

# Chalcopyrite Dissolution in and Fe Speciation Sulphate System pH: 1.0 at Room Temperature

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**Abstract**—In attempt to provide insight on the chemistry of dissolution of chalcopyrite, a carbonatitic copper concentrate was dissolved for 12 hours in ferric sulphate (0.05 mol/L) solution of pH 1.0 at room temperature and atmospheric pressure, in a stirred Erlenmeyer flask with 10% solid. The results revealed a low Cu recovery (<12%) however the solution speciation revealed that the extraction of Cu from the mineral does not only depend upon the nominal  $\text{Fe}^{3+}$  and  $\text{Fe}^{2+}$ , other Fe species including  $\text{Fe}(\text{H}_2\text{SO}_4)^{2+}$  play a major role during the withdrawal of Cu from  $\text{CuFeS}_2$ . However,  $\text{Fe}^{3+}$  followed by  $\text{Fe}(\text{HSO}_4)^+$  remains the principal Fe specie responsible for Cu dissolution. In addition to that, the solid leached residue showed the presence of new Cu phase: bornite, chalcocite and covellite mineral.

**Keywords**—Chalcopyrite, dissolution, Ion speciation

## I. INTRODUCTION

About 85% of the worldwide metallic Cu is extracted from the ore via pyrometallurgical route [1], which involves the use of reverberatory furnace techniques or flash smelting technologies. Pyrometallurgy is the most effective and rapid process of Cu extraction. However, this process is becoming less economic due to ever increasing stringent environmental regulations and concerns of  $\text{SO}_2$  emissions, on hand; while on the other hand the relative decrease in profit margins for mineral processing caused by a scarcity of high-grade ore bodies [2]. Consequently, hydrometallurgical route appears to be more economically attractive for production Cu metals from  $\text{CuFeS}_2$  minerals than pyrometallurgical [3,4]. This route involves extraction of mineral from the ore through leaching with aqueous solvents (aka medium) under specific and controlled conditions.

Some of the media used in dissolution of  $\text{CuFeS}_2$  include: sulphate media, which requires the presence of ferric sulphate ( $\text{H}_2\text{SO}_4\text{-Fe}_2(\text{SO}_4)_3$ ); chloride-based media ( $\text{HCl-FeCl}_3$ ); mixed chloride-sulphate media ( $\text{H}_2\text{SO}_4\text{-Fe}_2(\text{SO}_4)_3\text{-NaCl}$ ); and,

Manuscript received September 29, 2019. This work was supported and sponsored by the North-West University in South Africa

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nitrate-based media ( $\text{Fe}(\text{NO}_3)_3$ ). Regardless of the choice of media,  $\text{CuFeS}_2$  is a highly refractory mineral for aqueous extraction processing and is regarded as one of the most difficult-to leach minerals in general. Its leaching is characterized by a slow and incomplete dissolution, which is mainly due to the formation of a diffusion barrier that builds up between the leaching solution and the  $\text{CuFeS}_2$  mineral. The nature and composition of this barrier, together with the dissolution mechanism, remain subject to controversy up-to-date [5-8]. However, there are currently three hypotheses explaining the structure of the formed diffusion barrier (impermeable passive layer) in this system. The first hypotheses is that the elemental sulphur, which is formed as the reaction product during leaching, prevents further diffusion of the reactant from reaching the un-leached chalcopyrite [9,10]. The second theory and the commonly cited hold responsible the formation of Cu-rich polysulphides, which form as a result of solid-state transformation through the preferential Fe dissolution. This theory is referred to as the metal-deficient sulphide theory. The third theory pinpoints to iron-precipitate compounds, which act as a barrier hindering dissolution. The candidates for this last theory include jarosite, jarosite-like compounds and goethite.

The acidic ferric sulfate ( $\text{H}_2\text{SO}_4\text{-Fe}_2(\text{SO}_4)_3\text{-FeSO}_4\text{-H}_2\text{O}$ ) media is the preferred dissolution media system for chalcopyrite whether chemical or bio-assisted leaching process. It presents several advantages, including its simple chemistry, low capital and operational costs, environment friendliness and it being convenient for the recovery of copper by solvent extraction and electrowining [11]. This system is usually prepared/ obtained by the mix of a known amount of iron sulfate ( $\text{Fe}_2(\text{SO}_4)_3$ ), sulfuric acid ( $\text{H}_2\text{SO}_4$ ) and water ( $\text{H}_2\text{O}$ ). However, in aqueous sulfuric solutions Fe could be found distributed as free dissolved Fe species ( $\text{Fe}^{3+}$  and  $\text{Fe}^{2+}$ ), complexed iron compounds ( $\text{FeHSO}_4^+$ ,  $\text{FeSO}_4^\circ$ ,  $\text{Fe}(\text{HSO}_4)^{2+}$ ,  $\text{Fe}(\text{SO}_4)_2^-$  and  $\text{FeSO}_4^+$ ). The content of these various iron species within the solution depend on the solution pH, composition and temperature [12]. This work the presents chemistry related to the Fe speciation and its evolution during the dissolution study of  $\text{CuFeS}_2$  in  $\text{H}_2\text{SO}_4\text{-Fe}_2(\text{SO}_4)_3\text{-FeSO}_4\text{-H}_2\text{O}$  system at a media pH of 1.0 and at room temperatures ( $25^\circ\text{C}$ ). To that, speciation allows to identify responsible Fe species parking in the Cu dissolution. The study further highlights the effect of pH and temperature upon the leaching process.

## II. MATERIAL AND METHOD

### A. Materials

Solutions of the desired pH values were prepared using analytical reagent-grade sulphuric acid ( $\text{H}_2\text{SO}_4$  98% ACE), ferric sulphate ( $\text{Fe}_3(\text{SO}_4) \cdot \text{H}_2\text{O}$  ACE), sodium hydroxide (NaOH pellets ACE) and distilled, deionized water ( $7.0\mu\text{s}/\text{cm}$ ). A pH meter and temperature probe (Hanna pH HI 8424) were used to measure pH, which was regularly calibrated with 4 and 7 standard buffer solutions and the redox. The measured redox potential (Ag/AgCl) measurements were referenced to the Standard Hydrogen Electrode (SHE).

### B. Chalcopyrite sample preparation and characterisation

The chalcopyrite used in this study were obtained as wet concentrates from a Phalaborwa Copper Mining Company (Limpopo Province, South Africa). Approximately 0.5 kg of concentrate were sub-sampled and further dried in an oven for two hours at  $105^\circ\text{C}$ , followed by homogenisation to a grain size of more than 200 microns ( $< 200\ \mu\text{m}$ ) before using the dissolution studies. The powdered chalcopyrite sample was characterized in an earlier study by Nyembwe et al [13] for its chemistry, mineral composition and morphology using the XRF, X-ray diffraction (XRD) and scanning electron microscopy–energy dispersive spectroscopy (SEM-EDX), respectively. The bulk chemistry revealed that the chalcopyrite sample composed mainly of Cu (36.4%), Fe (26.2%), Ca (15.2%) and S (10.6%). While the mineral content (summarised in Figure 1) revealed the presence of chalcopyrite (58.37%), bornite (4.51%), anilite (3.41%), digenite (2.51%), calcite magnesium carbonate (29.27%) and silica (2.20%).

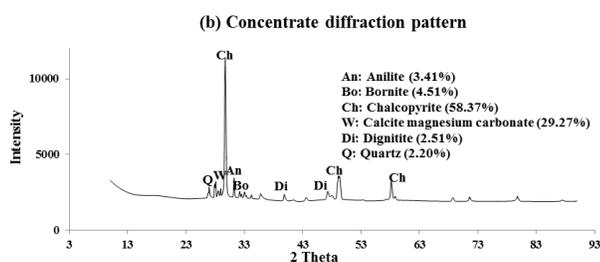


Fig. 1. Chalcopyrite sample mineral content

### C. Leaching media and tests

The dissolution media was acidified sulphate solution ( $\text{H}_2\text{SO}_4\text{-Fe}_2(\text{SO}_4)_3$ ), obtained by mixing analytical-grade ferric sulphate ( $\text{Fe}^{3+} = 0.05$  moles) with deionized water and sulfuric acid ( $\text{H}_2\text{SO}_4$ ). The ferric content was kept constant in all solutions, which were equilibrated in a reactor vessel for 12 hours prior to use.

The leaching tests were performed in batch set-up and atmospheric conditions at  $25^\circ\text{C}$  (the temperature was kept constant using a water bath), testing leaching capacity of media was assessed at a pH values of 1.0. The media pH was prepared at pH value of 1.0 and was maintained constant throughout the dissolution addition of concentrated sulfuric acid ( $\text{H}_2\text{SO}_4$

(98%)). A constant pulp density of 10% solids was used, with 40 g of the dried chalcopyrite sample, which were mixed with 400 ml of the leaching liquor in a 600 ml Erlenmeyer flask, shaken continuously for 12 hours on an orbital shaker operating at 200 rpm. To determine the dissolution kinetic information were obtained by periodic (every 20 min) withdrawal of a 10 ml aliquot from the mixture for chemical analysis, while the solution oxidation reduction potential (ORP) was recorded within the experimental set time interval and converted to the SHE, as prescribed by Stringgow [14]. Dissolved metals (Cu and Fe totals) were analysed using atomic absorption flame spectrometry (AAS [Thermo Scientific ICE 3000 series]). Prior to this, the extracted slurry was filtered, and the clean leachate solution was injected into the AAS for its metallic content (Cu and Fe totals).

### D. Leachate speciation

The chemical speciation of important ions in the leachate were calculated using geochemical modelling software, Phreeqc (version 3.3.12-12704, US Geological Survey) with inputs of the measured concentration of ions of interest in the leachate, the pH, Eh and temperature and other physical chemical properties recorded in the experiment [15-28]. The redox was defined as described in equation 1 (Eq. 1) with R is the gas constant ( $8.314\ \text{JK}^{-1}\text{mol}^{-1}$ ), T in the temperature (K) and F represents the Faraday constant ( $96485\ \text{C mol}^{-1}$ ).

$$Eh = pe \frac{2.303xRT}{F} \quad \text{Eq1}$$

Fig. 2. shows the initial Fe speciation at  $250^\circ\text{C}$ , media pH of 1.0 and recorded ORP 342 mV Ag/AgCl and pe value of 9.3.

### Fe speciation of intial solution

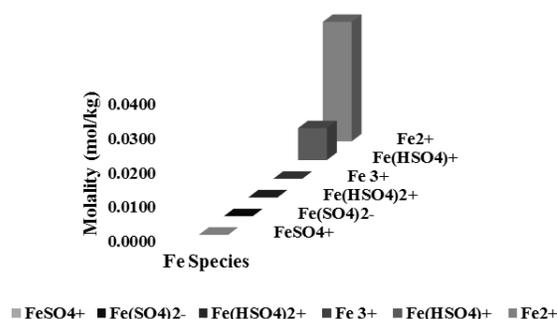


Fig. 2. Leaching media initial speciation

### E. Residue characterization

The solid residues were assessed for mineral composition using XRD (Rigaku Ultima IV, Rigaku Corporation, Tokyo, Japan), operating at 40 kV and 30 mA. Data were recorded over the range  $5^\circ \leq 2\theta \leq 95^\circ$ . The samples were milled further to 53 microns and were scanned at  $0.5^\circ/\text{min}$ , with a step width of  $0.01^\circ$ .

## III. RESULTS AND DISCUSSION

Fig. 3a summarizes the dissolution recovery for Cu and Fe at the investigated pH value (1.0) the 12 hours leaching test. Figure 3b shows the mineral phase content of the solid residue after the dissolution test. Cu dissolution curve (Fig 3a) obeyed the parabolic nature of the mineral and was characterized by rapid withdrawal of Cu at the early stage of the dissolution. The mineral's Fe dissolved was obtained after subtracting the initial Fe portion used as oxidant (leaching solution) from the total Fe reporting under the AAFS (i.e., Fe mineral = Fe Total (AAFS) - Fe used as oxidant (ferric sulfate)). In addition to that, it could be seen that the Cu dissolution took place in different stage. The first stage was the rapid Cu withdrawal at the earlier stage (20 min of dissolution), followed by a slow dissolution rate (from 20 to 600 min) and lastly the plateau stage (after 600 min of dissolution). This could suggest that during the dissolution chalcopyrite undergoes mineral mutation or conversion. This tends to agree with the XRD results (Fig 3b) were new phases, initially absent in the feed samples (Fig 1) were identified in the residual solid leached sample (Fig 3b). These phases were chalcocite (Cx) and covelite (Co).

The media Fe initial speciation is shown in Fig 2 and it revealed that the solution is composed of various Fe compounds which could be categorized as anions, cations and neutral complexes (free  $\text{Fe}^{2+}$  and  $\text{Fe}^{3+}$ ), complexed ions ( $\text{Fe}(\text{HSO}_4)^+$ ,  $\text{Fe}(\text{HSO}_4)^{2+}$ ,  $\text{Fe}(\text{SO}_4)^{2-}$ ,  $\text{FeSO}_4^+$ ,  $\text{FeSO}_4^0$ ) [29]. free  $\text{Fe}^{3+}$  only constitutes a small portion of the media which is mainly composed of  $\text{Fe}^{2+}$  and  $\text{Fe}(\text{HSO}_4)^+$ . It therefore appeared that accurate solution speciation could assist with explanation in regard to dissolution mechanism and pinpoint responsible Fe compound responsible for Cu dissolution from the  $\text{CuFeS}_2$ . The evolution of Fe speciation showed a decrease in all Fe species content (Fig 4 a, b, c and d) suggesting that all these species played a vital role during Cu withdrawal from the  $\text{CuFeS}_2$ . 90% decrease was observed for  $\text{Fe}^{3+}$ , 8% decrease for  $\text{FeSO}_4^+$ , 2% decrease for  $\text{Fe}(\text{SO}_4)_2^-$ , 17% decrease for  $\text{Fe}(\text{HSO}_4)^{2+}$  at pH 1.0 at  $25^\circ\text{C}$ . The relatively high decrease in content identified for  $\text{Fe}^{3+}$  and  $\text{Fe}(\text{HSO}_4)^{2+}$  agrees with the earlier study reporting that both these ions play a major role during Cu dissolution. The results confirm that  $\text{Fe}^{3+}$  is the principal specie followed by  $\text{Fe}(\text{HSO}_4)^{2+}$  [30]. However, the small decrease in regards to  $\text{FeSO}_4^+$  and  $\text{Fe}(\text{SO}_4)_2^-$  should not be ruled out for Cu withdrawal rather could suggest that these ions oxidizing strength could be much less than the two previous one at the investigated parameters (pH and T0).

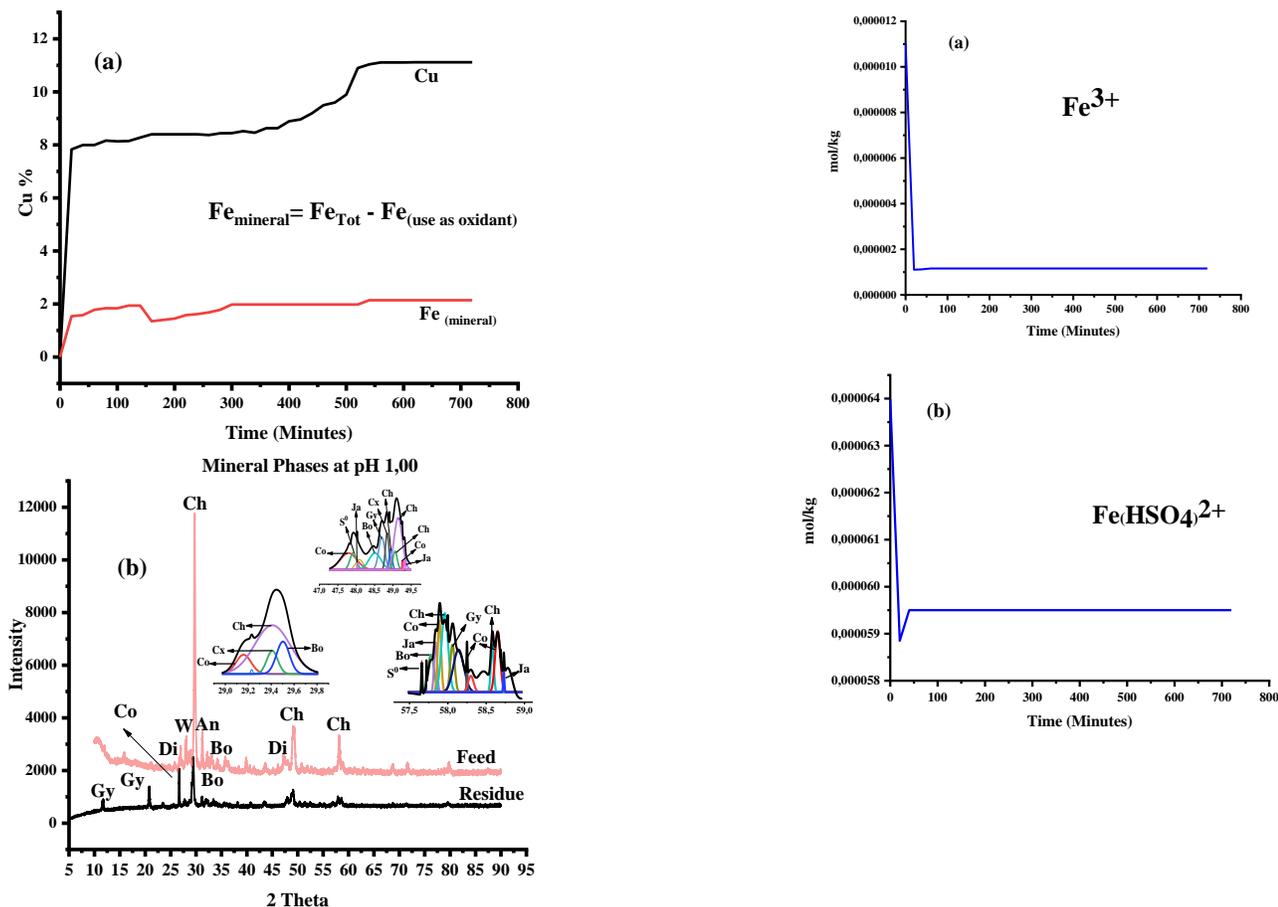


Fig. 3.  $\text{CuFeS}_2$  dissolution curve and solid residue mineral content characterisation

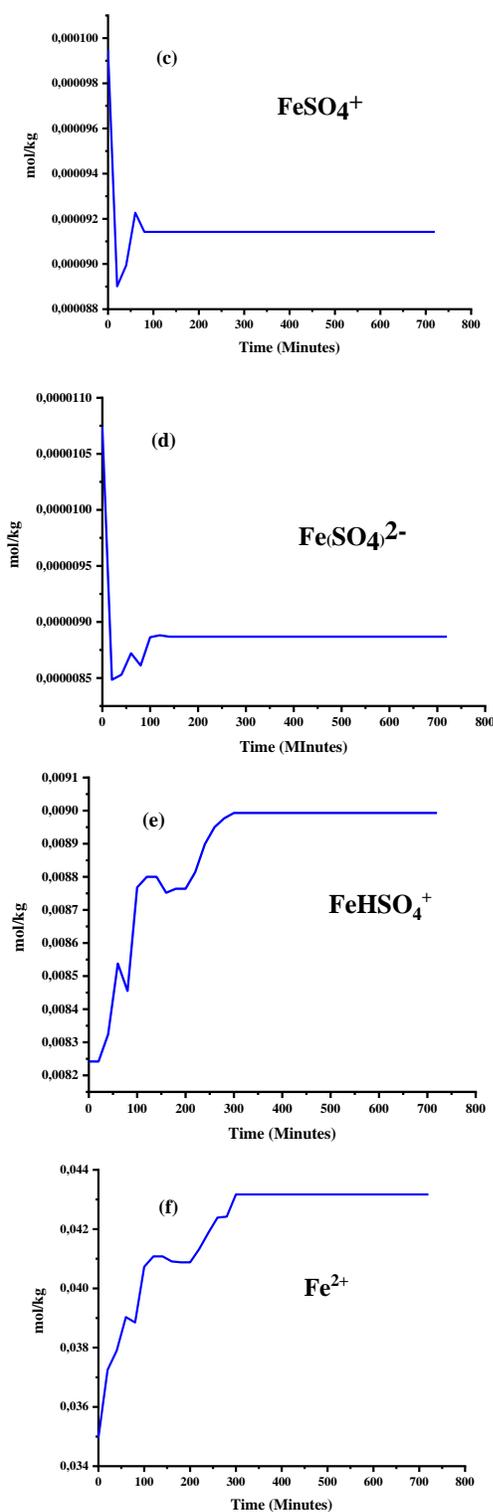


Fig. 4. Evolution of speciation of various Fe species during CuFeS<sub>2</sub> at pH of 1.0 at 25<sup>o</sup> C

The increase in the content of Fe<sup>2+</sup> and FeHSO<sub>4</sub><sup>+</sup> (Fig 4 e and f) support the mineral dissolution reaction (Eq1), producing and produces elemental sulphur (S<sup>0</sup>) and both copper and ferrous sulphate [31]. It could be said that during the dissolution of CuFeS<sub>2</sub>, the first step could be regarded as the ferric attack, taking place at the mineral surface and causing the

reduction of the oxidant (all ferric species including Fe<sup>3+</sup>, FeSO<sub>4</sub><sup>+</sup>, Fe(SO<sub>4</sub>)<sup>2-</sup> and Fe(HSO<sub>4</sub>)<sup>2+</sup>) thus promoting the withdrawal of Fe and Cu.



#### IV. CONCLUSION

In this study CuFeS<sub>2</sub> was dissolved in acidic ferric sulfate (Fe<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>+H<sub>2</sub>SO<sub>4</sub>+H<sub>2</sub>O) at a solution pH of 1.0 and 250C. only 12% of the Cu were recovered from the dissolution test. The results showed that both free Fe<sup>3+</sup> and Fe(HSO<sub>4</sub>)<sup>2+</sup> are the major species for Cu withdrawal. Fe<sup>3+</sup> was found as the main responsible ion and complete Cu dissolution was not attained due to the completion of this ion. Cu leaching curve displayed a stage wise dissolution process characterized by a rapid rate at the earlier stage, decreasing with time until the plateau stage is reached. The solid leached residue showed the presence of new copper sulphide phases including bornite, chalcocite and covelite mineral and suggested that CuFeS<sub>2</sub> undergoes mutation during Cu dissolution.

#### ACKNOWLEDGMENT

The authors are thankful to the local South African mining company who participated in this research by providing the samples, the extraction metallurgy laboratory at the University of Johannesburg for equipment utilization and the chemical engineering department at the North-West University for the support and promotion of this research are also acknowledged.

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