



# Potential for bioleaching copper sulphide rougher concentrates of Nchanga Mine, Chingola, Zambia

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## Synopsis

Laboratory investigations were conducted to establish the feasibility of bioleaching a mixed copper oxide/sulphide rougher concentrate from Nchanga Mine on the Zambian Copperbelt. The objective was to determine the kinetics and extent of copper extraction for this material. Batch experiments were conducted under different solution conditions in stirred tank bioreactors. The progress of (bio)leaching was monitored through measurements of soluble ferrous and ferric iron, copper, pH, and redox potentials, while bacterial activity was monitored online through O<sub>2</sub> and CO<sub>2</sub> gas utilization rates.

About 20 per cent copper was solubilized within 2 hours in all cases of non-oxidative (abiotic), oxidative (abiotic), and bioleaching experiments. This was attributed to the dissolution of mainly copper oxides. Subsequently, bioleaching experiments resulted in an overall copper extraction of 93 per cent, with up to 8 g.L<sup>-1</sup> copper after six leaching days, compared to 58 per cent copper extraction in the abiotic oxidative acid leaching experiments. However, there was little effect of time (i.e. poor dissolution kinetics) on copper recovery for abiotic non-oxidative acid leaching of the material.

Hence, the rate of sulphide leaching increased due to the activity of bacteria. Thus, the material is potentially bioleachable under mesophilic conditions. However, more exhaustive test work needs to be conducted to establish the effect of bioleaching variables and heat requirement.

## Keywords

bioleaching, bioreactor, copper sulphide ore, recovery, kinetics.

## Introduction

Bioleaching of low-grade, mixed copper oxide/sulphide ores in bioheaps is now an established technology<sup>1</sup>. However, bioleaching efficiency and kinetics vary significantly for ores from different localities due to different mineralogical compositions, bacterial species, solution conditions, and the leaching system employed.

Dreisinger<sup>2</sup> and Miller<sup>3</sup> reported that bioleaching may be a cheaper alternative to other traditional metal extraction techniques such as smelting and therefore more suited to treating marginal (or low-grade) ores in cases

where high-grade mineral reserves have been depleted. It was therefore necessary to investigate the amenability of Nchanga copper sulphide material to bioleaching. This preliminary leaching kinetics data may be useful for the design, and possible development and operation, of tank or heap bioleaching operations.

The purpose of this study was to determine the kinetics of copper extraction, and the extent of mineral dissolution and microbial activity, on copper dissolution using a mixed copper oxide/sulphide rougher concentrate from Nchanga Mine as being representative of the Zambian Copperbelt, using a mixed culture of iron- and sulphur-oxidizing mesophilic bacteria.

## Copper bioleaching

A literature survey on copper bioleaching has indicated that secondary sulphide minerals of copper (such as chalcocite, covellite, and bornite) have been demonstrated to show satisfactory bioleach kinetics with mesophilic bacteria<sup>4</sup>, with several bioheap operations worldwide extracting copper under these conditions<sup>1</sup>. However, chalcopyrite has been reported to be very difficult to leach under mesophilic conditions. Attempts to improve chalcopyrite bioleaching now involve the

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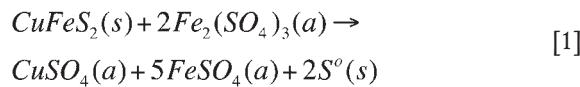
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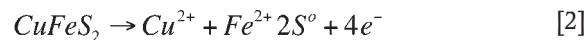
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application of controlled solution redox potentials, use of silver as a catalyst, and the application of thermophilic bioleaching to quickly and completely leach chalcopyrite<sup>5,6</sup>. A recent economic but non-bioleach hydrometallurgical system for the treatment of primary copper concentrates consisting predominantly of chalcopyrite is the Galvanox™ Process<sup>7</sup>. In this process, galvanic activity between pyrite and chalcopyrite promotes rapid and complete chalcopyrite oxidation to give over 98 per cent copper recovery within 4 hours at 80°C. A brief summary of the electrochemistry of the process is given in Equations [1–3].



Reaction [1] can be separated into anodic and cathodic half-cell reactions. The anodic half-cell reaction is:



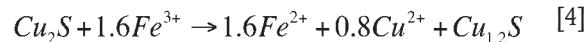
and the cathodic half-cell reaction is:



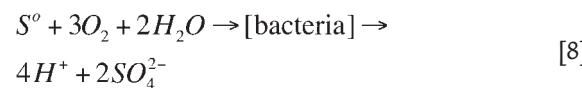
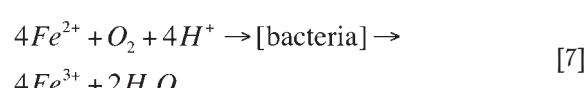
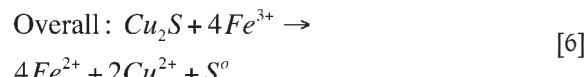
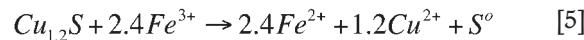
Chalcocite is an important secondary sulphide of copper that is reported to leach through a two-stage mechanism. It is rapidly attacked by ferric iron during the first stage to give a CuS intermediate product, while the second stage proceeds more slowly. About half (50 per cent) of the copper in Cu<sub>2</sub>S is leached quickly, though a complete dissolution requires much longer times<sup>8–10</sup>. Under mesophilic conditions, high rates of copper extraction from chalcocite have been reported for low pulp densities of typically less than 5% w/v solids, pH 1.3–2.3, 25–40°C, and quite low ferric iron concentration<sup>4,11,12</sup>.

It is well established that the first-stage leaching of chalcocite occurs faster than the microbial ferrous iron oxidation rate and produces non-stoichiometric pseudo covellite, which reacts further rather slowly in the second stage to produce S<sup>o</sup>, Cu<sup>2+</sup>, and Fe<sup>2+</sup> ions as detailed below<sup>8,10,13</sup>. At the same time, Fe<sup>2+</sup> and S<sup>o</sup> are microbially oxidized to regenerate Fe<sup>3+</sup> and produce more acid respectively<sup>14–17</sup>.

First-stage leaching of chalcocite:

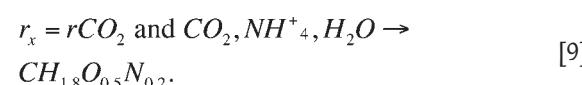


Second stage leaching of chalcocite or pseudo covellite by ferric iron is given by



### Microbial growth kinetics and O<sub>2</sub> and CO<sub>2</sub> gas uptake

According to Roels<sup>18</sup> the biomass (CH<sub>1.8</sub>O<sub>0.5</sub>N<sub>0.2</sub>) uses CO<sub>2</sub> and NH<sub>4</sub><sup>+</sup> as sources of carbon and nitrogen respectively for cell growth and maintenance. So the rate of bacterial growth,  $r_x$  (C-mol.L<sup>-1</sup>) is equal to the rate of carbon dioxide utilization,  $rCO_2$ .



The biomass concentration in a batch at time  $t$  is calculated from integration of the measured carbon dioxide rate<sup>14</sup> through a computer program called 'Off-gas analyser' that continuously monitors and logs CO<sub>2</sub> utilization rates by microbes.

$$C_{x,CO_2}(t) = C_x(0) + \int_0^t -r_{CO_2} dt \quad [10]$$

## Materials and methods

### Mineral characterization

The composite mineral samples were copper sulphide rougher concentrates provided by Nchanga Mine, Zambia. These samples were characterized in terms of particle size distribution (PSD) using a Malvern Mastersizer, chemical analysis was by atomic absorption spectrophotometer (AAS), while the mineralogical composition, distribution, and liberation characteristics were investigated using reflected light microscopy.

Chalcocite, at 15.27 weight per cent (wt%), was the major sulphide, followed by oxides of malachite (1.24 wt%), cuprite (1.67 wt%), and cupriferous micas (13.0 wt%). Chalcopyrite (1.53 wt%) also occurred in significant amounts. From elemental analysis the head grades were 16 per cent total copper, 7.01 per cent iron, and 9.51 per cent sulphur. Over 70 per cent of the particles were less than 38 µm, with  $d_{50}$  being 16 µm. The liberation characteristics were fairly good except for chalcopyrite, where 17 per cent remained locked in the gangue minerals.

### Experimental procedure

Several studies involving copper sulphides have been conducted in shake flask experiments where cell/microbial growth rates are often measured manually using such techniques as cell counts, protein analysis etc. Due to the relatively slow kinetics associated with shake flask experiments, low accuracy, and longer time requirements in the more common cell count technique, this study employed a stirred tank leaching method using off-gas analysis to enumerate microbial cells following the methods of Boon<sup>14</sup> and Breed<sup>19</sup>.

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In fact, reported bioleaching studies involving copper sulphide ores and/or minerals coupled to the off-gas for analysis of microbial growth are rather few, therefore it was necessary to conduct this study using the online off-gas technique as a faster alternative tool to enumerate microbial cells. Similar solution conditions to those of published studies involving shake flask experiments were therefore applied in this study to investigate the extent of copper extraction in stirred tank bioreactors.

### Determination of acid consumption

Since the concentrate contained mixed copper oxide/sulphide mineralization, it was necessary to determine acid requirement and acid soluble copper as a test prior to bioleaching. A batch stirred tank reactor was used with solids concentration of 5% w/v at pH 1.5, for which pH adjustments were done by the addition of sulphuric acid through a burette.

### Batch culture, nutrients, and shake flask experiments

Erlenmeyer flasks were used for growth and adaptation of bacteria in rotary shakers using a modified 9k (iron-free) basal salts growth medium with the ore as substrate at 5% w/v. Both the ore and glassware were sterilized separately by autoclaving at 121°C for 20 minutes. A mixed culture of iron- and sulphur-oxidizing, chemolithoautotrophic mesophilic bacteria, obtained from a University of Cape Town (UCT) laboratory stock culture, was used in the study.

The bacterial culture was grown in a modified 9k medium (i.e., mineral salt solution) with:  $(NH_4)_2SO_4$  (3.0 g.L<sup>-1</sup>), KCl (0.1 g.L<sup>-1</sup>),  $K_2HSO_4$  (0.5 g.L<sup>-1</sup>),  $MgSO_4 \cdot 7H_2O$  (0.5 g.L<sup>-1</sup>), and  $Ca(NO_3)_2 \cdot 4H_2O$  (0.013 g.L<sup>-1</sup>). The pH was adjusted to 2.0. The flasks were then inoculated with the bacterial culture at 10% v/v using aseptic techniques, and incubated at 35°C with a shaker speed of 170 r.min<sup>-1</sup>. The microbial growth rates were monitored frequently by cell counts and subculturing until growth of about 10<sup>8</sup>–10<sup>9</sup> cells per millilitre was observed.

### Batch chemical and microbial leaching experiments

These experiments were performed in a baffled, stirred, and aerated bioreactor (Figure 1) that was thermostated by an external water bath.  $O_2$  and  $CO_2$  contents in the dry off-gas and reference air were measured and monitored online using a data acquisition program.

The difference between reference and off-gas concentration was the microbial gas utilization rates, from which biomass concentration (C mole/litre) was calculated. Both microbial and chemical leaching experiments were conducted in the same reactor type and solution conditions, except that for the chemical leaching experiments the bacterial inoculum was replaced with an equivalent volume of sterile growth medium to make up 1 litre working volume.

Fifty (50) grams of mineral sample was added to the nutrient medium and the pH adjusted to the required value using sulphuric acid. The reactor was then inoculated with

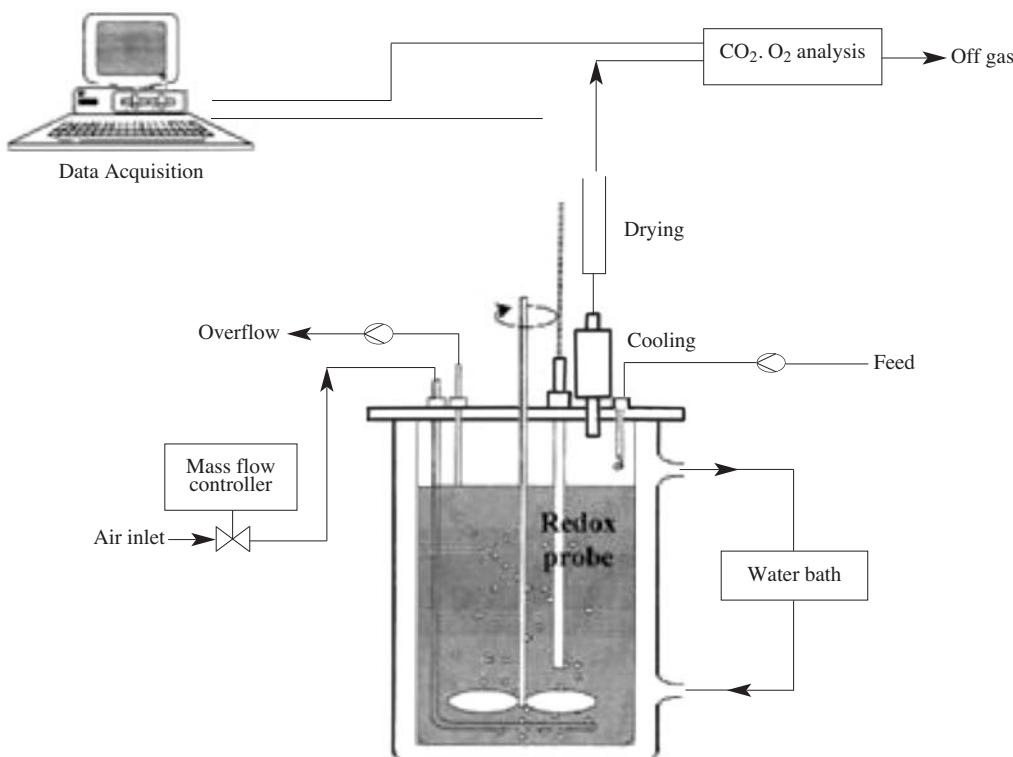


Figure 1—Stirred tank reactor (STR) with off-gas analyser

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100 ml of bacterial culture per 900 ml solution. When the pH was higher than the set value it was adjusted to this value by acid addition. If the pH was lower than required, a freely changing value was allowed to determine the acid-producing capacity of the bioleach system. If a constant pH was desired, it was manually controlled by the addition of either acid or base.

Daily 10 ml slurry samples were taken, filtered, and analysed for soluble species. The residues were digested to determine unleached copper and iron. Progress of bioleaching was monitored through measurements of  $[Fe^{2+}]$ ,  $[Fe^{3+}]$ ,  $[Cu^{2+}]$ , pH, and redox potential, while bacterial activity was monitored by  $CO_2$  and  $O_2$  utilization rates.

The redox potential was measured with a gel-filled Pt-Ag/AgCl combination redox probe. The pH values were measured by a combination probe using a Model 744 Metrohm pH meter filled with 3.0 M KCl. Copper and total iron concentrations were measured by AAS, while ferrous iron ( $Fe^{2+}$ ) was determined by spectrophotometry using 1–10 phenanthroline ( $C_{12}H_8N_2H_2O$ ) as an indicator and ammonium acetate ( $NH_4C_2H_3O_2$ ) as a buffer solution. Ferrous ( $Fe^{2+}$ ) iron was also determined by actual calculations from values of measured redox potentials, and total iron concentration using the Nernst equation<sup>20</sup> as shown in Equation [11]. The ferric ( $Fe^{3+}$ ) iron concentration was taken as the difference between total and ferrous iron contents ( $Fe^{total} = Fe^{2+} + Fe^{3+}$ ).

$$E = E_o + \frac{RT}{nF} \ln \frac{[Fe^{3+}]}{[Fe^{2+}]}; \text{ Hence, } \frac{[Fe^{3+}]}{[Fe^{2+}]} = \exp \left( \frac{e - E_o}{RT/nF} \right) \quad [11]$$

where  $E$  = potential of the solution (V),  $E_o$  = standard potential of  $Fe^{3+}/Fe^{2+}$  couple (V),  $R$  = universal gas constant ( $J.K^{-1}mol^{-1}$ ),  $T$  = absolute temperature (K),  $n$  = number of electrons transferred,  $F$  = Faraday's constant ( $kJ.v^{-1}$  per equivalent),  $[Fe^{3+}]$ , and  $[Fe^{2+}]$  = molar concentration of ferric and ferrous ions respectively ( $mol.L^{-1}$ ).

Thus, the Nernst equation relates the solution potential to the free ferric and ferrous iron activities with ideal values of  $E_o$  and  $RT/nF$ . Hence, the redox probe was calibrated<sup>20</sup> at the applied experimental conditions in order to obtain these coefficients,  $E_o$  and  $RT/nF$ . Calibration Equation [12] at 35°C and pH 2.0 was:

$$E = 26.405 \ln \frac{[Fe^{3+}]}{[Fe^{2+}]} + 473.33 \quad [12]$$

Hence, at each redox potential value and total iron concentration, values of ferrous and ferric iron concentrations were calculated by the simplified set of Equations [13]:

$$[Fe^{2+}] = \frac{[Fe]^{total}}{1 + \frac{[Fe^{3+}]}{[Fe^{2+}]}} \text{ and } [Fe^{3+}] = \frac{[Fe]^{total} * \frac{[Fe^{3+}]}{[Fe^{2+}]}}{1 + \frac{[Fe^{3+}]}{[Fe^{2+}]}} \quad [13]$$

The calculations of percentage copper extraction were based on both soluble copper and leach residue assays. The use of leach residue assays was found to be more consistent and reliable. This procedure was adopted and used in the calculations (Equation [14]). Total iron dissolution was calculated similarly.

$$\%Cu = \left( \frac{\%Cu_f^{t=0} - \%Cu_{res}^{t=t}}{\%Cu_f^{t=0}} \right) * 100\% \quad [14]$$

where  $\%Cu_{res}^{t=t}$  = per cent copper in leached residue at any time  $t$  and  $\%Cu_f^{t=0}$  = per cent copper in starting (feed) sample at time  $t = 0$ .

### Abiotic oxidative and non-oxidative acid leaching experiments

In line with the previous section, acid leach tests were conducted to determine acid consumption by oxide copper and gangue minerals. The extent of dissolution of acid-soluble copper was assessed through oxidative (with air) and non-oxidative (without air) leaching. The objective was to determine the extent of copper dissolution in the absence of bacteria.

### Microbial leaching

The bioleaching experiments were conducted on both fresh rougher concentrate feeds and leach residues from non-oxidative acid leach experiments. Details of the procedure are given in the previous sections.

## Results and discussion

### Acid consumption

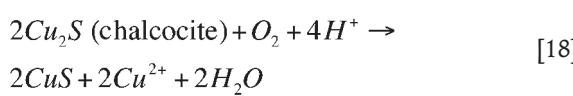
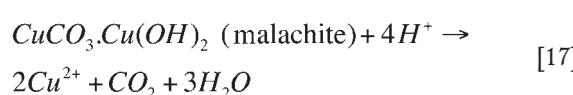
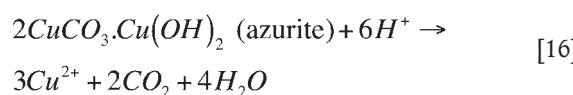
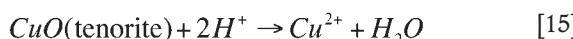
It is expected that copper oxides, carbonates, silicates, and other alkaline gangue minerals rapidly consume acid to break down to different solution products<sup>21</sup>. Therefore, preliminary acid consumption tests were designed to approximate the quantitative acid requirement of rougher concentrates at the desired pH, and also to infer the extent of dissolution of acid-soluble copper and slow-leaching oxides and sulphides.

In the initially observed pH profiles, the acidity of the leaching system decreased, and this was attributed to chemical breakdown of both acid-soluble copper mineral phases and gangue (waste), as noted by Jansen and Taylor<sup>21</sup>. The bioleaching tests exhibited higher acid consumption rates, resulting in higher copper extractions than the abiotic oxidative acid leaching experiments. This may be due to additional acid-consuming reactions in bioleaching, such as microbial ferrous iron oxidation (Equation [7]). Acid demand was 150–300 kg and 150–240 kg per ton of ore for bioleaching and abiotic oxidative acid leaching tests respectively. Similar rates of acid consumption were reported for copper oxide leaching<sup>22</sup>.

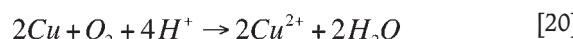
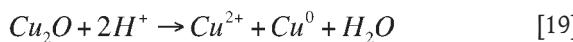
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### Oxidative, non-oxidative, and microbial leaching

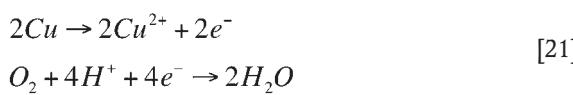
In Figure 2, the abiotic non-oxidative acid leaching tests solubilized about 20 per cent copper after 50 hours, compared to 45 per cent copper dissolution in the oxidative leaching tests using air. The dissolution profile starts with complete acid dissolution of  $CuO$  and copper carbonates and dissolution of about half of the  $Cu_2O$ <sup>22,23</sup>. Introduction of air ( $O_2$ ) probably leads to the complete dissolution of cuprite (which requires oxygen) and partial oxidation of chalcocite<sup>24</sup> to an overall 45 per cent copper extraction, since most other copper sulphides are relatively unaffected under these conditions<sup>25</sup>. The chemical leaching reaction mechanisms are given in Equations [15–21].



Cuprite ( $Cu_2O$ ) theoretically solubilizes only half of the copper (Equation [19]) while the other half is converted into metallic copper which subsequently undergoes dissolution in the presence of oxygen (Equation [20]).



Reaction [20] is a summation of two half-cell reactions:

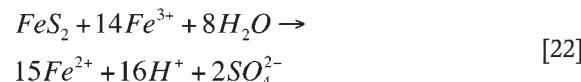


In the bioleach system over 93 per cent of the copper was solubilized in 6 days relative to 58 per cent copper extraction in the abiotic oxidative acid leach (Figure 2). The low leach efficiency in acid leach tests was attributed to limited acid dissolution of copper sulphides. Thus, in the presence of bacteria the rate of sulphide oxidation is enhanced. The production of ferric iron from microbial ferrous iron oxidation (Equation [7]) leads to the ferric leaching of copper, pyrite and other sulphides to varying degrees, as given for  $Cu_2S$  (Equations [4–8]), pyrite, and other sulphides (Equations [22–27])<sup>17,26</sup>.

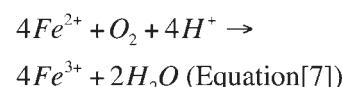
Secondary copper sulphides are nearly completely bioleached with mesophilic bacteria<sup>1,4,8,27</sup>, resulting in the observed high copper extraction rates. The residual copper

might be attributed to chalcopyrite because this mineral has been reported to be very difficult (refractory) to leach with mesophiles<sup>4,26,28,29</sup>.

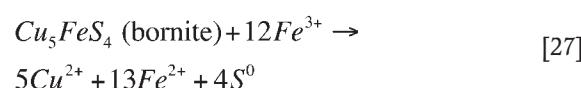
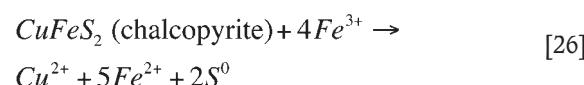
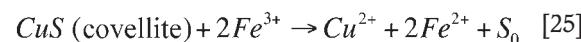
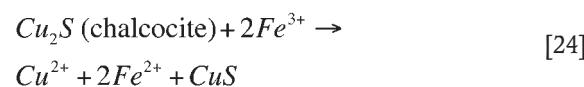
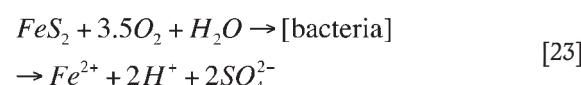
Chemical ferric oxidation of pyrite:



Bacterial ferrous iron oxidation:



Overall:



Dew *et al.*<sup>4</sup> reported the preferential order of mineral bioleaching with mesophile bacterial culture as  $Cu_2S > Cu_5FeS_4 > CuS > FeS > \dots > CuFeS_2$ .

The bioleaching of a pre-treated rougher concentrate (residue) from the non-oxidative acid leach experiment was observed to initially proceed slowly relative to the untreated ore, though eventually giving over 93 per cent copper

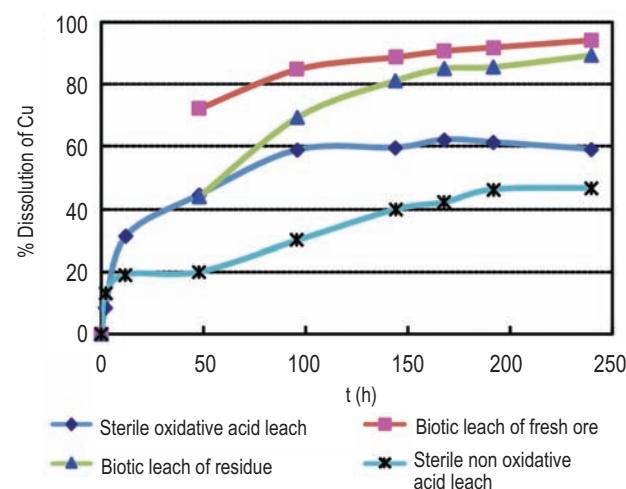


Figure 2—Copper dissolution (%) plotted against time at pH 1.50, 35°C, and 50 g.L<sup>-1</sup> solids loading in a stirred tank reactor (STR)

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extraction in both cases. This is because the leach residue probably did not contain acid-soluble copper oxides. Furthermore, the experimental copper dissolution data compared well with published laboratory bioleaching studies<sup>4,8,10,11</sup>. The consequence of these trends is that bioleaching of untreated ore may be preferable because of the faster oxidation kinetics.

### Variation of redox potential and ferrous/ferric iron

The initially low redox potential values noted during the bacterial lag phase were probably due to the relatively fast reduction of ferric iron by chalcocite during the first stage of leaching. This was followed by onset of bacterial oxidation of ferrous iron, as reflected in the rapid increase of redox potential (Figures 3 and 4). However, the rate of mineral ferric consumption at the second stage is now much lower and the solution redox potential attains high values. Redox potentials are reflected by changes in the  $\text{Fe}^{3+}/\text{Fe}^{2+}$  ratios of dissolved iron in the leach slurry reaching 690 mV (vs.  $\text{Ag}/\text{AgCl}$ ) (Figure 3). The bacterial ferrous oxidation reaction is probably limited by a low ferrous iron concentration (Figures 3 and 4).

Also, the dissolution of pyrite (Equations [7], [22], and [23]) was significant only at high redox potentials from observed measurements of total soluble iron (Figure 5). Thus, it is likely that pyrite dissolution started after about 75 hours when the solution redox potential was above 550 mV (vs.  $\text{Ag}/\text{AgCl}$ ) (Figure 5). It has been reported that pyrite dissolution occurs at high redox potential after considerable dissolution of secondary copper sulphides and to some extent chalcopyrite<sup>30</sup>.

Sulphur-oxidizing bacteria probably oxidized sulphur and/or reduced sulphur compounds to sulphuric acid (Equation [8]) since the pH was observed to decrease,

indicating a bacterial activity after 3 days (Figure 6). It appears therefore that ferrous iron and sulphur compounds (and not chalcocite) are the substrates for bacteria. They regenerate ferric iron that subsequently oxidizes chalcocite and other sulphides. These results are consistent with literature data, confirming and recognizing the fact that the presence of bacteria increases the rate of leaching. The role of bacteria therefore is to provide acid and the ferric iron oxidant (through sulphur and ferrous iron oxidation) required for dissolution of copper oxides/carbonates and sulphides.

In Figures 7 and 8, the growth and activity of bacteria were dependent on ferrous iron concentration. Bacterial  $\text{O}_2$  and  $\text{CO}_2$  consumption rates were rapid and reached 2.0  $\text{mmol} \cdot \text{L}^{-1} \cdot \text{h}^{-1}$  and 0.131  $\text{mmol} \cdot \text{L}^{-1} \cdot \text{h}^{-1}$  respectively after 24 hours. The concentration of bacteria was a maximum in

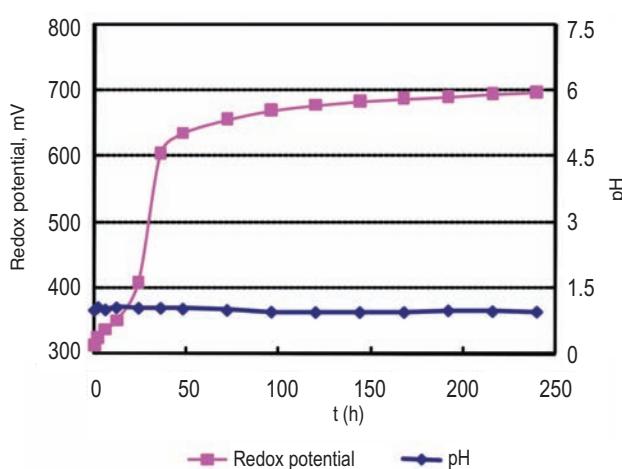


Figure 3—Variation of redox potential and pH with time in bioleach tests at 35°C, pH 1.50, and 50 g.L<sup>-1</sup> solids loading

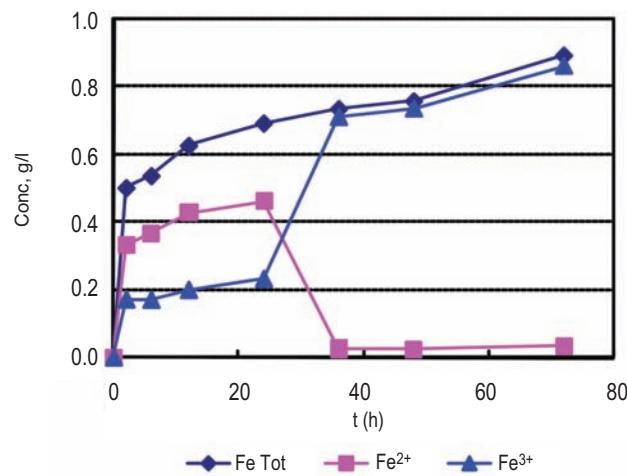


Figure 4—Variation of soluble iron and speciation in bioleach tests at 35°C, pH 1.50, and 50 g.L<sup>-1</sup> solids loading

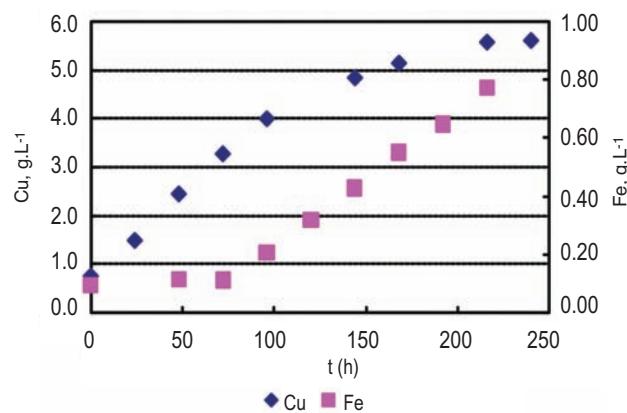


Figure 5—Variation of copper and iron concentrations for bioleaching of residue from non-oxidative acid leach tests at 35°C, pH 1.50, and 50 g.L<sup>-1</sup> solids loading

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the last phase at about 10.0 mmole carbon per litre, after which there was negligible rate of growth other than probably in maintenance terms.

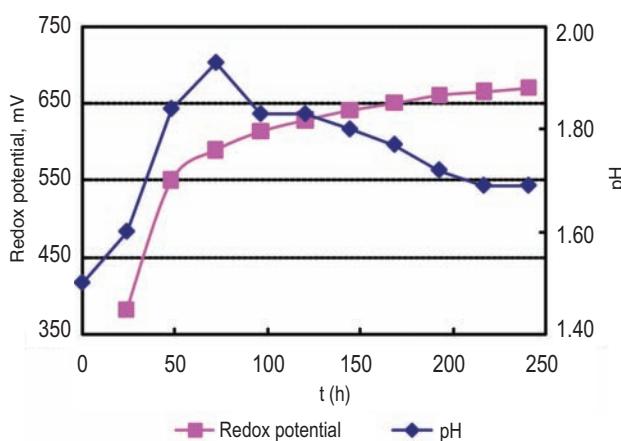


Figure 6—Variation of pH and redox potential for bioleaching residue from non-oxidative acid leach tests at 35°C, pH 1.50, and 50 g.L<sup>-1</sup> solids loading

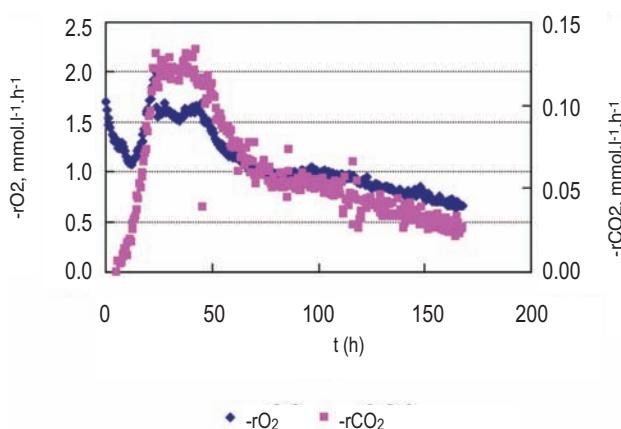


Figure 7—Variation of CO<sub>2</sub> and O<sub>2</sub> utilization rates plotted against time at 35°C, pH 1.50, and 50 g.L<sup>-1</sup> solids concentration

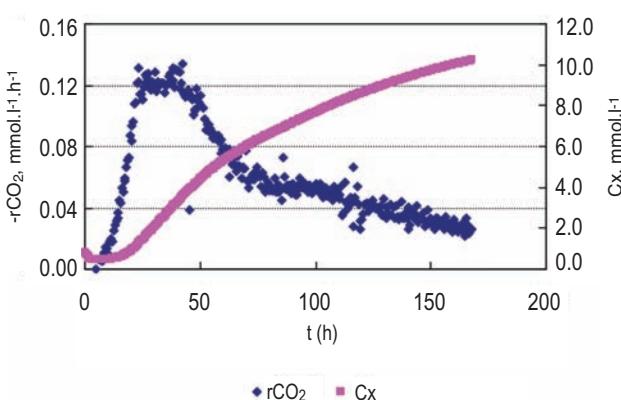


Figure 8—Variation of biomass concentration and CO<sub>2</sub> consumption rate plotted against time at 35°C, pH 1.50, and 50 g.L<sup>-1</sup> solids concentration

### Conclusions

The leaching profile of this material progressed in a three phase sequence. In phase one, 20 per cent copper was abiotically leached in dilute acid without air (oxygen), corresponding to the chemical (acid) dissolution of copper oxides/carbonates. The second phase resulted in 40-60 per cent copper being abiotically and chemically extracted in the presence of air. The last phase involving bioleaching of largely copper sulphides resulted in over 93 per cent copper recovery, with up to 8.0 g.L<sup>-1</sup> Cu after six leaching days. Hence, the material responded positively to bioleaching under mesophilic conditions as evidenced by high percentage copper dissolution and satisfactory microbial activity of up to 10.0 mmol carbon per litre bacterial population at the stationary phase. Thus, off-gas analysis is useful for calculating microbial growth rates and other kinetic parameters even in a multi-sulphide leaching system, as it has been done for single mineral substrates like pyrite and chalcopyrite<sup>14</sup>.

However, more exhaustive test work needs to be conducted to establish the effect of bioleaching variables and heat requirement. Further investigations in columns are needed to ascertain the leachability and scale-up of the present experimental data to a possible heap bioleaching operation. It is also proposed that a rigorous mathematical treatment of degree of reduction balances must be applied to a multi-substrate system such as this one in order to relate the rate of copper dissolution to bacterial O<sub>2</sub> and CO<sub>2</sub> consumption rates, as has been done for single (ferrous iron, sulphur, etc.) substrates by Boon<sup>14</sup>. One limitation in this study was the inability to determine the qualitative and quantitative mineralogical composition of the bioleach residues to fully interpret the bioleaching data.

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