due to the huge loses encountered in attempting to exploit low grade ores. The resultant socio-economic challenges experienced by the former employees, their families and other down-stream industries is motivation enough for the country to start considering exploiting high grade secondary sources of metallic ores. One such possible source is copper slag. Copper Slags mainly report to the tailings stream from the smelting of a copper concentrate [2]. The slag we used in this investigation was obtained from BCL mine located in the Northern part of Botswana in a mining town called Selebi Phikwe. This slag was classified as a metallurgical waste, hence was disposed by a way of stock – piling into heaps at designated places on the mine site. Besides being a possible secondary source of base metals, this slag poses an environmental threat to the environmental as it was disposed of with high concentrations of these base metals. BCL mine was commissioned in 1973 before it was put under final liquidation in 2017 [3], and its slag was formed from smelting dry feed of copper/nickel bearing concentrate in the Outokumpu flash - smelting furnace. This produced a low grade matte and a high grade slag [4]. The matte was upgraded by being fed into three Pierce – Smith converters while the high-grade slag was treated in the electric furnace for a further recovery of matte.

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Table 1. Production of copper slag from different regions around the world [7, 8].

Regions	Copper slag generation/annum in million ton
Asia	7.26
North America	590
Europe	5.56
South America	4.18
Africa	1.23
Oceania	0.45

At present, roughly 80% of the global copper production is from sulphide ores [5]. Copper needs to be concentrated to separate it from the gangue material in the ore by a pyro – metallurgical process which requires high temperatures for the reduction to occur. This process produces significant amount of the tailings that contain a bulk of other base metals. Most of these tailings contain between 30–40% iron and 35–40% silica, less than 10% alumina & oxidized calcium and 1% of copper [5, 6].

Globally a large volume of copper slag (Table 1) is produced which can be approximated to 2.2 tons of slag generated for every ton of copper produced [9]. The annual worldwide production of slag is approximately 24.6 million tons in the early century [6, 7] and increasing at a rate of 11% per year [6]. Table 1 illustrates the magnitude of slag produced in different areas in the world and Botswana contributes to this statistics. The aim of this article is to investigate how copper slag produced from BCL mine can be exploited as a secondary source of metals, while achieving the objective of reducing the concentration of these metals from the slag hence making it safe to dispose of the environment. This slag is concentrated mostly with metals that were not extracted during operational period of the mine but that can now be beneficiated by employing modern day technological advancements. There is also an environmental based motivation associated with processing this slag, brought about by the fact that the concentrated metals over time percolate into the ground, thereby modifying the soil composition and water table [10]. This puts lives of villagers and their livestock at risk. The slag stockpiles are also occupying a huge piece of vacant land that could have been used for valuable projects such as farming but is now used only to store metallurgical waste [11].

1.1. BCL mine

BCL mine over the years gave life to more than 5000 people living and working in this small mining town. The closure of the mine brought about a sense of hopelessness to the residents of Selibe Phikwe. The subsequent closure of a steel making plant called Pula Steel in the same town due to shortage of its main raw material (scrap iron) literally turned the town into a ghost town. The main purpose of this research is to try and find a pragmatic way to use the huge waste (slag) accumulated at BCL mine as a source of secondary ore for iron that can be extracted and used as ferrous supplement to Pula Steel feed, thereby resuscitate the steel making plant. Utilization of copper slag with the view of recovering valuable metals [12] such as copper and iron prior to disposal, helps in keeping the environment safe [2]. The other base metals in the slag can be processed at BCL with the intention of industrializing the town back to life.

1.2. Objectives

The objective of this present study is to characterize the BCL copper slag and analyze its composition properties. In the past, research has been focused on ferrous and non-ferrous slags with the intention of employing the findings in various applications [13]. We had to carry out the characterization task first in order to quantify the composition of the base metals in the slag, especially that of iron. In order to achieve this, we used the Scanning Electron Microscope (SEM), X – Ray Diffractometer (XRD), Inductively Coupled Plasma (ICP) and Electron Probe Micro Analyzer (EPMA) [14, 15]. Much as these techniques are important, a more comprehensive investigation on the distribution of target metals in the slag is vital for the design of a personalized slag treating process [16]. A lot of information can be gained from characterization as it can be elaborated that characterization of copper slag can aid in concluding probable concentrate grade and recovery of metals [17]. This in turn helps in designing flow sheets involved in the separation steps in a sustainable manner [18]. Copper slags may differ in their respective composition, morphology and mineralogy due to their difference in origin, techniques used in the recovery of primary target metals (previous metallurgical processes) as well as chilling techniques. In previous investigations [19], on the composition and phase of copper slag, it was found that indeed there are viable metals such as cobalt, nickel, copper and iron in slag that can be recovered.

2. Materials and methods

2.1. Materials

Copper slag from BCL mine was sampled and used in the experiments as feed material. BCL mine is located in Selibe Phikwe town found in the Central District region and situated in the South East part of Francistown. The slag was generated from BCL Outokumpu flash furnace during the refining and smelting of their concentrate. Slag formation varies depending on the way it is cooled, and BCL copper slag was cooled rapidly (using the water granulation process). As the slag has undergone rapid cooling, it has irregular small shapes of 1–4mm with black glassy luster. The characterization tests were carried out with the aim of recovering iron, and any other associated valuable metals e.g. cobalt.

2.2. Sample preparation

The sample was split into two representative samples. The first representative sample was liberated (crushed) to 100% passing -1mm. The representative sample was then split again and pulverized for XRD and Chemical analysis. The second representative sample of 50 g from the crushed material was again split and pulverized for XRD and Chemical analysis.

2.3. Characterization methods

The copper sample was chemically analyzed using an XRD borate fusion and ICP sodium peroxide fusion, including base metals. Mineralogical analyses carried out on the sample comprised of quantitative XRD and QEMSCAN Bulk Modal Analysis (BMA) and QEMSCAN Particle Map Ana lysis (PMA). These analyses would provide elemental deportment, mineral liberations and associations as well as grain size distribution for Co, Cu, Ni and Zn – bearing phases. Further elemental analyses comprised of Electron probe microanalysis (EPMA) providing elemental compositions for the Cu, Co, Ni and Zn – bearing phases and silicate phases.

The samples were tested for Major minerals by quantitative XRD and QEMSCAN bulk Analysis (BMA).

3. Results and discussions

3.1. Mineralogical analysis

Table 2 presents the results for mineralogical analysis tests. Iron was found to be the dominant component in the copper slag and this observation agrees with other investigators [12, 20].

Table 2 gives the abundance of the minerals from XRD spectrum. The iron is present in crystallized form as fayalite (Mg, Fe) $_2$ SiO_4 and chromite (Co, Fe) CrO_4 and this happens during the matte smelting process or

Table 2. Mineral phases in the copper slag, determined by XRD.

Mineral	Chemical Formula	Approximate Abundance (%)
Fayalite	$(Mg, Fe)_2\ SiO_4$	10–20%
Chromite	$(Co, Fe)\ CrO_4$	<3%
Amorphous Content		80%

water cooling. Crystal phases can also be observed in Table 2 and Figure 3 as morphologies characteristics.

Figure 1 shows the X – Ray Diffractogram (XRD) spectrum of the copper slag sample. It illustrates that the major crystal phases in the slag are chromite (Co, Fe) CrO_4) and fayalite (Mg, Fe) $_2$ SiO_4, as seen from the XRD pattern analysis (Figure 1), Scanning Electron Microscope (SEM) and EPMA (Spot analysis) images are shown in Figures 2 and 3 respectively. These results are similar to those previous researchers as [12, 21] illustrating crystalline phase composition comprising mostly fayalite ($2FeO.SiO_2$)and magnetite (Fe_3O_4).Figure 2 also illustrates the existence of chromite which shown by the peaks in the XRD curve (peaks). This indicates that it may prove somewhat difficult to directly iron using physical separation technique, hence extensive review carried out to try and manipulate methods of extracting the iron.

3.2. Mineralogical analysis using QEMSCAN

QEMSCAN was used to determine the distribution and portions of iron bearing phases [22] and Table 3 shows results from the QEMSCAN. Results from the scan indicate the different phases present in the copper slag with respect to the approximate abundance present in those phases. We can refer from Table 3 that most of the iron is present in the fayalite phase and glass phase at 12.85 and 85.19% respectively and this agrees with previous research [8].

The results are in agreement with those from XRD showing presence of chromite fayalite and the amorphous (in this case glass) phase. The addition here is the sulphide at 1%.

Scanning Electron Microscope (SEM) results provided images having spot analyses which can be identified from Figure 2.

The iron can be seen from the spot identified as a compound of Fe–Cu–Ni sulphide with Fe being dominant as it has concentrated due to being not the target recovery metal at the core process of the mine. The compound containing the Fe is spread throughout the glass host and can be judged by the concentration on the magnitude of the spots on the image. On Figure 2(b), the Fe–Cu–Ni sulphide is higher embedded in the copper slag. Previous researchers have similar results which highlight that fayalite are present in the glass host with sulphide phases illustrated with bright spots $Fe - Cu - NiSulphide$ [22]. This shows a detailed study of the distribution of copper bearing phases and this concludes that Sem is more adaptable to be used for characterization of minerals in small amounts.

3.3. Chemical analyses results

Spot analyses (Figure 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

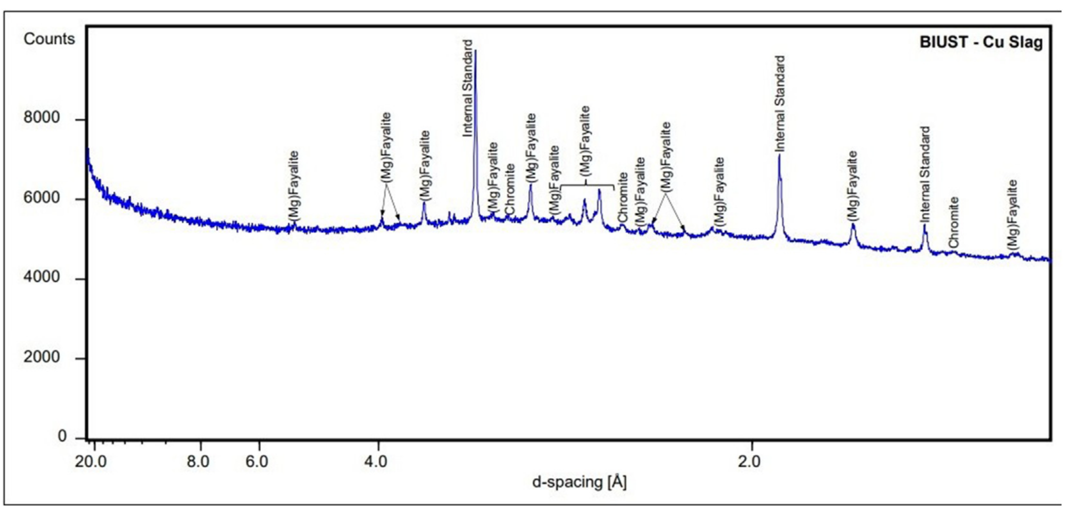

Figure 1. X – Ray diffractogram of BCL Cu slag cooled rapidly with water.

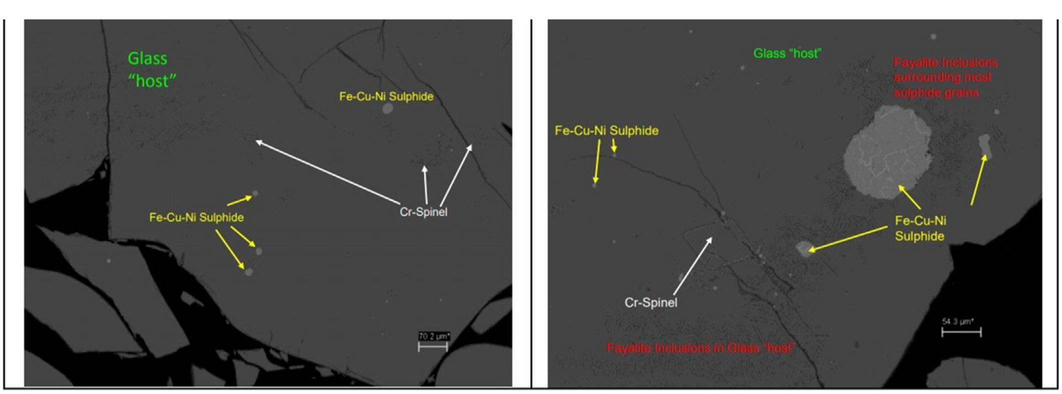

Figure 2. SEM imagine showing the minerals detected showing components of the BCL slag at different regions.

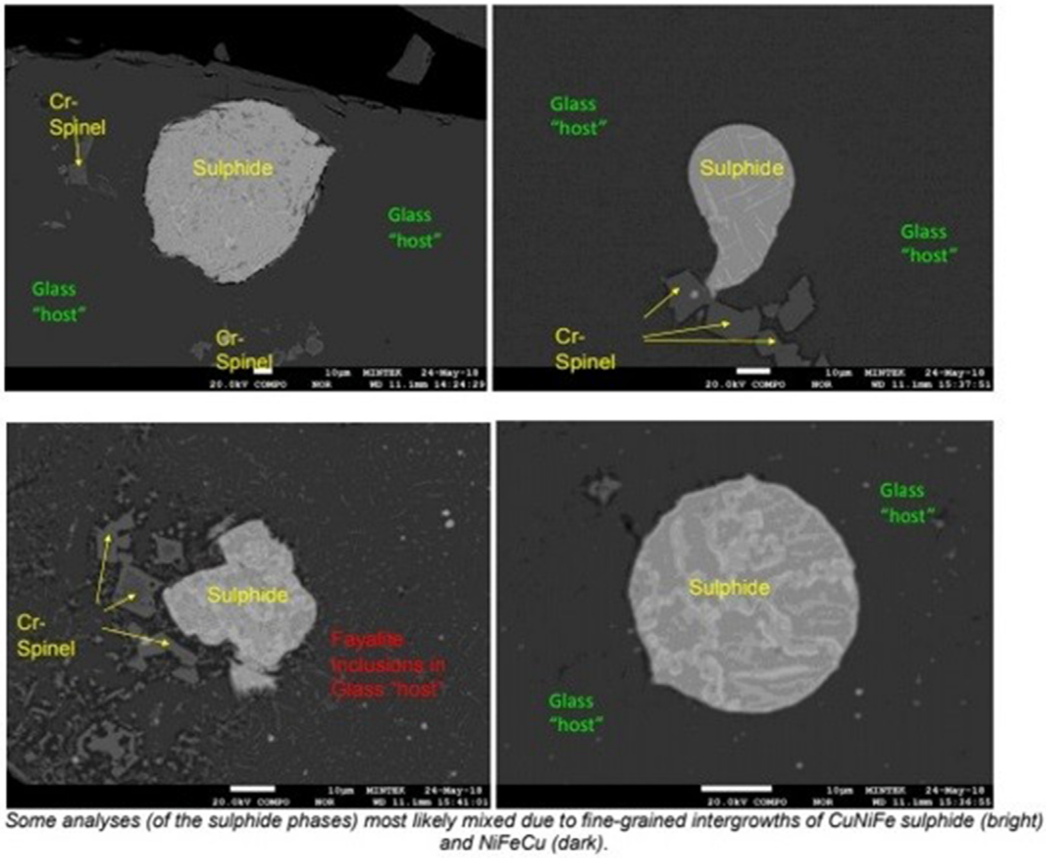

Figure 3. EPMA images showing the nature of occurrence of the detected sulphide and Cr - spinel phases within the glass host.

in high Ni content, while the lighter areas showed consistency in high Cu content.

Figure 4 further elaborates on the presence of fayalite within the glass host of the slag with the bright cent red colour suggesting presence of sulphide. This phenomena is well within research results with [23] having similar results having a concentration between that of the glass and Cr – Spinel. Figure 4 shows EPMA images revealing the nature of occurrence of the detected crystalline fayalite phases within the glass host.

3.4. Elemental analysis of copper slag using XRF & EPMA

Table 4 presents the chemical composition of the copper slag from XRF and EPMA. The results are in agreement with those of [23] indicating a high accumulation of metal elements in the slag with iron being the most abundant at 42 wt% followed by silicon at 14 wt%.

EPMA was used to analyze liquid phases and primary solid phases of copper slag and Table 4 shows results of composition of elements in these different phases. Each phase has its own characteristics that may require different approaches in order to unlock the target mineral elements for possible recovery. Table 5 also shows elemental composition but of the bulk sample and not in phases. EMPA results from Table 5 indicate that Fe is the most abundant element in all the phases present from the Cr – Spinel to Glass. According to EPMA results droplets of nickel and most of the metal elements were not present except for Fe, Cu and S and this also can be verified with previous research as [24] has similar results with respect to EPMA.

Table 5 shows XRF results and clearly indicates that the relative proportion of iron is predominant at 35.5% greater than all the other metallic elements. The other metal of interest includes cobalt, nickel and copper which have a relative proportion of 0.14%, 0.28% and 0.29 % respectively. Previous researcher agrees with these findings as [17] also stipulates to having higher iron content of 42.2% making it worth looking at. The XRF data from Table 5, have two different analysis techniques which are XRF77R and XRF79V. The XRF77R is used to analyze single elements such as Fe, Co and Ni while XRF79V is used for a combination of elements (oxides) such as CaO and MgO.

3.4.1. Possible treatment routes

The BCL Cu slag has mainly been oxides as the main treatment at the plant involved using a furnace to the ore which formed the slag as a byproduct. Previous researchers have different ways of approaching treating slag depending on the nature of the slag and how the slag was formed. There are two mains processes being pyro and hydrometallurgical processes to process slag [25]. outlines from their research the development of recovering cobalt from slag using a DC arc furnace due to the slag in Zambian mine being non – leachable and having a glassy component and this a pyrometallurgical route. This requires high temperatures for treatment and can be achieved in the DC arc furnace. All slags go through characterization stag to know the characteristics of the slag and how to approach treatment stage, and some illustrate that most of the valuable metal of interest are in the sulphide phase hence

Table 3. Bulk modal mineralogy for the Cu slag, as determined by QEMSCAN BMA.

Mineral	Approximate Formula	Approximate Abundance (%)
Glass Phase	(Al–Ca–Cu–Fe–K–Mg–Na–O–S–Si)	85.19
Fayalite	$(Mg, Fe)_2 SiO_4$	12.85
Chromite	$FeCr_2O_4$	0.90
Sulphide	Cu_xS_y	1.05
Total		100.00

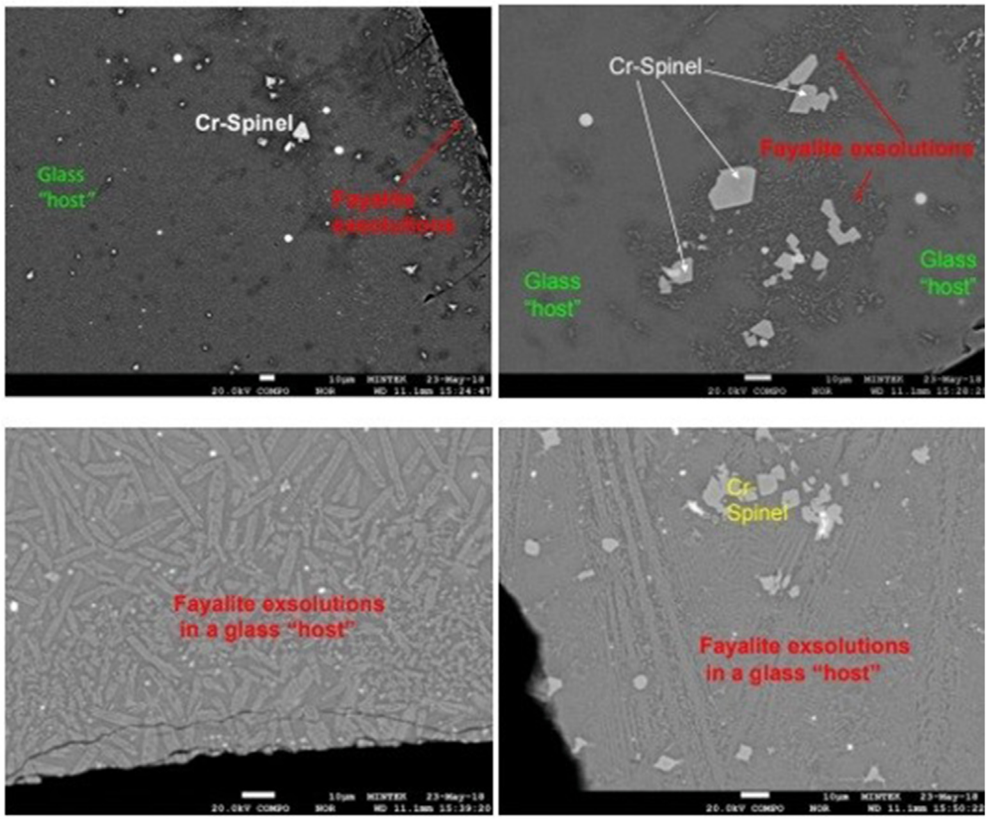

Figure 4. EPMA images showing the nature of occurrence of the detected crystalline fayalite phases within the glass host.

Table 4. Elemental compositions in the Cu slag, as determined by EPMA.

Phase	Approximate Elemental Abundance %										
	Al	Ca	Cr	Cu	Fe	K	Mg	Na	O	S	Si	Ni
Cr - Spinel	7.50	0.16	21.94	0.13	38.86	0.12	1.88	-	29.89	0.14	0.96	-
Sulphides	-	-	-	17.24	21.65	-	-	-	-	27.89	-	32.86
Glass 1	4.14	2.33	-	0.17	34.48	0.76	2.47	0.81	36.99	0.86	16.78	-
Glass 2	3.58	1.91	-	0.24	38.52	0.77	2.04	0.74	35.42	0.68	15.44	-
Glass 3	3.17	1.88	-	0.29	40.17	0.69	2.22	0.69	34.82	0.70	14.73	-

Table 5. XRF results for the copper slag.

Element	Fe (%)	Co (%)	Al_2O_3 (%)	Cu (%)	CaO (%)	Ni (%)	MgO (%)	Cr (%)
Method	XRF77R	XRF77R	XRF79V	XRF77R	XRF79V	XRF77R	XRF79V	XRF77R
Cu slag	35.4	0.14	7.09	0.29	2.67	0.28	3.53	0.17

rendering flotation as the route of concentrating the valuable metal (hydrometallurgical route) [8] and some of these metal elements include copper and nickel. This paper focuses on analyzing the content of BCL slag to draw a suitable process route for the metal elements worth recovering. The slag comprises of Cu, Ni and Fe with the copper and nickel existing in the sulphide phase and fayalite phase. A combination of pyro and hydro metallurgical process to recover the metals with respect which phases they exist in and characteristics they poses.

Copper can either exist in sulphide or oxide phase but in smelter slag copper is found to exist in sulphide phase with [26] indicating that copper in sulphide phase was 0.81 (wt.%) whereas in oxides was (0.15) hence making it more viable to concentrate the copper in the sulphide phase. This conclusion is similar from the slag from BCL which shows most of the copper was found in the sulphide phase. Most mines around the world which mine copper evolve over time to optimize the plants efficiency but with BCL mine it used old conventional recovery without taking into account beneficiation of slag hence the slag has concentrated copper in the slag since the inception of the mine. The most viable route to recover copper from sulphide phase is flotation using chemical reagents which previous researchers such as [2, 12, 27] further elaborates on using a two-stage flotation process to recover copper and get 80–87% with iron being further concentrated by a specified technique of molten slag added complex additives. The conditions of the BCL slag are in similar range as of previous research and in some of much higher concentration in terms of iron.

BCL slag contains both copper and iron which are of interest and subsequently copper as mentioned can be recovered using flotation and iron be recovered using magnetic separation [9]. talks about past work on recovery of iron from copper slag using direct reduction – magnetic separation which yielded iron concentrate of 90.68% assay. The BCL slag is similar, being an oxide needs to be reduced using a reductant. One of the goals for recovering iron from BCL is to feed the Pula steel making plan and the final product can be used as scrap steel which will be suitable.

4. Conclusions

A study of copper slag smelting indicates that there is value in characterizing copper slag with view of recovery of viable metals, especially iron. Analysis from the results showed that nickel, iron and copper existed in high amounts in the slag with these elements found in the fayalite and chromite phases. Three mineral phases were detected namely; fayalite, chromite and sulphide. XRD analysis established that the sample contains >80% amorphous (non-crystalline material) material. The amorphous material was established to be the glass phase. The elemental composition of the glass phase was established by EPMA. In addition to the glass phase, EPMA also detected Cr – spinel and sulphides in the sample. The sulphide occurs as blebs of Fe–Cu sulphide and Fe–Ni–sulphide intergrowths occurring as very fine occlusions within the glass. The fine grain size will dictate ultrafine milling to liberate the sulphides for possibility upgrading by flotation. In conclusion, we elucidate that this copper slag is a viable source of iron, which can be extracted and used as feed material in other processes. It can be concluded that iron can be recovered by Direct Reduction process, the iron into iron pellets and later be separated by magnetic separation.

Declarations

Author contribution statement

T.S. Gabasiane: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Wrote the paper.

G. Danha: Conceived and designed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

T.A. Mamvura: Conceived and designed the experiments; Analyzed and interpreted the data; Wrote the paper.

T. Mashifana & G. Dzinomwa: Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

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Data availability statement

The data that has been used is confidential.

Declaration of interests statement

The authors declare no conflict of interest.

Additional information

No additional information is available for this paper.

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