

Article

A Study of the Effect of Djurliete, Bornite and Chalcopyrite during the Dissolution of Gold with a Solution of Ammonia-Cyanide

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Abstract: The high solubility of copper sulphide minerals is an issue in the cyanidation of gold ores. The objective of this study was to quantify the effect of individual copper sulphide minerals on the Hunt process, which showed advantages over cyanidation. High purity djurleite, bornite and chalcopyrite, with a P₇₀ of 70–74 microns, were mixed with fine quartz and gold powder (3–8 micron) to obtain a copper concentration of 0.3%. The ammonia-cyanide leaching of slurry with djurleite proved to be more effective than cyanidation; producing comparable extraction of gold (99%), while reducing the cyanide consumption from 5.8 to 1.2 kg/t NaCN. Lead nitrate improved the Hunt leaching. The lower cyanide consumption is associated to a significant reduction of copper dissolved. XPS surface analysis of djurleite showed that lead nitrate favored the formation of Cu(OH)₂ species. Lead was also detected on the surface (oxide or hydroxide). Sulphide and copper compounds (cyanide and sulphide) were reaction products responsible for inhibiting the dissolution of gold. Lead nitrate added in the Hunt leaching of bornite produced 99% gold extraction. Surface reaction products were similar to djurleite. The cyanide consumption (~4.4 kg/t NaCN) was not reduced by the addition of ammonia. Cyanidation

of chalcopyrite showed a lower consumption of cyanide 0.33 kg/t NaCN compared to 0.21 kg/t NaCN for Hunt. No significant interferences were observed in gold leaching with a slurry containing chalcopyrite.

Keywords: gold; cyanide; ammonia, copper minerals; chalcopyrite; bornite; djurleite

1. Introduction

Australian organizations invested considerable efforts in the development and application of the ammonia-cyanide leaching (Hunt process) for the extraction of gold from ores containing a high concentration of copper in the 1990s [1–6] and 2000s [7,8]. Early work on leaching of the Buzwagi copper-bearing ores demonstrated the potential of the Hunt process in terms of gold extraction, consumption of cyanide and copper dissolution [9]. It was found that the control of cyanide concentration is a key parameter. High cyanide dosage improves the leaching of gold by providing more free cyanide. Meanwhile, the dissolution of copper minerals is enhanced. A high ratio of cyanide to ammonia jeopardizes the Hunt mechanisms due to the increased stability of Cu(CN)₃²⁻. Therefore, the optimum leaching conditions have to avoid high cyanide concentrations and to provide control the ratio of cyanide and ammonia within a narrow range.

There is limited information on the behavior of individual copper mineral in the Hunt process. In this paper, the effect of chalcopyrite ($CuFeS_2$), djurleite ($Cu_{31}S_{16}Cu_{1.94}S$) and bornite (Cu_5FeS_4), which are among the most common copper sulphide minerals associated to gold ores, are investigated.

2. Experimental Section

2.1. Material

A synthetic gold ore was produced by mixing gold grains at a concentration of 10g/t (3–8 micron) with 200 grams of quartz fines (P_{99} of 70–74 micron) and about 0.3% weight of copper as one of the following three natural copper sulphides minerals: djurleite, chalcopyrite and bornite with 93.8%, 99.9% and 96.85% purity, respectively. The mineralogical and elemental compositions of these minerals are shown in Tables 1 and 2. Djurleite ($Cu_{31}S_{16}$) has a chemical composition which is very similar to chalcocite (Cu_2S). The main differences between this mixture of quartz and gold and a real gold ore are the all the gold is free for leaching and the degree of purity of the mixture.

The minerals were ground to a P_{70} of 70–74 microns with a micro mill and stored in a desiccator in sealed plastic bags. The amount of minerals added in each test was based on the copper grade and mineral purity. The gold powder was added to ten kilograms of quartz fines, tumbled overnight to homogenize the sample and split in 200 g lots using a rotary separator.

Table 1. Elemental compositions of copper sulphide minerals.

Element	Bornite (%)	Djurleite (%)	Chalcopyrite (%)
Al	0.44	0.01	0.006
As	0.88	bdl	< 0.003
Bi	7.51	0.01	< 0.003
Ca	4.34	0.03	< 0.07
Co	0.12	bdl	0.02
Cu	67.7	79.7	33.2
Fe	10.8	0.07	32.2
Pb	0.02	bdl	0.01
S	26.6	21.8	34.5
Sb	0.01	bdl	< 0.002
Si	0.06	0.08	0.06
Te	Bdl	0.01	< 0.008
Zn	0.12	bdl	0.23

bdl: below detection limit.

Table 2. Mineralogical compositions of copper sulphide minerals.

Bornite		Dijurleite		Chalcopyrite	
Minerals	% w/w	Minerals	% w/w	Minerals	% w/w
quartz	0.1	quartz	0	chalcopyrite	99.9
bornite	96.8	dijurleite	93.8	hematite	0.1
chatkalite	0.8	Cu-Cl (hydromelanothallite)	2.9	_	_
acanthite	0.2	magnetite	0.03	_	_
pyrite	1.1	K-feldspar	0.04	_	_
pypsum	0.1	digenite	0.05	_	_
castaingite	0.1	brochantite	2.8	_	_
apatite	0.01	sphalerite	0.003	_	_
rutile	0.01	chrysocolla	0.2	_	_
wolframite	0.01	_	_	_	_
chalcopyrite	0.5	_	_	_	_
sphalereite	0.1	_	_	_	_
emplectite	0.1	_	_	_	_

2.2. Experimental Procedure

Immediately before each test, the ground copper mineral was removed from the desiccator and added to the 200 g mixture of quartz and gold in a jacketed glass cell of 1L capacity. The slurry was adjusted to 33.3% solid with double deionized water. Certified reagents, including ammonium hydroxide, sodium cyanide, lead nitrate and oxygen were used to condition the slurry. The cell cover has four openings that allow the insertion of electrodes, an agitator and a tube for adding air. The oxygen concentration was monitored by Oxi 340 oxygen meter and controlled by injecting air and oxygen into the slurry at various ratios. The temperature was controlled at 21 °C by circulating a mixture of water and ethylene glycol from a heat exchanger through the cell jacket. Agitation was accomplished with 255 mm Teflon paddles powered by a variable-speed electric motor.

The Hunt leaching tests lasted between 24 and 48 h, depending on the type of minerals. Ammonia was added as concentrated ammonium hydroxide solution (28%–30%), followed by NaCN. Calcium hydroxide was used to adjust the pH to above 11. Both NaCN and NH₃ in leaching were measured, on the filtrate, with ion selective electrodes (ISE) and adjusted after 1, 2, 4, 6, 24, 48 h of leaching. The S/L separation was realized by either micro-filters or centrifuge. The solids taken during this procedure were returned to the reactor. The accuracy of ISE for both cyanide and ammonia are within ±10%. At the end of the tests, the pulps were filtered and the filter cakes were washed, dried, homogenized, sampled and analyzed for gold by fire assay in duplicates. The pregnant solutions were analyzed for CNO⁻, CNS⁻, SO₄²⁻ and Cu_{tot}. The concentration of CNO⁻ and CNS⁻ and SO₄²⁻ were determined by gradient elution HPLC (high performance liquid chromatography). The Cu contents were analyzed by ICP-AES (inductively coupled plasma atomic emission spectrometry). Because of interferences encountered by direct analysis of solution with Atomic Adsorption Spectroscopy, the liquid samples were analyzed for gold content using the fire assay.

For cyanidation tests, free cyanide was determined by titration with silver nitrate using rhodamine as an indicator. For the free cyanide concentration, the controlled value was a time weighted average obtained by the summation of the average content values (C_i) within the sampling interval multiplied by the length of sampling interval (t_i) divided by the total time (t), *i.e.*, the free cyanide concentration = $(\sum (C_i \times t_i)/t)$. The variation of the realized average free cyanide concentration relative to the target free cyanide concentration was within $\pm 10\%$ in this investigation. Free cyanide was also determined for the final leach and wash solutions. The calculation of cyanide consumption assumes 100% recycling of the leach solution.

2.3. Analysis of Leached Residue and Gold Samples by XPS

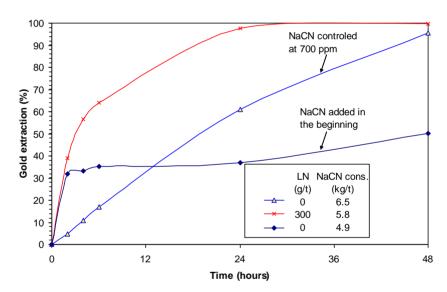
The XPS data were collected using a Kratos Axis Ultra XPS instrument equipped with monochromated Al K α radiation (1487 eV). XPS data was collected using two modes. In the standard operating mode data was collected from an irradiated area approximately 700×400 μ m. For the djurleite experiment, the XPS was operated using the imaging mode and the data was collected from individual particles using a 220 μ m diameter aperture. Survey spectra were collected with a pass energy of 160 eV and high resolution narrow region spectra were collected with a pass energy of 20 eV. The Kratos instrument has a novel magnetic confinement charge neutralization system. For the samples that experienced a build-up of positive charge, the neutralization settings were optimized to obtain the data with the highest quality. Absolute peak positions were calibrated using the C 1s peak as 284.8 eV. Surface compositions were measured using established procedures. CasaXPS software was used to calculate surface concentrations and peak fit all the spectra. Surface concentrations were calculated using peak areas and sensitivity factors. Spectra were peak fit using 70% Lorentzian and 30% Gaussian shapes. The S 2p peaks were fit using spin orbit doublet peaks separated by 1.2 eV and the S 2p1/2 peak was constrained to half of the area of the S 2p3/2 peak.

3. Results and Discussion

3.1. Djurleite-Bearing Synthetic Gold Ore

Figure 1 illustrates the gold leaching kinetics in the presence of djurleite. With 6.1 kg/t NaCN added at the start of the experiment, the leaching curve reaches a plateau after two hours due to passivation of gold grains. The gold extraction slowly increases to a maximum of 51% in 48 h. The leaching curve has a totally different profile with the control of free cyanide at 700 ppm NaCN (linear for the first 24 h). Under these conditions, the curve is almost linear. This profile is also an indication of passivation by sulphide, but the passivation is less severe. The overall gold extraction reached 95.8% in 48 h. The addition of lead nitrate alleviated the passivation and enhanced the gold leaching kinetics. Over 95% of gold was leached within 24 h and the extraction was complete (99.7%) in 48 h. The cyanide consumption was reduced from 6.5 to 5.8 kg/t due to lead nitrate addition.

Figure 1. Effect of NaCN concentration and lead nitrate on gold extraction in presence of djurleite; 0.3% Cu, pH 11, 8 ppm DO.



The dissolution of djurleite in a cyanide solution (using chalcocite as a proxy for djurleite) may be expressed as follow:

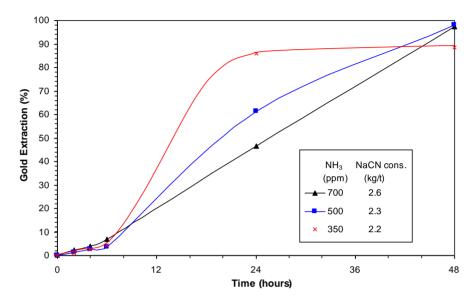
$$Cu_2S + 7CN^- + 0.5O_2 + H_2O = 2Cu(CN)_3^{2-} + CNS^- + 2OH^-$$
 (1)

An XPS analysis of the gold surface identified various compounds (Cu/Ag oxides, Cu(I)cyanide and sulphide). By adding lead nitrate, the amount of Cu(I)cyanide was reduced. In addition, the sulphide disappeared and lead oxide and hydroxide precipitated. These findings are in line with previous experimental results identifying lead compounds at the surface of gold [10]. The formation of these compounds reduced the passivation of gold.

The effect of ammonia was investigated using 350 ppm, 500 ppm and 700 ppm ammonia with 400 ppm NaCN. The leaching rate in the final six hours remained low under all conditions (Figure 2). The fastest leaching rate was achieved with 350 ppm ammonia. However, the passivation of gold occurred after 24 h to level the extraction of gold at 88.5%. High concentration of ammonia is detrimental for gold extraction after 24 h. With 500 ppm and 700 ppm NH₃, the gold extraction

approached completion (>99%) within 48 hours. The associated cyanide consumptions were 2.3 and 2.6 kg/t NaCN. The copper concentration did not vary much and remained in the range of 115 ppm. The results demonstrate that ammonia concentration is critical for producing a high overall gold extraction.

Figure 2. Effect of NaCN:NH₃ ratio on gold leaching in presence of djurleite; 0.3% Cu, 400 ppm NaCN, pH 11.5–11.8, 8 ppm DO.



XPS analysis of leached djurleite samples indicated a significant amount of CuS and trace amount of Cu(II) on the mineral surface for the experiment using 350 ppm ammonia while, on the djurleite surface leached with 700 ppm ammonia, CuS was not significant, instead Cu(II) was found possibly as $Cu(OH)_2$. The formation of CuS, in the 350 ppm ammonia experiment, indicates the formation of a metal deficient sulphide at the mineral surface and it appears that it has a chemical composition that is similar to covellite, $(Cu_3)^+(S_2)^{2-}(S^-)$ (Figure 3, [11–13]). Some Cu(I)sulphide, two N species (possibly ammonia and cyanide or their complexes) and polymerized sulphur were also found on the gold surfaces of the two leaching systems. Because of the similarities in the XPS spectra, it was not possible to identify the nature of the compounds that modified the leaching kinetics.

Oxygen oxidizes copper(I) to copper(II), which forms a copper amine in the presence of ammonia. Copper amine plays an important role in the oxidation of gold [9]. Meanwhile, the oxygen level may affect mineral oxidation and precipitation of copper. Therefore, tests were carried out at DO levels of 8, 15 and 20 ppm to determine the effect of dissolved oxygen concentration. Figure 4 shows that the gold extraction increased from 88.5% with a DO of 8 ppm to 99.7% when the DO increased to 15 ppm or above. The change in oxygen concentrations, however, has no significant effects on the solution compositions.

The effect of NaCN concentration was investigated by changing the concentration of NaCN from 200 to 350 ppm (Figure 5). The consumption of cyanide was reduced from 2.4 kg/t at 350 ppm NaCN and 350 ppm ammonia to 1.2 kg/t at 200 ppm NaCN and 100 ppm ammonia. The gold leaching kinetics decreased with the decrease in cyanide concentration but the overall gold extraction still reached 99.7% in 48 h. The ammonia concentration has to be adjusted in tandem with the cyanide concentration to target the right ammonia/cyanide ratio. Surprisingly, the dissolved copper increased

from about 130 ppm to about 390 ppm when the cyanide concentration was 250 ppm or lower. The increase of copper cyanide species is likely to be the result of the decrease in copper hydroxide formation. Only 1.2 kg/t NaCN was required to have a complete gold dissolution using the Hunt process, compared to 5.8 kg/t for cyanidation.

Figure 3. The S2p spectra from djurleite powder reacted in (a) 350 ppm ammonia; and (b) 700 ppm ammonia.

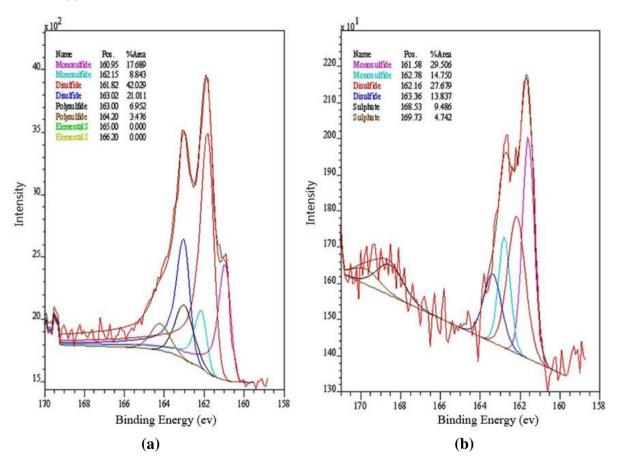


Figure 4. Effect of dissolved oxygen concentration of gold leaching in presence of djurleite; 0.3% Cu, 400 ppm NaCN, 350 ppm NH₃, pH from 11.5 to 11.8.

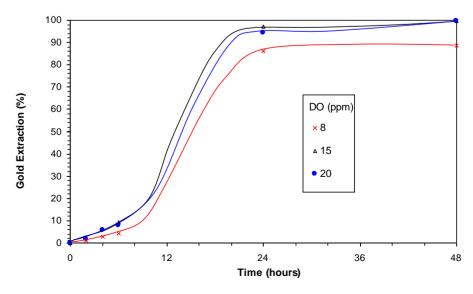
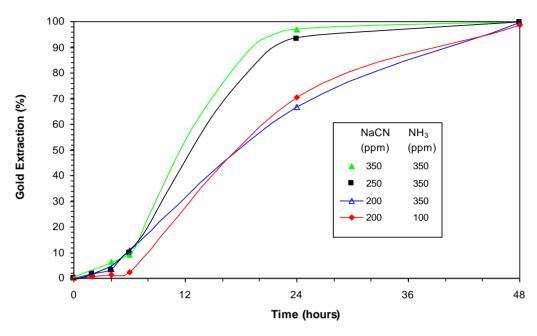


Figure 5. Effect of NaCN concentration on gold extraction in presence of djurleite; 0.3% Cu, 15 ppm DO, pH >11.



3.2. Bornite-Bearing Synthetic Gold Ore

The leaching kinetics profiles of experiments conducted at 800 ppm and 1600 ppm NaCN show that passivation of gold occurs in absence of lead nitrate as the gold extraction remains less than 99% (Figure 6). Leaching with 800 ppm NaCN and 1600 ppm NaCN produced 85.4% and 96.1% respectively. The addition of 500 g/t lead nitrate totally eliminated gold passivation and a complete extraction of gold was obtained within 6 hours even with only 500 ppm NaCN. Obviously, the dissolution of bornite undergoes a different mechanism in the presence of lead nitrate. The decrease of free cyanide concentration in leaching reduced the cyanide consumption from 6.1 kg/t to 4.7 kg/t NaCN. The concentration of dissolved copper was reduced from 1370 ppm to 1074 ppm. The dissolution of bornite in a cyanide solution may be expressed as:

$$Cu_5FeS_4 + 19CN^- + 2O_2 + 3H_2O = 5Cu(CN)_3^{2-} + 4CNS^- + FeOOH + 5OH^-$$
 (2)

Three tests were conducted with different DO, NaCN and ammonia concentrations. In terms of gold extraction, these tests failed to increase gold extraction above 80% (Figure 7). Increasing the DO level from 8 to 20 ppm did not introduce any significant enhancement of gold extraction at 350 ppm NaCN. However, the increase of NaCN from 350 ppm to 550 ppm brought the extraction up from 46.1% to 65.5% in 48 hours. The consumption of cyanide was similar to that in the straight cyanidation (2.0 kg/t at 350 ppm). At 550 ppm NaCN, the consumption increased to 3.2 kg/t. Gold dissolution at the initial stage of these Hunt tests was slower than straight cyanidation and it became faster after six hours. No significant reduction was observed on the dissolution of copper. In the straight cyanidation with 350 ppm NaCN, 560 ppm Cu was dissolved whereas, in these Hunt tests, the dissolved copper ranged from 439 to 818 ppm.

Figure 6. Effect of NaCN concentration and lead nitrate addition on gold extraction in presence of bornite; 0.3% Cu, 8 ppm DO, pH >11.

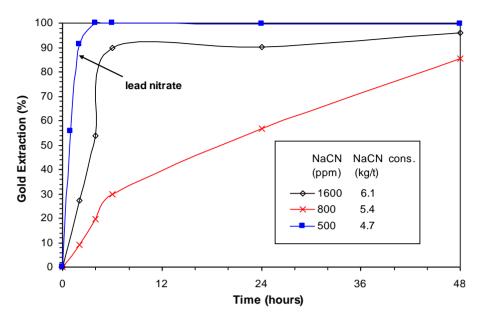
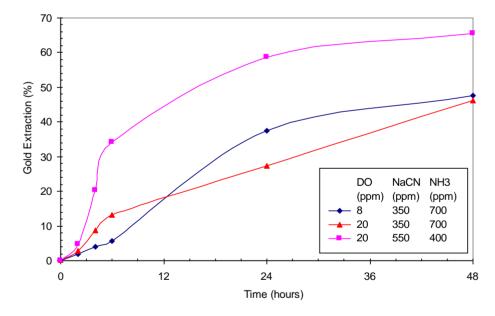
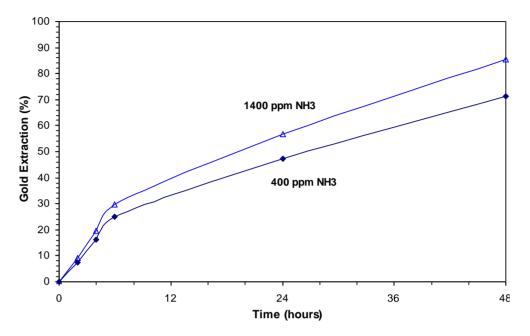


Figure 7. Hunt leaching test under typical Hunt conditions; 0.3% Cu as bornite, pH >11.



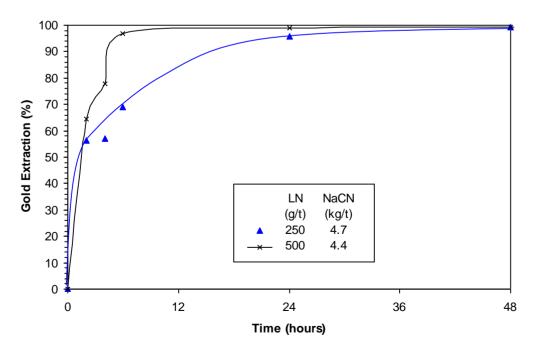
Tests were conducted at 400 ppm and 1400 ppm ammonia while maintaining the free cyanide at 850 ppm NaCN. Figure 8 shows that gold extraction increased from 71% to 88% with cyanide consumption and 7.1 and 7.6 kg/t NaCN. The results indicate that the Hunt leaching has limited efficiency for leaching gold in presence of bornite. This approach had limited effect on retarding the dissolution of copper and consequently the consumption of cyanide. The bornite consumes more cyanide than djurleite. However, due to the deficiency of Fe in bornite, the FeOOH formed is not enough to prevent further dissolution and oxidation of bornite. The high copper concentration indicates that the precipitation of copper is low. The volume of oxygen required to maintain the dissolved oxygen concentration is significantly larger for bornite than for djurleite. This is possibly due to the highly reductive nature of bornite (Equations 1 and 2).

Figure 8. Hunt leaching tests at high cyanide concentration; 0.3% Cu as bornite, 850 ppm NaCN, pH 11.7–11.9, 8 ppm DO.



It was possible to improve the efficiency of ammonia-cyanide leaching by including a 4-hour pre-leach at pH 11 with the addition of 250 g/t or 500 g/t lead nitrate (Figure 9) prior to gold leaching. The concentration of cyanide and ammonia were controlled at 550 ppm. The cyanide consumption was reduced to 4.4 kg/t NaCN. It is partly associated to a reduction of thiocyanate formed (~50% reduction).

Figure 9. Effect of lead nitrate on gold leaching in presence of bornite; 0.3% Cu, 550 ppm NaCN, 400 ppm NH₃, pH 11.5–11.8.



XPS study of the gold surface and bornite samples in the ammonia-cyanide leaching with or without lead nitrate showed several precipitated species. Gold was passivated by Cu(I) cyanide and possibly Cu(I) sulphide. With lead nitrate, the copper deposit on the surface was largely reduced. Instead, lead oxide and hydroxide appeared on the gold surface. Due to the decrease of copper precipitates, the gold dissolution is more active than that in the test without lead nitrate.

On the leached surface of bornite, the surface characteristics were found to be similar to djurleite. This change indicated that both copper and iron were dissolved. With lead nitrate, Cu(I) became Cu(I)sulphide and Cu(II)oxide and hydroxide at the surface of bornite. No Cu-cyanide species were found. The metal deficient sulphide surface produced thiosulphate and polysulphide. Analysis of the leach solution showed the presence of sulphate and cyanate were not significantly modified by adding lead nitrate (32 to 54 ppm for sulphate and 7 to 19 ppm for cyanate). The effect of lead was to reduce the concentration of sulphide and copper on the gold surface. Lead also reported on gold as an oxide or hydroxide. Such a change explains the improved gold extraction rate.

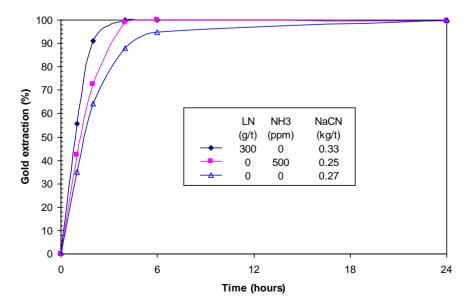
3.3. Chalcopyrite-Bearing Synthetic Gold Ore

In the presence of chalcopyrite, the gold extraction in a cyanidation experiment with lead nitrate was 99.7% in four hours with 0.33 kg/t of cyanide consumed (Figure 10). In absence of lead nitrate, the leaching kinetics were lower and the maximum extraction of gold was reached in 24 h. At the same concentration, the reactivity of this pure chalcopyrite is lower than the one contained in Mouska ore [14]. However, it is comparable to the reactivity of chalcopyrite contained in Geant Dormant ore [15]. The addition of 500 ppm ammonia improved gold leaching kinetics and overall extraction very close to the conventional cyanidation. The consumption of cyanide was slightly lower with ammonia (0.25 kg/t vs. 0.33 kg/t NaCN).

The dissolution of chalcopyrite in a cyanide solution may be expresses as follow:

$$CuFeS_2 + 5CN^- + O_2 + H_2O = Cu(CN)_3^{2-} + 2CNS^- + FeOOH + OH^-$$
(3)

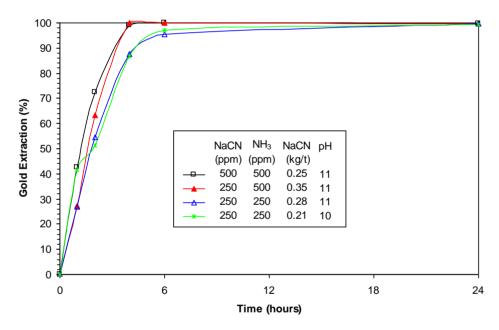
Figure 10. Effect of lead nitrate and ammonia on gold extraction in presence of chalcopyrite; 0.3% Cu, 500 ppm NaCN, 8 ppm DO.



The XPS analysis indicated that the chalcopyrite powder was partially oxidized. The chalcopyrite surface contains Cu(I), an Fe(III) species, an Fe(II)–S species, and a small amount of Fe in chalcopyrite. The sulphur data shows sulphur in chalcopyrite, along with sulphur in disulphide, polysulphide, and sulphate species. The oxygen data shows that oxide and hydroxide species are present. Some pristine chalcopyrite is detected and this suggests that the oxidized material coating particles is either very thin, or it may also occur as islands or some combination of both. This may explain the lower reactivity of chalcopyrite with cyanide.

Figure 11 shows that using lower cyanide and ammonia did not compromise gold extraction, which remained >99%. The gold leaching kinetics slightly decreased with the reduction of reagents concentration. The lowest consumption of cyanide recorded is 0.21 kg/t NaCN. Comparison of actual leaching results with prior work on high purity chalcopyrite shows similarities in terms of low dissolution of copper [16].

Figure 11. Effect of cyanide and ammonia on gold extraction in presence of chalcopyrite; 0.3% Cu, 8 ppm DO.



4. Conclusions

Efficient cyanidation of gold in the presence of djurleite and bornite had to be conducted with the addition of lead nitrate to avoid passivation of gold and excessive addition of cyanide. The ammonia-cyanide leaching of the slurry with djurleite proved to be more effective than cyanidation; producing a high gold extraction (99%) while reducing the cyanide consumption from 5.8 to 1.2 kg/t NaCN. The lower cyanide consumption was associated to a significant reduction of copper dissolved. XPS surface analysis showed that the formation of Cu(OH)₂ on djurleite grains inhibited its reaction with cyanide as well as minimizing the formation of sulphide on gold. Lead nitrate had to be added to the Hunt process to produce an efficient leaching of gold in presence of bornite. The cyanide consumption (~4.4 kg/t NaCN) was not reduced by the addition of ammonia. Additional work is required to find out if the Hunt process performs better than cyanidation. Cyanidation of chalcopyrite showed a lower consumption of cyanide at 0.33 kg/t NaCN. The addition of ammonia reduced the

cyanide consumption to 0.21 kg/t NaCN. No significant interferences were observed in gold leaching with a slurry containing chalcopyrite. Cyanidation and Hunt process demonstrated comparable leaching efficiency.

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