The selective leaching of copper from a gold–copper concentrate in glycine solutions

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ABSTRACT

The presence of copper minerals with gold is known to lead to many challenges during the cyanidation of gold ores, such as high consumption of cyanide with low gold extraction and undesirable impacts on gold recovery during the downstream processes. An alternative selective leaching process for copper minerals from copper–gold gravity concentrate (3.75% Cu, 11.6% Fe, 11.4%S and 0.213% Au) using alkaline glycine solutions was studied and evaluated. The lixiviant system containing glycine and peroxide showed that total copper dissolution of 98% was obtained in 48 h at ambient conditions and a pH of 10.5–11. The results show that 100% of chalcocite, cuprite, metallic copper, and about 80% of chalcopyrite in the concentrate were also dissolved. Pyrite remained intact during the leaching time and iron concentration in the final pregnant solution was found to be 12 mg/L when copper is solution is at 4745 mg/L, whilst the gold concentration was limited to 0.8 mg/L Au. QEMScan analysis indicated that unleached copper in the leach residue was mostly distributed amongst larger chalcopyrite grains and covellite. The effects of single versus two-stage leaching, pH, oxidant concentration, pulp density and glycine concentration on copper extraction rate and extent were explored.

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1. Introduction

Recently, many researchers have focused on the recovery of copper from ores and concentrates using hydrometallurgical solutions in order to replace the traditional pyrometallurgical processes. Currently, more than 20% of world copper production is produced by using hydrometallurgical processes, particularly by acidic heap leach (or heap bioleach)–solvent extraction–electrowinning processes for low grade ores.

A review of the selective leaching of gold from oxidised copper–gold ores with ammonia–cyanide, and their plant control and operation has been published by Muir (2011). The application of cyanide–ammonia system to leach gold over copper from oxidised ores has been tested successfully using different gold–copper ores (Drok and Ritchie, 1997; La Brooy et al., 1991; Muir et al., 1989). However, this system in the treatment of transition or sulphidic ores gave poor gold recovery and required higher reagent concentrations (Muir, 2011). Moreover, both cyanide and ammonia have detrimental environmental effects.

Muir et al. (1989) reported that there are five methodologies for treating copper–gold ores: (1) float and produce a copper–gold concentrate for smelting; (2) selectively mine and cyanide leach ore with <0.5% Cu as oxidised minerals and <1% as sulphidic minerals; (3) cyanide leaching and selectively absorbing gold using either activated carbon or ion-exchange resins; (4) cyanide leaching and

selectively recovering copper from solution; and (5) selectively leaching either copper or gold with alternative reagents. The decision as to which of these approaches to apply depends upon the relative amounts of copper and gold present, the mineralogy and process economics.

This paper presents a process of selectively leaching the reactive copper before gold, as it is well known that the presence of copper minerals has detrimental effects on the cyanide leaching of gold resulting in a huge consumption of cyanide (La Brooy et al., 1994). Copper minerals consume about 30 kg/t NaCN for every 1% of reactive copper present, making conventional cyanidation of copper–gold ores or concentrates uneconomic (Muir, 2011). According to La Brooy (1992), the oxidised copper–gold ores are selectively mined to minimise the presence of reactive copper to below 0.5% and otherwise; sulphidic copper–gold ores are floated and gold is recovered as a by-product of copper smelting and refining. However, when copper is present in significant levels with gold in gravity concentrates, it can also lead to complexities with direct smelting of the concentrates.

There are a number of large copper–gold mines in Australia and the Asia-Pacific region. These plants produce copper–gold concentrates and ship them for copper smelting and gold recovery from anode slimes (e.g. Telfer, Mt Carlton, Boddington and Cadia Valley, etc.). However, the increase of pyrite content in the high grade copper ores results in the production of low grade copper concentrate. In addition, the presence of arsenic limits the flotation mass pull of chalcopyrite rich concentrates, so that a significant portion of the gold have to be recovered by gravity and leaching of the flotation tails. The transportation of the

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low grade concentrate overseas is often uneconomical. Therefore, an alternative process would be useful for recovering both copper and gold from such a low-grade concentrate. Cyanide–ammonia leaching of Telfer flotation tailings has been carried out by Muir et al. (1991) for 24 h with 0.3 M NH₃/0.1 M (NH₄)₂SO₄. This process extracted 82% Cu and no gold, followed by subsequent cyanidation of the washed leach residue. The proposed process depends on copper selective leaching before gold in an alkaline glycine solution followed by washing and gold leaching either using the same process at elevated temperature (40–60 °C), as patented by the authors, or using a cyanidation process. Copper selective leaching will reduce the gold losses due to a cementation mechanism of gold in the presence of metallic copper as all of the metallic copper will dissolve in the pre-leaching stage. Nguyen et al. (1997a, 1997b) found that, at ambient conditions and pH 11, all gold in a solution containing 1000 mg/L NaCN and 0.5 g/L Cu had been cemented on copper in the first 2 h.

According to Stewart and Kappes (2012), the presence of reactive copper in high percentages in a gold ore has negative effects on the efficiency and economic viability of any gold project due to (1) high cyanide consumption in which copper will consume at least 2.3 kg of sodium cyanide for every kilogramme of copper leached; (2) copper competing with gold in the adsorption/stripping circuit, which can effectively reduce the gold loading capacity of the carbon, increasing the plant size and the cost of the adsorption circuit. In addition, the high level of copper in cyanide as weak acid dissociable (WAD) cyanide creates a problem that requires expensive processes such as sulfidisation, acidification, recycle and thickening (SART) to be operated to recover cyanide (Simons and Breuer, 2013), often in a dilute form which may create water balance problems with the plant. Alternatively a range of WAD cyanide destruction processes are to be implanted, each with significant concomitant costs.

The main aim of this work was therefore to investigate an environmentally benign hydrometallurgical process to selectively leach copper from copper–gold resources with mixed mineralisation. To this end, a gravity gold concentrate was selected with high levels of copper distributed amongst a range of sulphide and oxide minerals, as well as native copper. The gravity concentrate has sufficient gold to evaluate the effect of the lixiviant system on gold dissolution under the proposed leaching conditions.
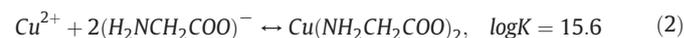
1.1. Background to copper–glycine leaching

Glycine is one of the simplest and cheapest amino acids, and is available in bulk. It has a number of attractive chemical and physical properties and it will be used as a main reagent in this research. Glycine has a number of advantages over many other copper lixiviants: it is an environmentally safe and stable reagent, yet it is enzymatically destructible and is easily metabolised in most living organisms. Due to its complexing action, glycine can also enhance the solubility of copper ions in aqueous solutions (Aksu and Doyle, 2001, 2002). Potentiometric

studies on complexes formed by essential metals (Cr, Mn, Fe, Co, Ni, Cu and Zn) and different amino acids have been conducted by Aliyu and Na'aliya (2012). The stability constant of glycine with copper according to Aliyu and Na'aliya (2012) is 18.9.

The complexing mechanism of copper in solutions containing glycine involves, initially, the formation of a hydroxyl copper complex by an ion-exchange mechanism.

Glycine can exist in aqueous solutions in three different forms, namely $^{+}H_3NCH_2COOH$ (cation), $^{+}H_3NCH_2COO^-$ (zwitterion), and $H_2NCH_2COO^-$ (anion). It also forms a strong complex with copper (II) as $Cu(H_2NCH_2COO)_2$ and with copper (I) as $Cu(H_2NCH_2COO)^-_2$ (Eqs. (1), (2) and (3)) and can enhance the solubility of copper ions in aqueous solutions due to its ability to chelate copper (Aksu and Doyle, 2001).

$$Cu^{2+} + (H_2NCH_2COO)^- \leftrightarrow Cu(NH_2CH_2COO)^+, \quad logK = 8.6 \qquad (1)$$

$$Cu^{2+} + 2(H_2NCH_2COO)^- \leftrightarrow Cu(NH_2CH_2COO)_2, \quad logK = 15.6 \qquad (2)$$

$$Cu^+ + 2(H_2NCH_2COO)^- \leftrightarrow Cu(NH_2CH_2COO)^-_2, \quad logK = 10.1 \qquad (3)$$

A glycine–peroxide system is widely used as a polishing mixture in the chemical–mechanical planarization (CMP) technique. Mixtures of glycine and hydrogen peroxide have shown promising copper CMP behaviour and it was found that the glycine–peroxide mixture is successful to leach metallic copper from the exposed areas during the planarization (Aksu and Doyle, 2001; Ein-Eli et al., 2004; Hirabayashi et al., 1996).

As an example, the stoichiometry of cupric ions complexing in alkaline glycine solutions is described in Eq. (4).

$$Cu^{+2} + 2(NH_2CH_2COOH) + 2OH^- \rightarrow Cu(NH_2CH_2COO)_2 + 2H_2O \qquad (4)$$
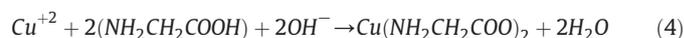
In a recent provisional patent, Oraby and Eksteen (2013) and recent research, Eksteen and Oraby (2014) and Oraby and Eksteen (2014), the authors developed a process using an alkaline amino acid–peroxide lixiviant system to leach gold, silver and their alloys at mildly elevated temperatures (40–60 °C) and dilute and concentrated concentrations. It was found that the elevated temperature (~60 °C) and hydrogen peroxide addition was crucial to obtain feasible leach rates of precious metals. This should be contrasted to the temperatures and hydrogen peroxide requirements as investigated for the alkaline copper–glycine system discussed below.

This study therefore addresses a persisting challenge in the copper and copper–gold processing, i.e. finding an appropriate lixiviant with lower environmental and safety risks for heap, dump, in-situ and vat leaching operations, which are slow leaching operations open to the surrounding environment. Alternatively, it may also be implemented in a counter-current decantation (CCD) circuit, allowing for sufficient residence time.

2. Experimental

2.1. Sample preparation

The concentrate was produced from the cyclone underflow feeding into a batch centrifugal gravity separator at a copper–gold plant in Western Australia. The gravity concentrate conveniently concentrates many non-sulphide minerals of copper, native copper, as well as sulphide minerals, with gold to provide a wide distribution of copper mineralogy. The gravity concentrate sample was then ground using a disc mill and screened using 150 and 106 μm screens. The +150 μm was recycled back to the mill. The particle size of the ground sample used

Table 1
Solubility of Cu minerals in 1 g/L NaCN solution (Hedley and Tabachnick, 1968).

Mineral	Formula	%copper dissolved[a]
Azurite	$2Cu(CO)_3 \cdot Cu(OH)_2$	94.5
Malachite	$2CuCO_3 (OH)_2$	90.2
Chalcocite	Cu_2S	90.2
Covellite	CuS	95.6
Chalcopyrite	$CuFeS_2$	5.60
Native Copper	Cu	90.0
Cuprite	Cu_2O	85.5
Bornite	$FeS \cdot 2Cu_2S$	70.0
Enargite	Cu_3AsS_4	65.8
Tetrahedrite	$(Cu \cdot Fe \cdot Ag \cdot Zn) 12Sb_4S_{13}$	21.9
Chrysocolla	$CuSiO_3 \cdot (nH_2O)$	11.8

[a] Percent total copper dissolved at 23 °C in agitated tank for 24 h, ratio of solution to ore 10:1 (cited in Coderre and Dixon, 1999).

Table 2
Elemental analysis of the copper–gold concentrate.

Concentration (%)											
Sample ID	Au	Ag	Cu	As	Fe	Si	Ni	Al	K	Co	Pb	S
Conc.	0.213	0.03	3.75	0.76	11.6	27.0	0.06	2.0	0.69	0.34	0.12	11.4

in the leaching experiments was 100%–150 μm and 80%–106 μm. The original sample has been sent for elemental analysis using acid digestion followed by solutions analysis for different metals using inductively coupled plasma optical emission spectrometry (ICP-OES). The mineralogical compositions of the gravity concentrate before and after leaching were analysed by an integrated automated mineralogy solution providing quantitative analysis of minerals using quantitative evaluation of minerals by scanning electron microscopy (QEMSCAN) technique at CSIRO Process Science and Engineering branch, Waterford, WA.

2.2. Leaching

All experiments were carried out using solutions prepared from analytical grade reagents and Millipore water. Unless specified, all experiments are conducted using a bottle roller, and the concentrate and glycine solution were placed in a 2.5 L Winchester bottle. The slurry was agitated by rolling the bottle on a bottle roll at 150 rpm. Bottles are vented to allow for oxygen transfer, ensuring that a lack of oxygen does not limit the reaction rate. At different times, samples of the leach solution were obtained using a syringe-membrane filter (pore size 0.45 μm). The filtrate were analysed for copper and iron by using atomic absorption spectrometry. The trace elements were analysed using inductively coupled plasma optical emission spectrometry (ICP-OES). The elemental analysis of residue was conducted using acid digestion followed by ICP-OES analysis.

3. Results and discussions

3.1. Two-stage leaching

From the data shown in Table 1, it can be seen that chalcopyrite (CuFeS$_2$) is the least soluble in cyanide and other copper oxides and native copper show high solubility in cyanide solutions.

Table 3
The mineralogical analysis of copper and gangue minerals in the concentrate before and after leaching (leaching conditions: 0.3 M glycine, 1% H$_2$O$_2$, pH 11, room temperature).

Bulk mineralogy, mass%		
Mineral	Before leaching	After leaching
Chalcocite/digenite	1.46	0.01
Cu-metal	1.30	0.00
Cuprite	0.88	0.00
Chalcopyrite	0.71	0.15
Bornite	0.27	0.02
Covellite	0.18	0.12
Exotic complex Cu-sulphides	0.09	0.00
Cu boundaries	0.34	0.02
Pyrite	28.55	32.33
Pyrrhotite	0.35	0.20
Arsenopyrite	0.01	0.00
Quartz	48.49	51.28
Feldpars	5.32	5.59
Calcite	0.12	0.10
Dolomite	0.09	0.11
Ankerite/Dolomite	0.78	0.61
Rutile/Anatase/Ilmenite	0.52	0.75
Hematite	0.69	1.71
Goethite	2.72	2.21
Other	7.16	4.75
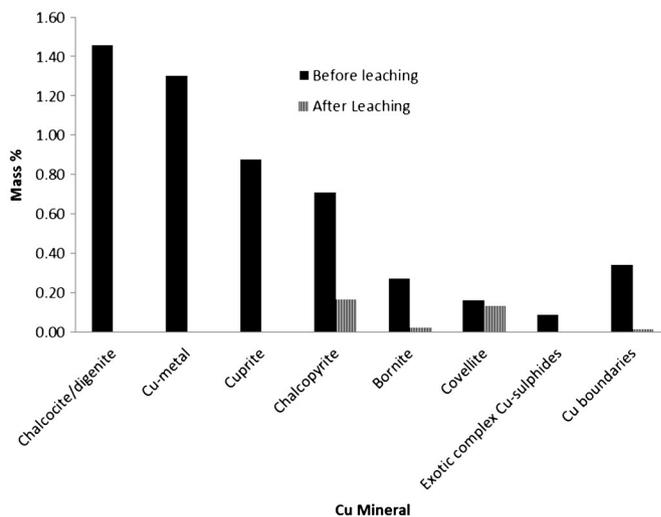
Fig. 1. Copper mineral in the concentrate before and after leaching in glycine solutions (leaching conditions: two-stages, 0.3 M glycine, 1% peroxide, RT, pH 11, 48 h).

Table 2 shows the elemental analysis of the copper–gold concentrate. The copper–gold concentrate has 3.75% Cu distributed amongst a range of copper minerals, including native copper.

The experiments showed that 98% of Cu was extracted in 48 h in two stages under the following conditions: 0.3 M glycine, 1% H$_2$O$_2$, pH 11.0, 23 °C and 16% (% w/v) pulp density. The mineralogical compositions of the copper–gold concentrate before and after leaching were analysed by QEMSCAN as shown in Table 3. Fig. 1 shows also the mass percentages for each copper mineral in the concentrate before and after leaching. The results presented in Fig. 1 show that 100% of the metallic copper and sulphide copper minerals, such as bornite and chalcocite/digenite, were dissolved. About 80% of chalcopyrite was dissolved. QEMScan analysis of the residue showed that the unleached chalcopyrite occurred as liberated particles in the largest size fraction.

Table 4 presents the percentage of each copper mineral dissolved after leaching in glycine solution. The presence of covellite (CuS) in the final residue may be attributed to the re-precipitation of copper during leaching according to Eq. (5). The formation of covellite (CuS) during the leaching of copper sulphide minerals, such as chalcopyrite, has been identified by some studies (Ammou-Chokroum et al., 1981; Klauber et al., 2001; McMillan et al., 1982; Parker et al., 1981). It can be concluded that covellite dissolves in the leach solutions and re-precipitates again by the reaction of copper with either sulphur or sulphide during the leaching.

$$Cu + S^0 \rightarrow CuS \quad \text{Or} \quad Cu_2S \rightarrow CuS + Cu^{2+} + 2e^- \tag{5}$$
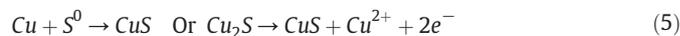
Fig. 2 shows the schematic diagram of a conceptual two-stage counter-current copper leach circuit.

Table 4
The observed dissolution of copper minerals in 0.3 M glycine solution at room temperature, 1% H$_2$O$_2$, pH 11.

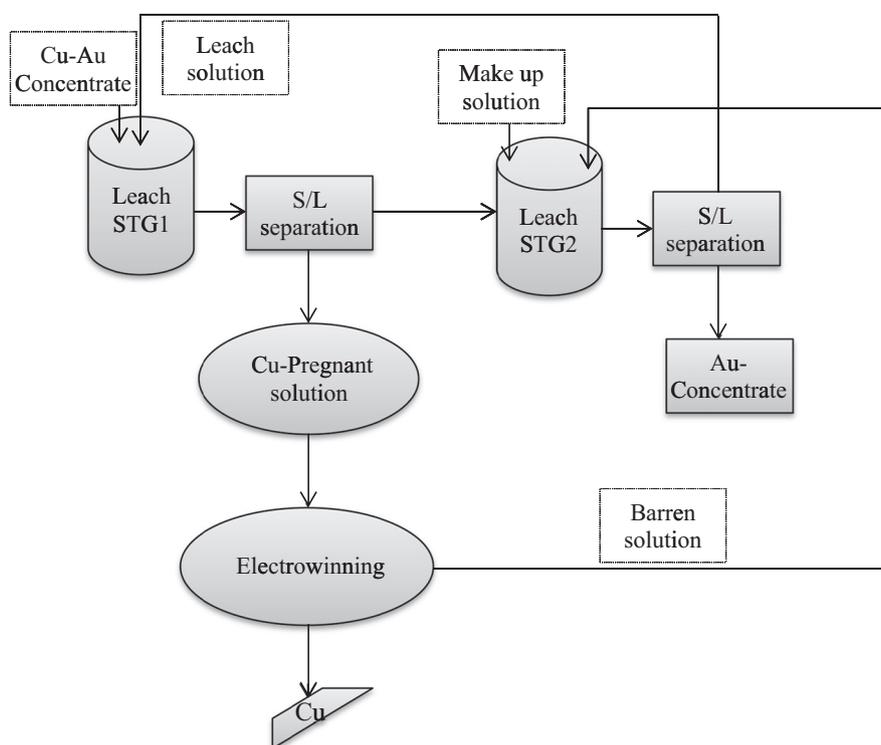| Mineral	Cu dissolution, %
Chalcocite/digenite	100
Cu-metal	100
Cuprite	100
Chalcopyrite	80
Bornite	92
Covellite	19
Exotic complex Cu-sulphides	99
Cu boundaries	95

Fig. 2. Schematic diagram for two-stage counter-current Cu leach process.

In order to achieve a high copper dissolution, the leaching can be conducted in two stages (two leaching reactors) with identical leaching conditions in each stage. At the steady state, fresh Cu–Au concentrate will feed reactor 1 (stage 1) and fresh solution will feed reactor 2 (stage 2). Table 5 shows the copper and other impurity concentrations in the final leach solution from stages 1 and 2.

About 98% of copper was leached using 0.3 mol/L glycine at room temperature with only about 12 mg/L Fe, 16 mg/L Pb and low impurity concentration transferred into the pregnant solution. It appears that iron does not dissolve significantly in the alkaline glycine solutions.

Fig. 3 shows the copper dissolution as a function of time after leaching in two stages. It can be seen that more than 98% of copper has been dissolved. It can also be observed that, after 5 hour leaching, copper extraction is about 66% of copper and this initial rapid dissolution of copper is due to the presence of the highly soluble cuprite and metallic copper in the concentrate.

3.2. Single stage leaching

In this section, leaching of a copper–gold concentrate is performed in a single stage by increasing glycine concentration from 0.3 M to 0.4 M and extending the leaching time to 96 h. Fig. 4 shows the mass percent of the copper minerals analysed by QEMSCAN before and after leaching for a comparison. From the copper concentration in the final leach solution, and copper in the final residue, the copper extraction was 82%. From the results shown in Fig. 4, it is apparent that 100% of chalcocite, cuprite, metallic copper, and only 50% of chalcopyrite have been

dissolved. It is interesting to observe that, as shown in Fig. 4, copper has been re-precipitated as a covellite (CuS) or as very fine copper either adsorbed on clays or incorporated in the silicates (cu-boundaries).

3.2.1. Effect of pH

The effect of leaching solution pH on copper dissolution is shown in Fig. 5. It can be seen that the initial copper dissolution at lower pH (pH 8.0) is higher than the dissolution at pH 10 and 11.5. However, by extending the leaching time it was found that the copper dissolution increases at more alkaline pH (pH 11.5). From the results shown in Fig. 4, it can be observed that copper dissolution slightly increases by increasing the leaching pH from pH 10 to pH 11.5.

3.2.2. Effect of peroxide

To study the effect of using peroxide as an oxidant on copper leaching in glycine solution, 0%, 1% and 2% of peroxide were added to 0.3 M glycine solution at room temperature. The results shown in Fig. 6 show that peroxide slightly increases copper dissolution. According to Du et al. (2004) the cathodic reaction in glycine solutions in the presence of copper ions without H_2O_2 may be written as shown in Eq. (6). However, in the presence of peroxide, the cathodic reaction changes to Eq. (7) or (8):

$$O_2 + 2H_2O + 4e^- \leftrightarrow 4OH^- \qquad (6)$$

$$H_2O_2 + 2e^- \leftrightarrow 2OH^- \qquad (7)$$

Table 5
Copper and impurities concentrations in stage 1 and 2 final leach solutions.

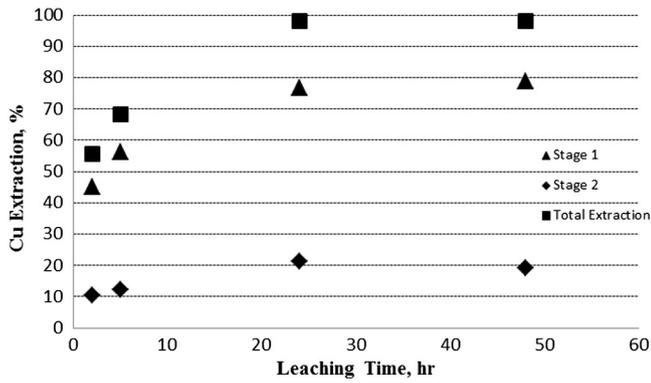| Concentration (mg/L)											
Sample ID	Cu	Au	As	S	Fe	Si	Ni	Co	Pb	K	Mg	Ca
Stage 1	4745	0.85	24.7	185	12.1	9.14	5.21	3.14	16.4	14.8	14.4	25.1
Stage 2	1069	1.28	29.4	146	6.69	6.45	4.34	2.27	10.2	9.2	2.99	14.1

Fig. 3. Copper extractions from Cu–Au concentrate after two-stage leaching (Stage 2 starts at zero time after stage 1).

Or

$$H_2O_2 + e^- \leftrightarrow OH^\bullet + OH^- \qquad (8)$$

In the solution containing hydrogen peroxide, the copper extraction increases due to the simultaneous increase of the cathodic reaction rate. In addition, the copper extraction increases due to the presence of hydrogen radical ($OH^\bullet$), which has a highly positive standard reduction potential of 2.38 V (Hoare, 1985). The most interesting result here is that copper extraction reaches up to 75% in a solution containing only glycine on a vented bottle roll that allows enough oxygen transfer from surrounding air.

3.2.3. Effect of pulp density

To study the effect of pulp density on copper leaching in glycine solution, 10, 16 and 20% pulp densities were used. The effect of pulp density on copper dissolution is shown in Fig. 7. Increasing the pulp density from 10% to 20% decreases the copper extraction by about 10%. The decrease of copper extraction at a higher pulp density can be attributed principally to the efficiency of oxygen transfer to the leach solution. Deng and Muir (1993) also found that copper extraction from leaching the same concentrate in chloride solution decreases by increasing the pulp density.
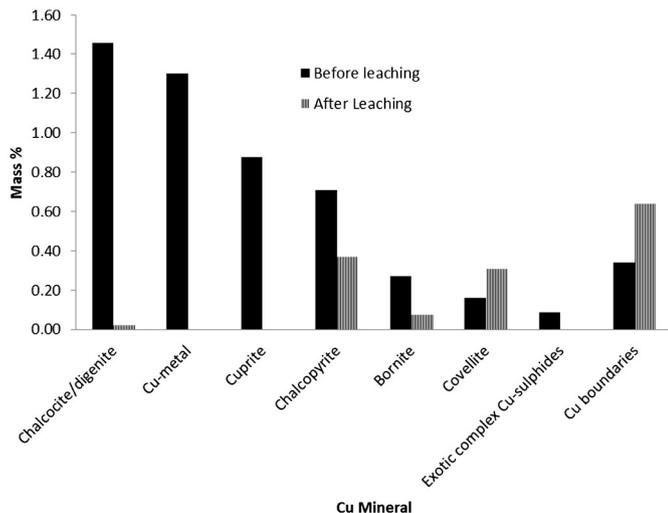

Fig. 4. Copper minerals in the concentrate before and after leaching in glycine solutions (leaching conditions: one-stage, 0.4 M glycine, 1% peroxide, RT, pH 11.5, 96 h).
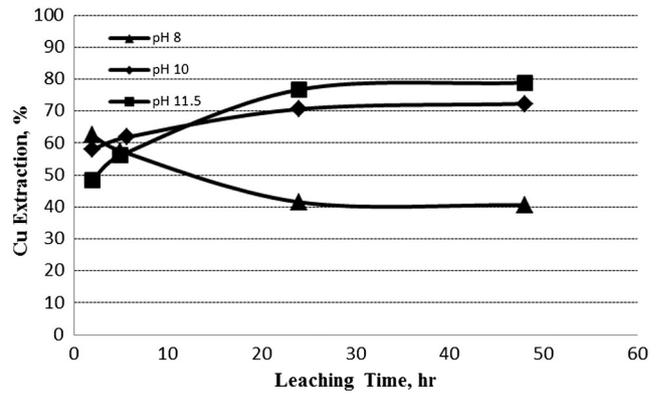

Fig. 5. Effect of leaching solution pH on copper dissolution: 0.3 M glycine, 1% H_2O_2, pH, at room temperature.
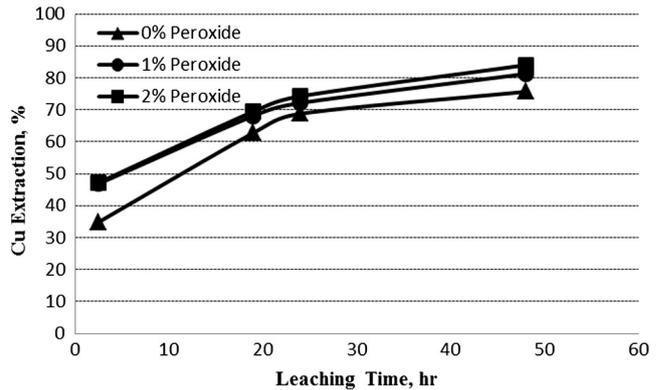

Fig. 6. Effect of peroxide on copper dissolution: 0.3 M glycine, H_2O_2, pH 11, RT, 10% pulp density.

3.2.4. Effect of glycine concentration

The effect of glycine concentration on the copper dissolution is shown in Fig. 8. It is clear that by increasing the glycine concentration, the copper extraction slightly increases. It can be generally reported that copper dissolution depends on the concentration of glycine in the glycine–peroxide solutions.
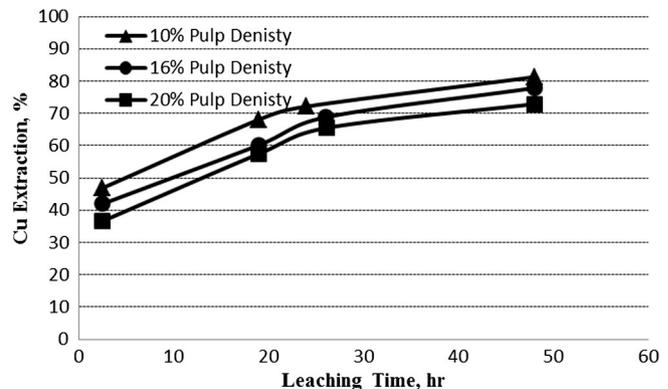

Fig. 7. Effect of pulp density on copper dissolution: 0.3 M glycine, 1% H_2O_2, pH 11, RT, pulp density.
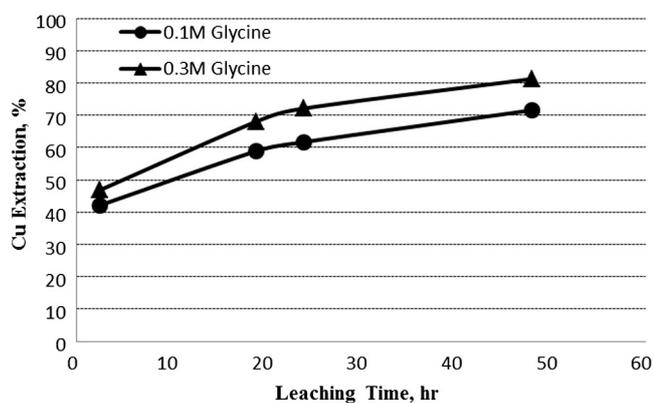
Fig. 8. Effect of glycine concentration on copper dissolution: Glycine, 1% H$_2$O$_2$, pH 11, RT, 10% pulp density.

4. Conclusions

Selective leaching of copper minerals over gold from copper–gold concentrate has been developed in glycine solution in the presence and absence of peroxide. The results obtained from this study can be concluded as follows:

1. Copper can be selectively leached over gold in glycine–peroxide solution. Nearly about 98% of copper present in the concentrate was leached in 48 h in two stages.

2. 100% of chalcocite, cuprite, metallic copper, and about 80% of chalcopyrite have been dissolved in glycine–peroxide solution in 48 h at ambient temperature.

3. Gangue element dissolution, in particular that of iron, is minimal compared to conventional sulphuric acid based leaching.

4. Leaching experiments showed that the copper extraction increases by increasing the glycine and peroxide concentrations. However, it was found that glycine can effectively leach copper in the absence of peroxide.

5. The leaching process shows that pyrite remains intact during the leaching time and iron concentration in the final pregnant solution is about 12 mg/L.

6. The leaching process is conducted under an alkaline leaching condition so the final reside can be directly leached by the cyanidation process.

7. The cyanide-soluble fraction of the copper minerals has been significantly reduced, thereby having a large impact on downstream cyanide consumption during the leaching of gold. The alkaline leaching of the copper minerals also implies that no swing in pH (as for a conventional acid leach) is required that would increase caustic requirements downstream.

Given that a mixture of copper minerals was leached, there might have been galvanic interactions between minerals that may have promoted the dissolution of some of the more refractory copper minerals such as chalcopyrite.

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