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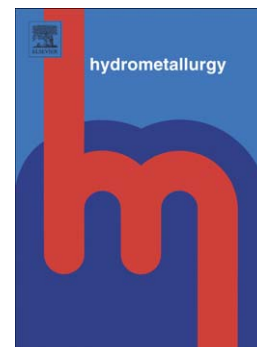
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The dissolution of chalcopyrite in chloride solutions.

Part 1. The effect of solution potential

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Abstract

A study of the dissolution of several copper concentrates under controlled potential conditions in chloride solutions has demonstrated that the rate of dissolution of chalcopyrite is strongly dependent on the potential of the solution. Linear rates were obtained for the dissolution of chalcopyrite in solutions containing 0.2 M HCl and 0.5 g L⁻¹ Cu(II) at 35 °C. The rate is enhanced within a range of potentials of 550 to 620 mV (versus SHE) and the presence of dissolved oxygen is essential for enhanced rates within this potential window. Even though dissolved oxygen is important in order to achieve acceptable rates of dissolution, excessive oxidation can increase the potential into a region in which passivation is possible.

Reduction of the potential into the optimum region results in restoration of enhanced rates of dissolution. On the other hand, dissolution at low potentials (< 540 mV) results in reduced rates of copper dissolution which increase significantly when the potential is subsequently increased to above 580 mV. Mineralogical studies have shown that chalcopyrite remains un-leached and small amounts of covellite appear to be formed on the surface of the chalcopyrite at low potentials. All concentrates appear to dissolve at roughly the same rate under the same conditions, despite differences in the composition and mineralogy of the samples.

Keywords: Chalcopyrite; Chloride leaching rates; Controlled potential, Mineralogy; Passivation

1. Introduction

The dissolution of sulfide minerals and, in particular, that of chalcopyrite are potential-dependent reactions and many studies have been carried out in order to establish the relationship between the solution potential and the leaching rate. In the context of leaching under ambient conditions that are appropriate to heap or dump leaching, it is now well known that the leaching rate of chalcopyrite in sulfate solutions is dependent on the potential and that the rate is enhanced in a certain potential range (Nicol and Lazaro, 2002). Conventional mixed-potential theory for dissolution does not appear to apply to the leaching of chalcopyrite under these conditions.

Several studies have been devoted to the elucidation of the potentials at which dissolution is possible and that at which apparent passivation occurs. Thus, Nicol and Lázaro (2002) used electrochemical and chemical experiments to establish the relevant leaching potential region as being 0.45 to 0.75 V under appropriate experimental conditions. However, few actual dissolution studies have been conducted under controlled potential conditions, particularly in chloride media (Hiroyoshi et al., 2000; Rivera-Velasquez et al., 2006; Third et al., 2002). In addition, several studies have focused on enhanced dissolution at low redox potentials (Hackl et al., 1995; Hiroyoshi et al., 2000) and a reaction model to explain the phenomenon has been proposed (Hiroyoshi et al., 1997; 2001; 2000).

Preliminary experiments showed that it is not possible to maintain constant potential conditions throughout the duration of a leach test due to continuous changes in variables such as pH, chloride and iron and copper concentrations. In order to develop an ambient temperature process for successful heap leaching of chalcopyrite

in a chloride or chloride–sulfate system it is not only appropriate to establish the boundaries of the potential range required for adequate leaching but it is also necessary to develop and implement strategies for the control of the potential if required.

This paper, which is the first of three, starts by defining the conditions and the potential range for optimum chalcopyrite leaching using three different potential control systems i.e. electrochemical, chemical and gaseous. The results of experiments to establish whether chalcopyrite concentrates from different localities leach at different rates under the same conditions will be presented without any detailed interpretation or discussion. The second paper will deal with the effects of various parameters on the rate of dissolution of chalcopyrite under controlled potential conditions. The final paper will discuss the experimental results obtained in the first two parts and will present additional information in support of a novel interpretation of all the data and will suggest alternative mechanisms for the enhanced leaching process which are consistent with the experimental observations.

2. Experimental

2.1. Materials and procedures

Chalcopyrite concentrates were obtained by flotation from several operations in Chile and the USA and a relatively pure massive sample of chalcopyrite was obtained from the OK Tedi mine. Because of the fact that dry screening of the concentrates produced significant amounts of agglomerated fine material that made comparisons of the relative leach rates difficult, the procedure included wet screening of each concentrate to obtain a well-defined chalcopyrite size fraction. Most experiments

were carried out with -38+25 μm or -38 μm size fractions. The chemical analyses of the screened samples are shown in Table 1 and mineralogical data obtained from mineral liberation analyses (MLA) for three of the four concentrates are presented in Table 2.

2.2. *Leaching experiments*

Agitated leaching experiments were carried out in instrumented reactors constructed at Murdoch University. The baffled cylindrical glass reactors have a volume of 1000 cm^3 and are sealed with a multi-port PVC lid on which is mounted a stirrer motor with a titanium impeller. The lid has ports for ORP and chalcopyrite electrodes and nitrogen or oxygen gas inlets. The reactor is enclosed in a temperature-controlled PVC surround. Unless stated otherwise, the leach experiments were conducted with several concentrates and the mass of material used was adjusted to provide approximately 3 g of copper in 900 cm^3 of a solution containing 0.2 M HCl and 0.5 g L^{-1} of Cu (II) at 35 °C at a stirring speed of 800 rpm. As will be shown in the second paper of this series, these conditions were selected as being an appropriate compromise between optimum conditions and those most likely to be economically possible in a heap leach process.

During each test, liquid samples were withdrawn periodically after compensating for evaporation by the addition of distilled water and settling of suspended solids for approximately 30 min. An equal volume of the lixiviant was added to replace that removed in the sample. The samples were filtered (0.2 μm) prior to analysis for copper, total iron and in some cases silver using atomic absorption spectrometry (AAS). When solid samples were required, a slurry sample was collected from the

reactor and vacuum filtered. The solid was washed and dried in air. The dried solid samples were sent to the Newcastle Technology Centre of BHP Billiton for MLA analysis and microscopic examination.

The solution potentials were monitored using a platinum ring electrode with a combined Ag/AgCl reference electrode (3 M KCl). In some cases the mixed potential was also measured using a chalcopyrite electrode/reference electrode combination. The electrodes were connected to high impedance analogue input channels on a National Instruments data acquisition board controlled by LabviewTM software. A schematic of the potential control system used with injection of nitrogen or oxygen gas is shown in Figure 1. A similar system was used for electrochemical control which consisted of two platinum electrodes in the slurry. The anode is used to oxidise copper(I) and/or iron(II) and the cathode is separated from the leaching solution by a fritted glass disk sealed into the bottom of the cathode compartment which initially only contained acid. Passage of an appropriate current between these electrodes enabled the potential of the ORP electrode to be controlled to a set value. An alternative potential control system involving the addition of an oxidant, in this case, 0.2 M permanganate to the reactor using a Metrohm titration unit linked to the measured solution potential was also used.

Daily measurements of pH and the dissolved oxygen concentration in the reactor were made manually. In most experiments with those concentrates that contained predominantly chalcopyrite, the fraction of copper dissolved at any time was very similar to that of iron except at pH values above about 1.5 in which case precipitation of some iron was observed.

3. Results and discussion

Preliminary experiments were based on a published (Hiroyoshi et al, 2001) simple experimental technique in which small amounts of the mineral sample were leached in aerated shake flasks without any attempt to control the potential, other than the initial value which was varied by changing the iron(II)/iron(III) ratio. The results shown in Figure 2 confirmed the potential dependence of the rate of dissolution of chalcopyrite in that greater rates are observed at potentials between about 530 and 580 mV. These experiments were conducted over a short term, during which only a small fraction of the mineral was dissolved, and the results do not reflect the relative rates under more appropriate conditions.

Several additional experiments were therefore conducted with Concentrate 1 in aerated agitated reactors without control of the potential. In particular, the effect of the addition of ferric ions on the rate of dissolution was investigated. The results of two such experiments are shown in Figure 3. It is apparent that the rate of dissolution in a dilute chloride solution at low pH is significantly greater than that in the presence of ferric ions. The measured potentials of the solution during these experiments are also shown and confirm the detrimental effect of high (greater than about 650mV) potentials on the rate. Thus, conventional wisdom that ferric ions are suitable as an oxidant in this system and that increased potentials are desirable under these conditions is not correct.

3.1. *Electrochemical control of the potential*

The electrochemical control system was used to investigate the effect of potential on the rate of dissolution of Concentrate 1. Four experiments were carried out in the instrumented reactors using de-aerated solutions at various potential set-points under otherwise standard conditions. The results are shown in Figure 4. It is apparent that the rate of dissolution of chalcopyrite is low by comparison with the results of Figure 3 with a maximum of about 10% dissolved after 600 h.

Three further experiments were conducted in which the potential was electrochemically controlled at 580 mV while the slurry was sparged with nitrogen, air or oxygen. The results are shown in Figure 5. The dissolved oxygen concentrations were maintained approximately constant at about 12 mg L⁻¹ in the oxygen case and 5 mg L⁻¹ in the case of the air-sparged reactor. The rate of dissolution is obviously strongly dependent on the presence of dissolved oxygen but is relatively insensitive to the concentration in the range 5 to 12 mg L⁻¹. As a result of this observation, the use of electrochemical control was discontinued and control of the potential by injection of the appropriate gas was subsequently employed.

3.2. *Control of the potential by addition of oxygen*

As a result of the need to have some dissolved oxygen present in the leach solution, it was decided to modify the control system to one which varies the amount of air or oxygen injected into the leach pulp. Experiments were carried out at various potentials on Concentrate 1 under standard conditions. The results presented in Figure 6 confirm the importance of dissolved oxygen in that, at the appropriate potentials, over 90% dissolution was achieved after 1200 h. The results also demonstrate that

enhanced rates of leaching can be achieved in the potential window of about 560-600 mV with significantly lower rates at 540 and 620 mV. The reduction in rate at the low potential is due to the enhanced stability of covellite (CuS) at potentials below about 550 mV under these conditions while passivation of chalcopyrite occurs at potentials above about 620 mV - as will be discussed in a subsequent paper. Of interest is the relatively constant rate of dissolution that is observed in most experiments even up to 80% dissolution. It should be noted that the initial relatively rapid dissolution of about 5-7% of the copper in these and subsequent experiments with Concentrate1 is due to the greater reactivity of small amounts of bornite in the concentrate (Table 2).

3.3. *Chemical control of the potential*

One experiment was conducted in which the potential was controlled by injection of permanganate into the leach slurry of Concentrate 3 which was de-aerated by sparging with nitrogen. The results are shown in Figure 7 which also shows the corresponding curve for the dissolution of this concentrate under conditions of oxygen control. This result reconfirms the importance of dissolved oxygen and also that the phenomenon also applies to chalcopyrite from a different source. The role of dissolved oxygen will be discussed in a subsequent paper but it should be pointed out that enhanced rates are only observed when dissolved oxygen is present together with an oxidant such as copper(II) or iron(II) ions.

3.4. *Reversibility of operation outside the potential window*

3.4.1. *Effect of high potentials*

The above results have shown that at potentials above about 620 mV, the rate of dissolution decreases substantially. It is probable that this is due to the so-called passivation of chalcopyrite which has been previously observed from both dissolution and electrochemical studies (Nicol and Lazaro, 2003). In a practical implementation of controlled potential leaching, it would be desirable if passivation could be reversed by reduction of the potential into the operating window. Experiments were therefore conducted during which the potential was controlled initially at 650 mV and, after a period at this potential, it was reduced to 580 mV.

The results for experiments carried out with Concentrate 3 at both 25 and 50 °C are shown in Figure 8. The significantly higher rate at 580 mV at 50°C than at 25°C suggests a large activation energy for the dissolution reaction. It is apparent that slow passivation occurs at a potential of 650 mV even at 50 °C with the rate becoming almost zero after about 500 h. Reduction of the potential after 700 h results in restoration of the rate normally observed within the potential window. Similarly, the much slower rate at 25 °C at 650 mV increases to the normal rate at 580 mV when the potential is reduced after 1400 h. These results have shown that the reactions responsible for passivation are reversible. The relatively slow onset of passivation at elevated temperatures is at least a partial explanation for the fact that finely milled chalcopyrite can be effectively leached under relatively mild conditions.

3.4.2. *Effect of low potentials*

Two tests were carried in order to establish the extent to which chalcopyrite dissolves at low potentials (450 mV) and whether a subsequent increase in potential

results in similar or enhanced rates over those observed in the optimum potential window. The tests were carried out under otherwise standard conditions using nitrogen to maintain the low potentials. The results are shown in Figure 9 from which it is apparent that the test at low potential showed negligible dissolution of copper until the potential was increased to 550 mV after 940 h. It is clear that the rate on increasing the potential is similar to that observed under standard conditions i.e. there does not appear to be any net advantage in carrying out a 2-stage process with the first involving reducing conditions. Solid samples for mineralogical analysis were taken just before increasing the potential and on completion of the tests.

Figure 10 shows an optical micrograph of a reacted chalcopyrite particle removed after 936 h in this experiment. There is obvious conversion of chalcopyrite to covellite and small amounts of chalcocite. The results of mineralogical analyses of the head and residue samples from this test as estimated from a MLA investigation are presented in Table 3. As shown, the chalcopyrite content is slightly reduced after 936 h at low potential while there appears to be an increase in the content of covellite and bornite suggesting that chalcopyrite could have been reduced to these copper sulfides. After 2640 h, the amount of chalcopyrite and bornite has decreased while there is a no detectable covellite, but increased amounts of sulfur in the residue. It should be pointed out that the method of preparation of the polished sections can result in significant losses of elemental sulfur globules and that the amounts analyzed are therefore lower limits only.

The mineral maps (not shown) show that most of the un-reacted chalcopyrite is present as liberated particles with a few particles rimmed by elemental sulfur. In addition, it was interesting to observe the occurrence of sulfur as isolated and relatively large globules suggesting that the formation of elemental sulfur occurs by

precipitation from a dissolved sulfur species as has been previously suggested (Dutrizac, 1990; Schippers and Sand, 1999). This aspect will be dealt with in more detail in a later paper in this series.

3.5. *Dissolution of chalcopyrite concentrates from different sources*

A number of leach experiments were carried out using each of the concentrates described in Tables 1 and 2. The experiments were conducted with the +25-38 μm size fraction for Concentrate 1 and with the -38 μm fraction with the other samples due to the limited amounts of these concentrates available.

3.5.1. *Mineralogy*

The various samples of screened concentrates were analyzed chemically and mineralogically with the results given in Tables 1 and 2. The silver content of the various samples ranged from 50 to 10 ppm and this is of potential interest since soluble silver is known to accelerate the dissolution of chalcopyrite (Hiroyoshi et al., 2002; Hiroyoshi et al., 2007; Parker et al., 2003; Yuehua et al., 2002). It is apparent that the Concentrate 1 is the highest grade with respect to chalcopyrite and although data for Concentrate 4 was limited to chemical analysis for copper and iron only, mineralogical analyses of another sample from the same source indicated the presence of chalcopyrite and pyrite as the only measurable sulfide minerals. The other two materials contained significant proportions of the copper as other copper sulfides, as shown by the copper source ratios (percentage of copper present in the form of each mineral) in Table 4. The MLA analyses showed that the chalcopyrite particles are

mostly liberated in all samples with only small amounts associated with other minerals.

3.5.2. *Leaching experiments*

Each of the chalcopyrite samples was leached at a controlled potential of 580 mV using the nitrogen/air control system under standard conditions i.e., 0.2 M HCl and 0.5 g L⁻¹ Cu(II) at 35 °C. Figure 11 summarizes the leaching curves, which are approximately linear with remarkably similar rates for all concentrates. This confirms other work conducted under different conditions (Dutrizac, 1982). The initial relatively rapid rate of dissolution in some cases is due to the greater reactivity of the secondary copper sulfide minerals present mainly in Concentrates 2 and 3.

Chemical analyses of some of the residues gave values of the extent of dissolution of copper that were consistent with those calculated from the corresponding solution concentrations. In some cases, depending on the pH during the latter stages of leaching, precipitation of iron was observed which resulted in lower extraction of iron into solution. However, this did not appear to affect the rate of dissolution of copper.

4. Conclusions

Leaching experiments under controlled potential and standard conditions have demonstrated that the rate of dissolution of chalcopyrite is potential dependent and the presence of dissolved oxygen is essential for enhanced rates. However, it appears that the rate is not strongly dependent on the oxygen concentration in the range 5 to 15 mg L⁻¹. The linear rate of dissolution of chalcopyrite from several concentrates in chloride

solutions under standard conditions with a solution of 0.2 M HCl with 0.5 g L⁻¹ copper(II) at 35 °C is enhanced within a range of potentials of 550 to 620 mV (potential window).

Even though dissolved oxygen is important in order to achieve acceptable rates of dissolution, an excess of oxygen can increase the potential into a region in which passivation is possible. Initial leaching at potentials above 630 mV for extended periods results in low rates that are increased when the potential is subsequently reduced to 580 mV. Therefore, it appears that the “passivation” is reversible.

On the other hand, leaching at low potential (<540 mV) results in reduced rates of copper dissolution which increase significantly when the potential is subsequently increased to above 580 mV. Mineralogical studies have shown that chalcopyrite remains un-leached and small amounts of covellite and chalcocite appear to be formed on the surface of the chalcopyrite under these conditions suggesting the possibility of a two stage process in which chalcopyrite is converted to a more reactive secondary copper sulfide. However, the overall rate in this two-stage process appears to be similar to that achieved by operation as a single stage within the potential window.

All concentrates studied appear to dissolve at roughly the same rate under the same conditions, despite differences in the composition and mineralogy of the concentrate samples.

5. Acknowledgements

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Figures

Figure 1 Schematic of the potential control system

Figure 2 The effect of solution potential on the extent of copper dissolution from the mineral sample after 24 h in a solution of 1 M chloride, 0.1 M H₂SO₄ and 0.01 M Fe(III). (□) Initial potential, (■) Final potential

Figure 3 Copper dissolution from Concentrate 1 in a solution of 10g/L chloride at pH 0.5 and 20°C in the absence (■) of iron(III) and in the presence (▲) of 10g/L iron(III). Also shown are the corresponding potentials(----) during the experiment.

Figure 4 Copper dissolution from Concentrate 1 in a solution of 0.2 M HCl and 0.5 g L⁻¹ copper(II) under electrochemical control. (▲) 560 mV, (●) 580 mV, (■) 600 mV and (◆) 610 mV.

Figure 5 Copper dissolution from Concentrate 1 in a solution of 0.2 M HCl and 0.5 g L⁻¹ copper(II) at 35 °C under electrochemical control with sparging of (▲) nitrogen, (●) oxygen, (■) air.

Figure 6 Copper dissolution from Concentrate 1 in a solution of 0.2M HCl and 0.5 g L⁻¹ copper(II) at 35 °C under air control. (▲) 540 mV (●) 560 mV, (×) 580 mV, (◆) 600 mV and (■) 620 mV.

Figure 7. Copper dissolution from Concentrate 1 in a solution of 0.2 M HCl and 0.5 g L⁻¹ copper(II) at 35 °C at a controlled potential of 580 mV using (▲) oxygen and (●) permanganate.

Figure 8. Reversibility of passivation of copper dissolution from Concentrate 3 under air control. (Δ) 1 M HCl + 0.5 g L⁻¹ copper(II) at 50 °C and 580 mV, (▲) 1 M HCl + 0.5 g L⁻¹ copper(II) at 50 °C and 650 mV/550 mV, (■) 0.2 M HCl + 0.5 g L⁻¹ copper(II) at 25 °C and 650 mV/550 mV.

Figure 9. Dissolution of copper from Concentrate 1 in a solution of 0.2 M HCl and 0.5 g L⁻¹ copper(II) at 35 °C and (▲) 580 mV and (■) 450 mV with potential increased to 550 mV after 936 h.

Figure 10. Optical micrograph showing the conversion of chalcopyrite (yellow) in Concentrate 1 to covellite and chalcocite after 936 h at 450 mV. Conditions as in Figure 9.

Figure 11. Dissolution of copper from chalcopyrite concentrates from different localities in 0.2 M HCl and 0.5 g L⁻¹ copper(II) at 35 °C. (▲) Conc. 1, (■) Conc. 2, (×) Conc. 3, (●) Conc. 4

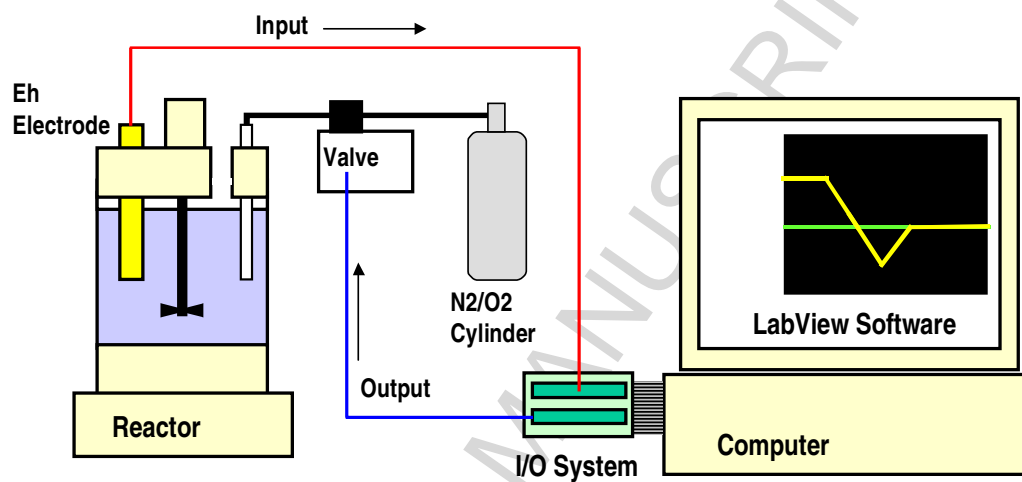


Figure 1

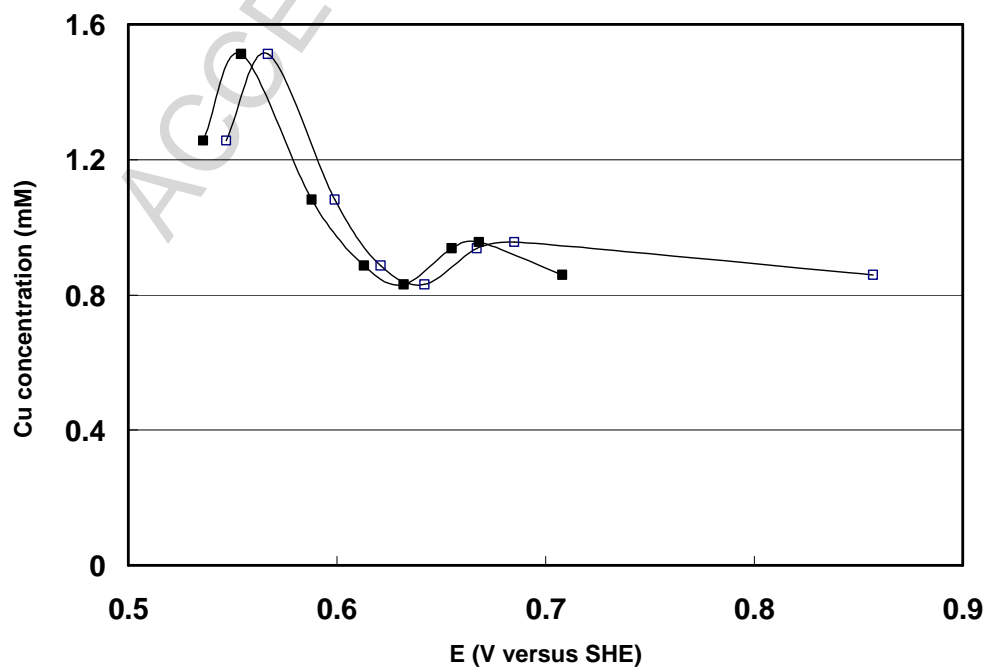


Figure 2

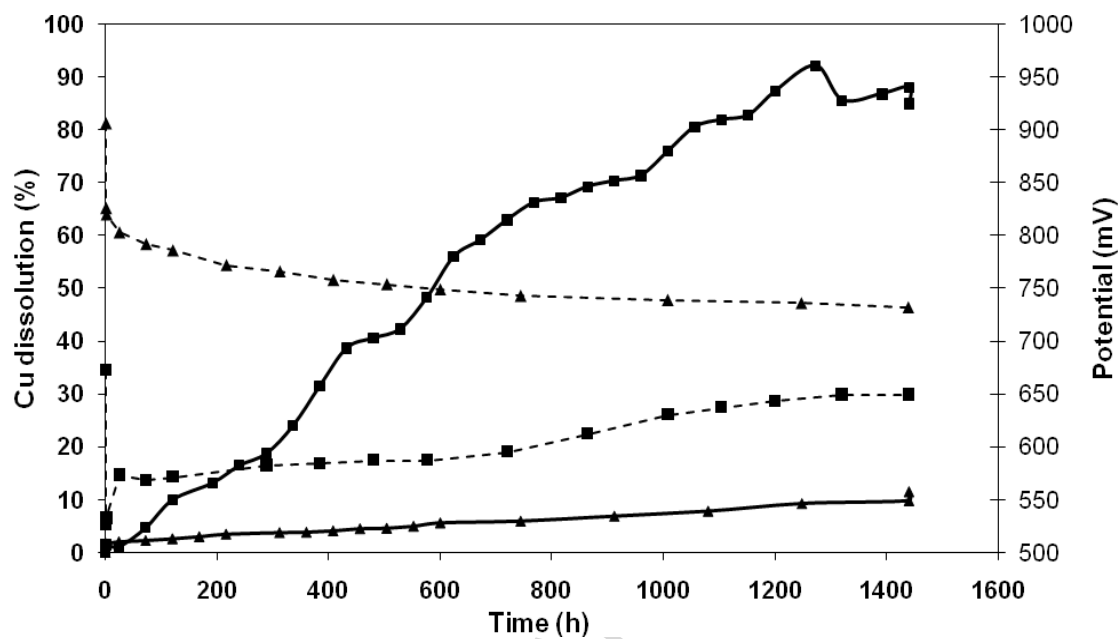


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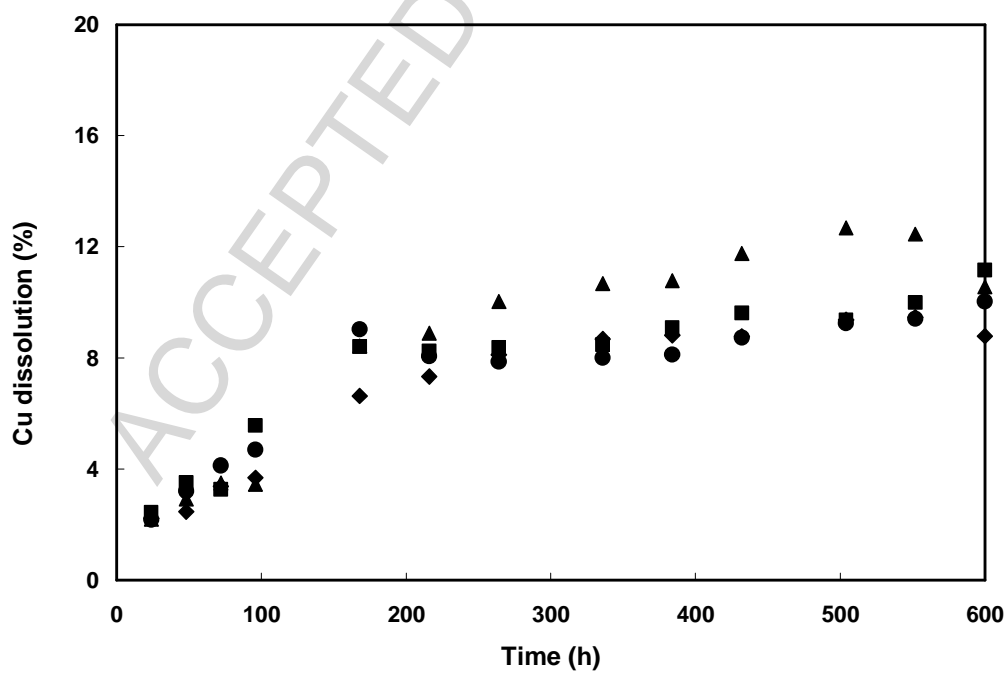


Figure 4

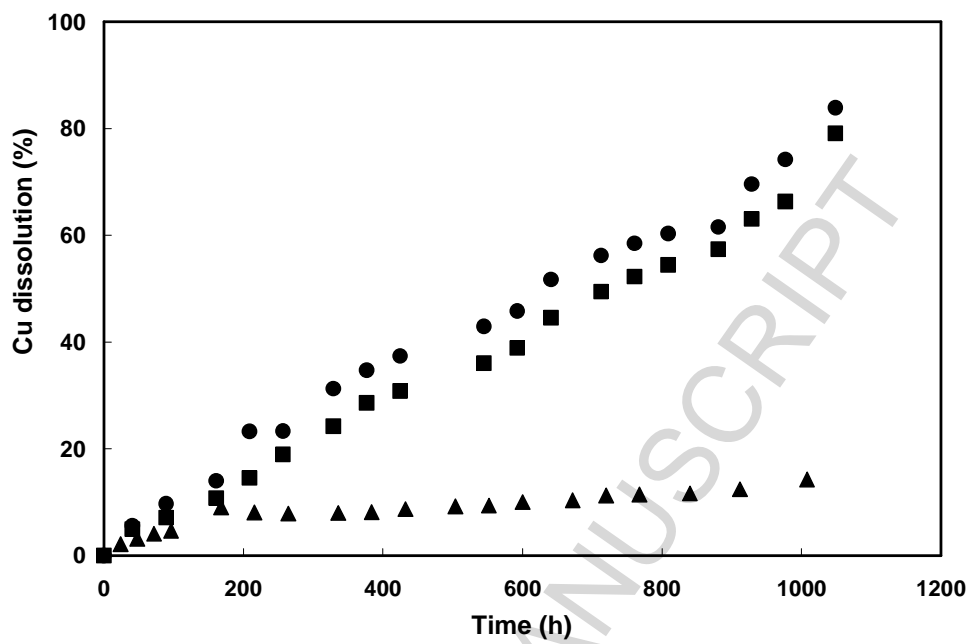


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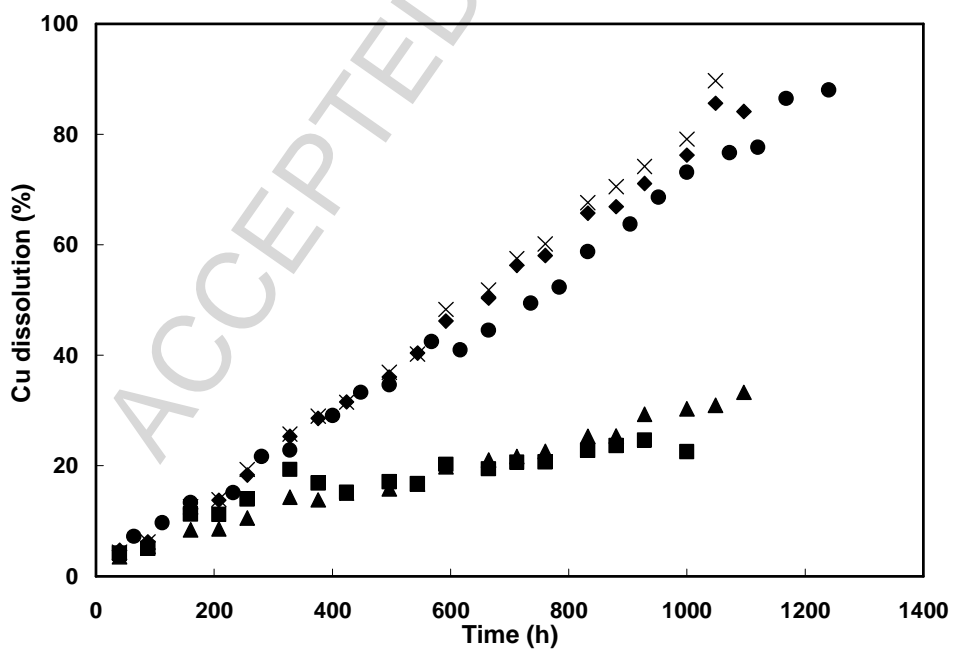


Figure 6

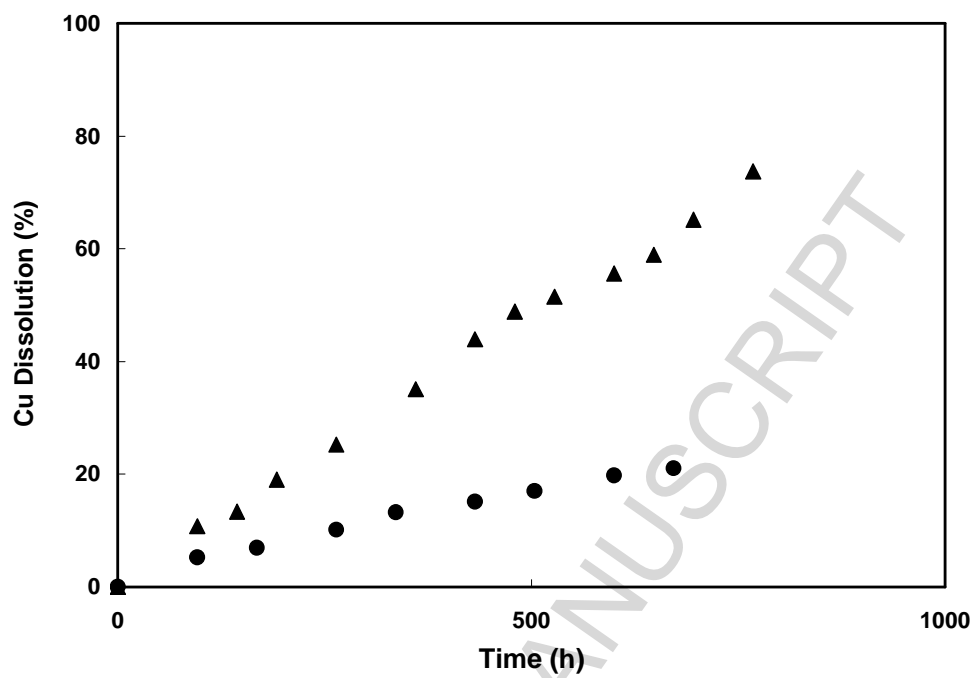


Figure 7

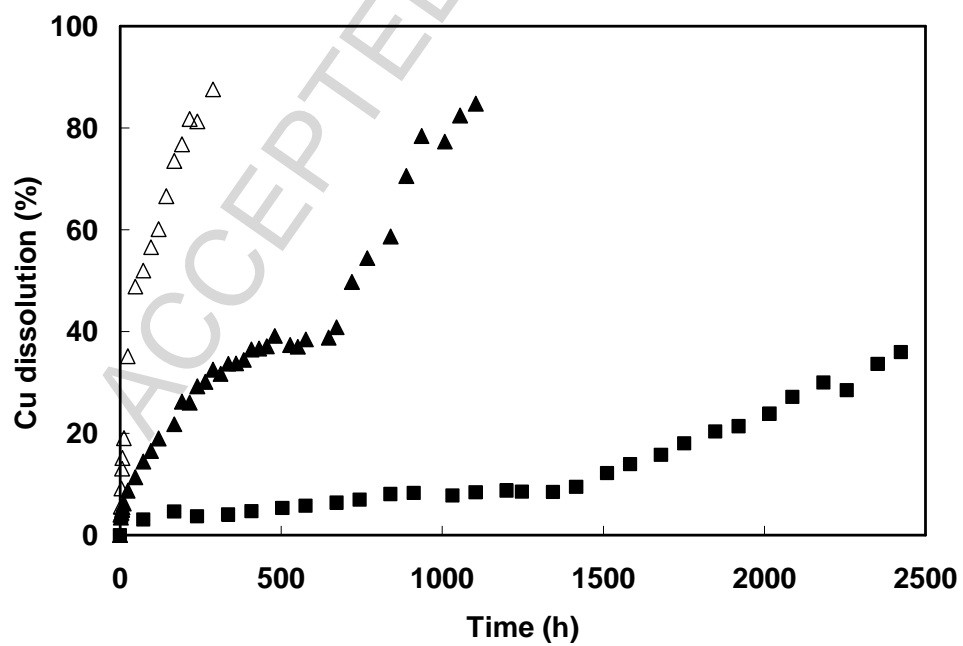


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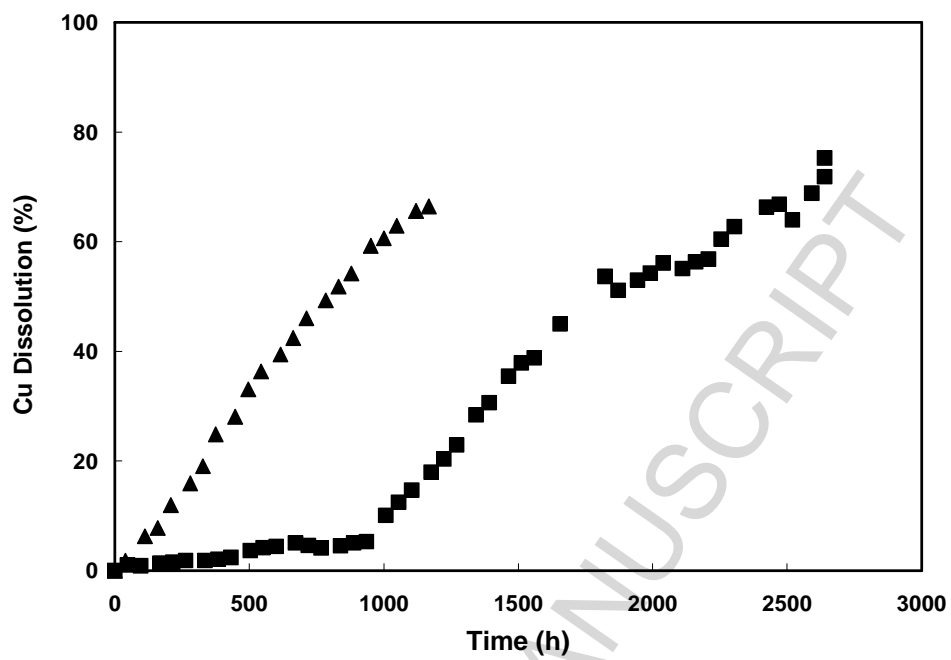


Figure 9.

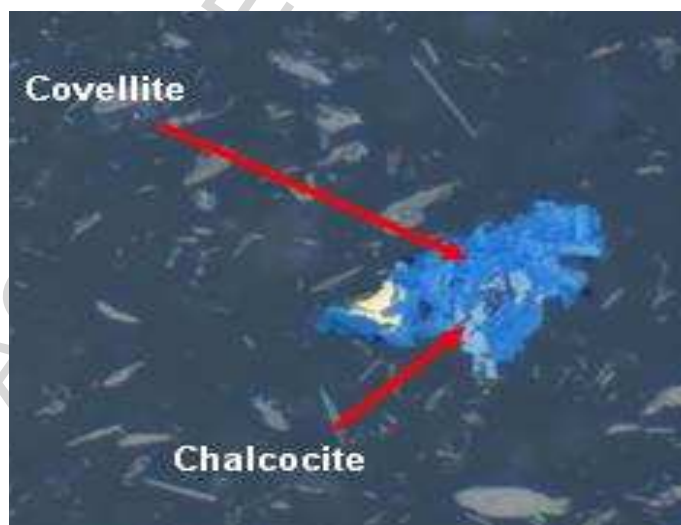


Figure 10.

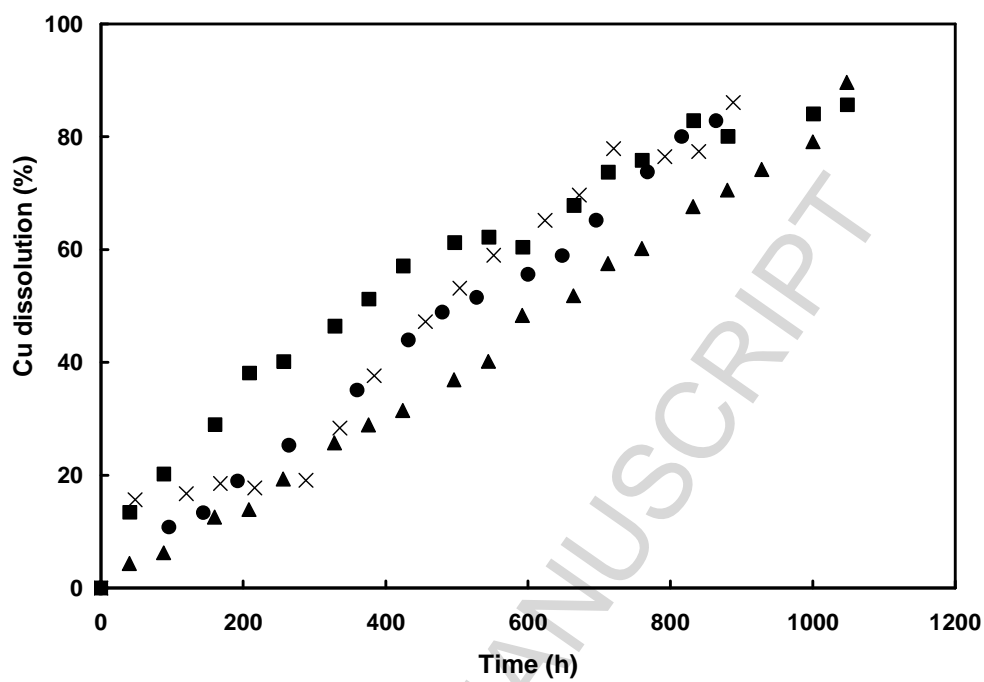


Figure 11.

Table 1. Chemical composition (element, mass %)

<i>Sample</i>	<i>Size (μm)</i>	<i>Cu</i>	<i>Fe</i>	<i>S</i>	<i>Ag (ppm)</i>
Mineral	+25-38	20.7	30.5	35.4	-
Concentrate 1	+25-38	30.8	28.3	32.4	50
Concentrate 2	-38	8.1	30	35.3	20
Concentrate 3	-38	13.2	21.8	23.3	30
Concentrate	-38	28.0	27.3	NA	NA

Table 2. Mineralogical composition of concentrates (mass, %)

<i>Mineral</i>	<i>Mineral +25-38 μm</i>	<i>Conc. 1 +25-38 μm</i>	<i>Conc. 2 -38 μm</i>	<i>Conc. 3 -38 μm</i>
Chalcocite	0.00	0.00	0.0	2.49
Chalcopyrite	77.90	82.50	17.6	32.98
Covellite	0.00	0.16	5.0	5.65
Bornite	0.01	3.40	1.7	2.68
Native_Cu	0.00	0.00	0.0	0.00
Enargite	0.00	1.70	1.2	0.73
Brochantite	0.00	0.00	0.0	0.23
Chrysocolla	0.00	0.00	0.0	0.04
Other Cu	0.00	0.00	0.0	0.05
Pyrite	17.9	2.70	62.2	34.33
Quartz	0.02	5.00	3.4	5.33
Galena	0.41	0.47	0.1	0.1
Gangue	4.02	4.03	8.8	15.39

Table 3. Modal mineralogical analysis (mass %) of Concentrate 1 and residues after dissolution under the conditions shown in Figure 9

<i>Mineral</i>	<i>Head</i>	<i>Residue 936, h</i>	<i>Residue 2640, h</i>
Chalcopyrite	82.5	80.2	57.1
Covellite	0.16	0.4	0.0
Bornite	3.4	4.0	1.9
Pyrite	2.7	5.5	12.0
Elemental Sulfur	0.0	0.0	5.7
Total	100	100	100

Table 4. Pyrite content and copper source ratios (mass %) of major copper minerals

<i>Sample Mineral</i>	<i>Concentrate 1</i>	<i>Concentrate 2</i>	<i>Concentrate 3</i>
Chalcopyrite	90.2	54.0	59.1
Covellite	0.2	31.0	19.6
Bornite	6.8	9.0	8.8
Enargite	2.6	5.0	1.8
Pyrite	2.7	62.2	34.3