

The Kinetic Dissolution of Copper from Chalcopyrite-containing Carbonatite Tailings Samples in Sulphate Media

Brad Barlow, Elvis Fosso-Kankeu*, Kolela J Nyembwe, Frans Waanders and Edward Ntumba Malenga

Abstract— The aim of this study was to determine the rate at which Cu is extracted from the surface of chalcopyrite contained in the tailings from the carbonatitic ore obtained from the carbonate complex in Looлекop, Phalaborwa, South Africa; the rate formation of the passivation phenomena during mineral dissolution was also observed. The objectives of the study, in which the mineral was subjected to sulphate based leaching with ferric sulphate as oxidant were as follows: firstly, to observe the effects of room temperature conditions (25°C) on the extraction rate of Cu in the carbonatite tailings system and secondly to determine the effects of mineralogical composition of species within the host ore on the extraction rate and passivation rate formation. The tailings was subjected to X-ray fluorescence spectroscopy (XRF) for mineralogical characterization as well as X-ray diffraction spectroscopy (XRD) for bulk elemental composition. Phreeqc simulation software was used for the prediction of mineral species using acidity, alkalinity, chloride ion, pH, ORP and sulphate ion as input parameters. The tailings associated with calcite-magnesium, hematite and quartz was prone to acid attack at room temperature conditions during the initial phases of leaching. During the course of the set leaching time, accumulation of gypsum and jarosite occurred, disrupting the transfer of ions from the mineral surface as 5.55wt% Cu extraction was obtained after the leaching period (12 hour) as opposed to the peak extraction rate after 4 hours of leaching (16.58wt%).

Index Terms—chalcopyrite, tailings, ferric sulphate media, mineralogy, temperature.

I. INTRODUCTION

Leaching copper from low grade chalcopyrite ores are in high demand as other secondary copper sulphides on shallower depth zones are depleting [1], furthermore, chalcopyrite (CuFeS₂) accounts for 70% of the world's copper reserves

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further promoting the extraction of copper from this mineral. Pyrometallurgical extraction of copper accounts for 80-85% of copper extracted from CuFeS₂ but is less desired as it causes high sulphur dioxide (SO₂) emissions [2]; alternatively hydrometallurgical extraction of copper from CuFeS₂ also exist which is more cost effective than the former as extraction requires high capital cost smelters and refineries for treatment processes where only small scale leaching plants are required for the latter [3]; better waste management strategies is also characteristic of hydrometallurgical extraction. Despite this hydrometallurgy only accounts for up to 18% of Cu production as leaching rates of CuFeS₂ are low as a result of a passivation film forming on the surface of the mineral [4][5]. Many studies have focused on identifying the exact cause of the formation of the passivation film but no general accepted theory has been identified as of yet [6][7]. However, some of the most popular theorized mechanisms for the formation of the passivation layer are elemental sulphur enveloping the surface of the mineral, preventing contact of reactants with the mineral surface, metal deficient polysulphides forming on the surface of the mineral and the presence of jarosite which slows leaching kinetics by impeding mass transfer of ions from the mineral surface [8][9][10][11]. Although the exact mechanism for the formation of the passivation film has not been identified, many parameters effecting the recovery rate of copper have been studied and identified. Additive addition, particle size distribution, oxidation redox potential, solution pH and reaction temperature are but a few of these parameters [12][13][14]. For the purpose of this study the effects of mineralogical composition and solution pH are considered. Carbonatite tailings from PMC's Rio Tinto mine in Phalaborwa, RSA are used in this study where the highest grade of Cu (1% copper) is present at the core of the carbonatite complex; carbonatite complexes can be divided into two separate groups, these being alkaline-carbonatites and mineralized carbonatites with the former being of particular interest as they are identified as a noteworthy source of copper [15]. The alkaline-carbonatite complex in Phalaborwa is however geologically unique as it is the only carbonatite complex in the world that is constituted of enough copper sulphide minerals to offer economically feasible operations [16]. The tailings mineralogical composition will differ from the run of mine hoisted from the mine as during flotation of the ROM, phase mutation is said to occur [17]. The effects of the new formed mineral phases on the extraction rate is observed in this study.

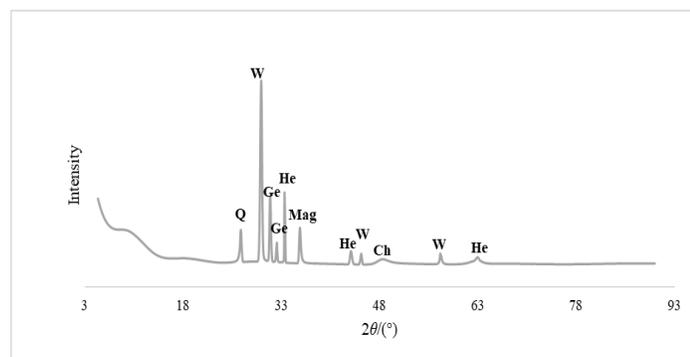
II. EXPERIMENTAL SETUP AND MODELLING

A. Materials

Similar tailings samples as was earlier characterized by Nyembwe et al [18] was used in this study. The samples were provided by the Phalaborwa Mining Company (PMC) and received from their Rio Tinto mine in Phalaborwa, RSA. Through XRF the chemical compositions of the major and trace minerals were exposed as available from Table I; the mineralogical content of the samples is presented in Fig. 1: Diffraction spectrum of tailings mineral

TABLE I: CHEMICAL/ELEMENTAL CHARACTERIZATION OF CARBONATITE TAILINGS MINERAL

Bulk stream chemistry (XRF)	
Element	Composition (%)
Al	1.63
Ca	47.5
Cu	0.47
Fe	32.6
Mg	6.38
P	2.47
Si	5.18
Sr	1.08



Ch: chalcopyrite (0.47wt %); Ge: Gehlenite (5.78wt %) H: hematite (10.07%); Mag: Magnetite; Q: quartz (12wt %); W: Calcite magnesium carbonate (73wt %)

Fig. 1: Diffraction spectrum of tailings mineral

B. Chemical leaching

For the purpose of leaching the carbonatite tailings mineral samples, an incubator with a built in orbital shaker was used. The incubator acted as a containment chamber used to prevent contamination of the solutions during experimentation as well as to keep both the temperature and rotational speed (200 rpm) stable. 500 mL volumetric flasks were used to prepare a 10% mass by volume solution; the liquid solution was prepared using distilled water, sulphuric acid and ferric sulphate to produce a ferric sulphate solution of 0.1 M [6]. Rotational speed for the orbital shaker was stable at 200 rpm whilst temperature was set to 25°C. Each of the solutions was prepared at a pH value of 0.5-1 after which pH evolution was allowed to occur over the course of the 12 hours leaching test. The dissolution kinetic information was obtained as 30 mL sample was withdrawn from the leaching vessel hourly and was analysed for its dissolved metal content using inductively coupled plasma optical emission spectroscopy (ICP-OES). Prior to that, the withdrawn

slurry was filtered using 150 mm filtration paper. Lastly the filtered residue was then dried, stored in a desiccator and were subjected to mineralogical and chemical characterization using X-ray diffraction (XRD) and X-ray fluorescence (XRF) as previously described [19-40].

C. Leachate analyses

Leachate samples obtained after leaching chalcopyrite solutions were characterized by measuring pH, oxidation redox potential (ORP) and temperature. For the measurements of pH, temperature and ORP (for each leachate solution) a portable pH meter with an analytical electrode HI8424 (Hanna Instruments Inc.) was used. Samples were also measured for acidity, alkalinity, chloride ion concentration, sulphate ion concentration. For sulphate ion measurements, a COD and Multiparameter Photometer HI 83099 was used.

D. Titration tests

The alkalinity was measured through titration method with 0.1N sulphuric acid (H_2SO_4) where the solution was titrated from pH values lower than 8.3 to 4.5 using an indicator for clear endpoint visibility. The acidity was measured through titration method with 0.02N sodium hydroxide (NaOH) where 0.02N sodium carbonate (Na_2CO_3) was used as standardization procedure. For chloride ion concentration, titration was conducted using silver nitrate ($AgNO_3$) and potassium dichromate (K_2CrO_4) as indicator [41].

E. Speciation modelling

To determine the aqueous speciation of the major metal ions in the leachate of the carbonatitic tailings, AQUACHEM software was used interfaced with PHREEQC (program version 3.3.12-12704.) modelling software [5][41]. The phreeqc database, Minteq.v4.dat was used for the purpose of this report. The ORP had to be adjusted with a correction factor before it could be used as input data. The correction factor was obtained from Field Measurements of Oxidation-Reduction Potential [42]. The corrected ORP value was then used to calculate the pe value using the following equation:

$$pe = E_h / (0.059) \quad (1)$$

pe donates the negative logarithm of the electron activity.

Eh donates the oxidation redox potential of the sample.

To predict the speciation of the major metals present within the leachate of the chalcopyrite bearing minerals the following input data was used within the phreeqc workbench interface: Acidity, alkalinity, pH, pe, temperature (°C), ICP data (metal ion concentrations), Cl^- and SO_4^{2-} .

III. RESULTS & DISCUSSION

A. pH variation

Fig. 2: pH behavior of tailings samples at varying time

displays the pH evolution of the carbonatite tailings samples at room temperature conditions:

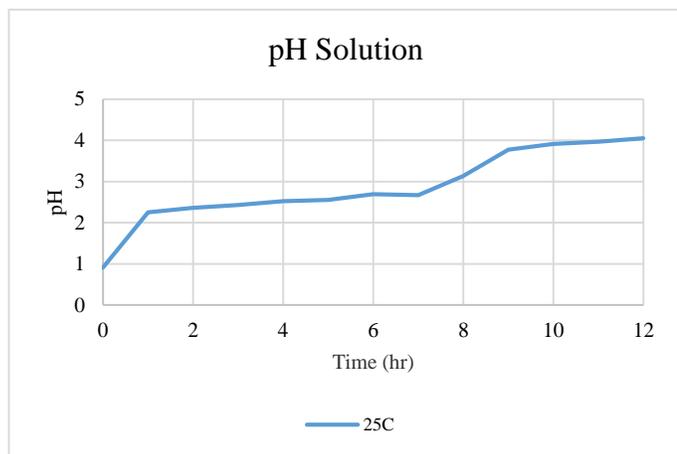


Fig. 2: pH behavior of tailings samples at varying time

The evolution during experimentation under room temperature conditions (25°C) was generally acidic but observed to gradually increase during the set leaching time as higher concentration of Ca is released in solution. The tailings is mainly constituted of calcite magnesium (Fig. 1: Diffraction spectrum of tailings mineral

); Ca is used to reduce soil acidity as Ca^{2+} ions reduce H^+ ions in solution which neutralized the solution pH [43]. The same effect are assumed to occur during mineral dissolution as the high Ca content (Table I) releases Ca^{2+} ions leading to the observed pH evolution phenomena. Acid attack plays a vital role during the early stages of leaching at room temperature conditions as the formation of calcium salt (CaSO_4) is believed to occur due to the high H_2SO_4 concentrations at these conditions [44]. Insoluble salt species of Ca is therefore likely the dominant mineral species at the early stages of leaching (first 4 hours) which aids in stabilizing acidic conditions.

B. Extraction behavior at room temperature

Fig. 3 displays the recovery rate of Cu and Fe during leaching in sulphate solution using ferric sulphate. After the set leaching time of 12 hours, extraction rate of 5.55% Cu from the mineral surface was obtained. Rapid Cu withdrawal was observed during the first 2 hours of leaching which can be attributed to the acidic pH conditions observed at this time, where the effects of acid attack on Ca in solution are the highest. The peak extraction rate of 16.58% was observed after 4 hours dissolution as the pH conditions were favorable at this time; this agrees with previous findings according to which the dissolution of CuFeS_2 is identified as an acid consuming reaction [45]. The recovery of Cu is reduced after 6 hours dissolution with increasing pH conditions likely due to higher concentration of Ca^{2+} ions cumulating in solution. From the graph no plateau stage is observed indicating that, although the rate of mineral dissolution displays retarding behavior, mineral passivation has not yet been reached.

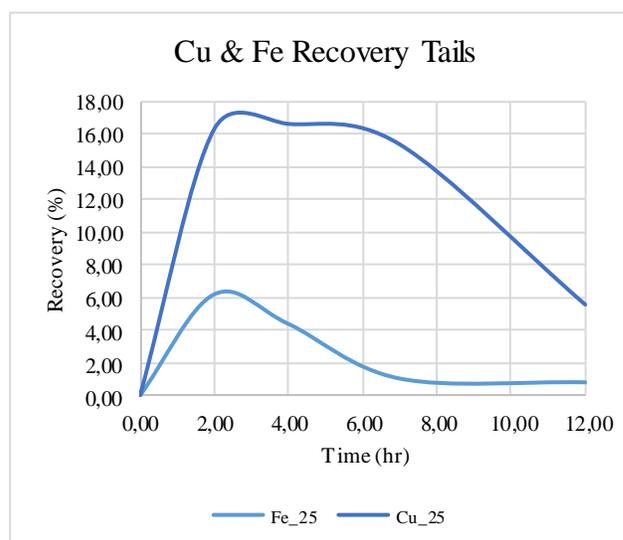


Fig. 3: Recovery of Cu & Fe during mineral dissolution

Fe extraction was also influenced by an increase in the pH conditions. Fe displays similar behavior to that of Cu indicating that during acid attack of hematite (the main constituent of Fe in the carbonatite tails solutions), Fe is more likely to form ferrous iron (Fe^{2+}). From previous studies it was observed that Fe is preferentially released in solution [46] before Cu as Fe^{2+} ions compete with Cu^{2+} ions; competition of Cu with Fe could reduce the concentration of soluble species in solution which contributes to the observed extraction behavior.

C. Speciation results

Table II displays the speciation results for the tailings samples obtained using phreeqc simulation software and were accurately predicted within a 10% error threshold:

TABLE II: CARBONATITE TAILINGS STREAM SPECIATION RESULTS

Element	Species	Carbonate tailings (%)			
		120 min	240 min	420 min	720 min
Ca	CaSO_4	53.28	52.00	50.42	50.52
	Ca^{2+}	46.74	48.03	49.61	49.46
	CaOH^+	<1	<1	<1	<1
Cu(2)	CuSO_4	50.73	50.59	52.16	53.43
	Cu^{2+}	49.27	49.41	47.87	46.55
	CuOH^+	<1	<1	<1	1
Fe(2)	Fe^{2+}	70.34	69.43	68.23	67.61
	FeSO_4	29.66	30.59	31.77	32.39
	FeHCO_3^+	<1	<1	<1	<1
	+				

As seen from Table II, a reduction in the formation of calcium salt species (CaSO_4) along with an increase in calcium ion (Ca^{2+}) occurs over the course of the leaching period (12 hours), evident from the pH evolution behavior (Fig. 2) as higher concentrations of Ca^{2+} free hydrated ionic species compete with hydrogen ion to stabilize solution pH [46]. The

speciation data for Cu species also agree with the observed recovery behavior (Fig. 3) where the highest concentration of soluble free hydrated cupric ions (Cu^{2+}) were the greatest after 4 hours of leaching and observed to reduce as leaching progressed; additionally an increase in insoluble Cu salt species (CuSO_4) were promoted as leaching progressed together with an increase in pH conditions. Higher concentrations of soluble ferrous ion (Fe^{2+}) species was available but identified as reducing in concentration over the set leaching time as higher concentrations of insoluble Fe salts (FeSO_4) formed

D. Mineralogical investigation of solid residue

The mineral content for the tailings residue samples for the complete leaching period at room temperature conditions is displayed in Fig. 4 with mineral content summarized in Table III:

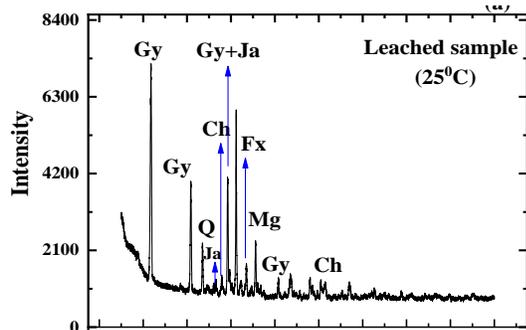


Fig. 4: Diffraction spectrum of tailings residue at 25°C

Progressive dissolution of the CuFeS_2 mineral from the carbonatite tailings host ore was observed for the measured temperature conditions as a decrease in major peak intensity was observed from the initial feed indicating that mineral dissolution had occurred [47][52]. Additionally, new mineral phase formation of Cu species was identified during the course of the leaching period; these intermediate phases being bornite, chalcocite and covelite. Iron mineral phases of hematite (He), magnetite (Mg) and jarosite (Ja), linked to the dissolution media, were also formed during chemical leaching of the tailings mineral; furthermore, precipitates of gypsum, associated with the carbonatite hosting ore, was also observed.

TABLE III: MINERAL COMPOSITION OF CARBONATITE TAILINGS RESIDUE SAMPLES

Mineral phase	Chemical composition	Mineral Concentration (%)	
		25°C	Feed
Bo	Cu_5FeS_4	0,09	***
Ch	CuFeS_2	0,58	0.88
Cx	Cu_2S	0,29	***
Co	CuS	0,90	***
Gy	$\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$	63,30	***
He	Fe_2O_3	1,06	10.4
Ja	$\text{XFe}^{3+}_3(\text{OH})_6(\text{SO}_4)_2$	4,96	***
Mg	Fe_3O_4	10,78	***
Q	SiO_2	16,99	12.00

IV. CONCLUSION

The purpose of this study was to observe the dissolution kinetics of the tailings stream minerals with particular focus on the effects of mineralogical composition of the new formed phases from concentration of the Rom material on the leaching kinetics. The major compositional mineral (calcite-magnesium [73wt%]) from the host ore had a big impact on the leaching kinetics as the speciation prediction showed an increase in free hydrated calcium ion (Ca^{2+}) which reduced the solution acidity as these ions compete with H^+ for dissolution. As concentration of Ca increases, the formation of gypsum at these conditions are further promoted. The reduction in potential and increase in pH also further promoted the formations of ferric salt species during the course of the set leaching period. At these conditions, the dissolution of Cu could be hindered as the surface of the mineral is enclosed by a matrix mixture of gypsum and jarosite that impedes the transfer of ions from the surface of the mineral.

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