



# Mechanism of enargite pressure leaching in the presence of pyrite

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## ABSTRACT

Hydrometallurgy is a promising alternative to treat copper concentrates that contain large amounts of enargite ( $\text{Cu}_3\text{AsS}_4$ ). However, leaching of enargite is difficult due to its slow dissolution rate in acidic solutions. The pressure leaching of an enargite concentrate containing about 40% pyrite was carried out in sulfuric acid/oxygen at temperatures in the range of 160 to 200 °C and oxygen partial pressure of 345 to 1034 kPa. The results showed that the dissolution of enargite in the presence of pyrite was considerably faster than the dissolution of pure enargite. By leaching an enargite–pyrite concentrate sample of particle size  $-75 + 53 \mu\text{m}$  at 200 °C and 689 kPa  $\text{O}_2$ , all the copper was extracted in 15 min. It was also found that temperature, oxygen partial pressure and particle size had a significant influence on the rate of enargite dissolution from the enargite–pyrite concentrate, while the sulfuric acid concentration had little effect. Ferric ions produced in the leaching were responsible for the enhanced dissolution of enargite in the presence of pyrite and pure enargite leaching was similarly enhanced by the addition of ferrous ions. Analysis of the data for the enargite–pyrite concentrate showed linear kinetics for the dissolution of pyrite, whilst the rate of dissolution of enargite increased substantially with time as ferric ions build up in solution.

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## 1. Introduction

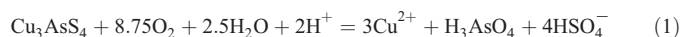
Arsenic, usually in the form of enargite ( $\text{Cu}_3\text{AsS}_4$ ), is present in most Chilean copper deposits. In the froth flotation process used for concentrating sulfide copper ores, most of the enargite reports to the final copper concentrate. The increasing presence of enargite in the copper concentrates is starting to complicate the traditional pyrometallurgical treatment of the sulfide minerals. In addition, there are large copper ore resources in Chile, where enargite is not an impurity but one of the main copper minerals. Enargite rich copper concentrates cannot be treated by conventional smelting/converting technology because of the environmental risk of arsenic emissions to the atmosphere and because of a deteriorated quality of the final copper product. Therefore, nonconventional hydrometallurgical methods such as pressure leaching must be sought for the processing of these arsenic contaminated concentrates.

Previous investigators have found that enargite is a refractory mineral in aqueous media, especially in acidic solutions. The leaching rate of enargite in sulfate media at atmospheric pressure is very slow using either oxygen or ferric ions as oxidants. On this matter, Dutrizac and MacDonald (1972) leached discs of synthetic and natural enargite in acidified sulfate solutions under atmospheric conditions. They found that the rate of dissolution of enargite was extremely slow and the dissolution kinetics was linear in all cases. The activation energy associated with the dissolution of the synthetic sample was 13.3 kcal/mol, in the temperature

range of 60 to 95 °C. They also found that pure natural enargite samples dissolved at approximately the same rate as the synthetic enargite. Flynn and Carnahan (1989) found that silver or mercury sulfate salts could be used to catalyze the ferric sulfate leaching of enargite at atmospheric pressure. They found that by adding 0.25 g/l of silver sulfate to a leaching solution containing 0.8 mol/l  $\text{Fe}_2(\text{SO}_4)_3$  and 1 mol/l  $\text{H}_2\text{SO}_4$ , they could leach 97% of the enargite in 6 h at the boiling point of the solution. They also claimed that mercury sulfate was less effective as a catalyzer than silver sulfate.

The use of chloride ions to accelerate the leaching of enargite has also been studied. Padilla et al. (2005) leached natural enargite particles in  $\text{H}_2\text{SO}_4$ –NaCl solutions using oxygen as oxidant at atmospheric pressure. These investigators found that the leaching rate was also slow in this system, although it was considerably faster than in the absence of chloride ions. In a 2 M NaCl and 0.25 M  $\text{H}_2\text{SO}_4$  solution, about 7% of the enargite was dissolved in 7 h leaching at 100 °C.

On the other hand, pressure leaching has been found to be a more effective method for the dissolution of enargite in acid media, although the leaching rate is still slow as compared to other copper sulfides, including chalcopyrite. Padilla et al. (2008) studied the kinetics of pressure dissolution of enargite in sulfuric acid oxygen media in the temperature range 160 to 220 °C and oxygen partial pressures of 303 to 1013 kPa. They determined that enargite dissolution occurred with total oxidation of the sulfide sulfur according to the following reaction:



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These investigators also found that enargite leached with linear kinetics and that the dissolution rate increased substantially with increasing temperature. Complete dissolution of enargite with particle size  $-75 + 53 \mu\text{m}$  was obtained in 120 min at 220 °C and 689 kPa  $\text{O}_2$  partial pressure. Riveros and Dutrizac (2008) also investigated the pressure leaching of enargite with ferric sulfate–sulfuric acid solutions in the temperature range of 130 to 180 °C without oxygen addition, and with an overpressure of oxygen of 100 psi. They found that in the latter case the rates were relatively faster than those obtained with only ferric sulfate. They also reported that in both cases the dissolution of copper was incomplete at temperatures below 170 °C because of the formation of coatings of elemental sulfur; while at higher temperatures the sulfide sulfur oxidized to sulfate and complete dissolution of copper was obtained.

Rivera-Vásquez and Dixon (2009) studied the dissolution of enargite mixed with various amounts of pyrite at atmospheric pressure and 80 °C, using a solution containing sulfuric acid, ferric and ferrous ions with a redox potential of 435 mV vs. SCE which was controlled with oxygen gas. They reported enhanced dissolution of enargite in the presence of pyrite which they attributed to the formation of a galvanic couple between enargite and pyrite – similar to the well established interaction that occur between chalcopyrite and pyrite (Berry et al., 1978; Murr, 1980; Mehta and Murr, 1982; Metha and Murr, 1983; Dixon et al., 2008) with pyrite providing the cathodic surface for the anodic dissolution of enargite.

Nadkarni and Kusik (1988) reported that the addition of pyrite also increased the rate of dissolution of enargite under pressure leaching conditions. These authors mentioned a leaching process for enargite concentrates where enargite and pyrite were mixed in an approximately ratio 10/1 and leached at a temperature of about 225 °C and 150 psi  $\text{O}_2$  partial pressure. The copper dissolution was about 98% from this mixture as compared to less than 70% in the absence of pyrite. Unfortunately, the leaching time was not given.

Considering that pyrite is a common impurity in copper–arsenic concentrates, the effect of pyrite on enargite dissolution rate is of great practical interest. Therefore, the main objective of this work was to study the pressure leaching of a pyrite rich enargite concentrate in  $\text{H}_2\text{SO}_4\text{--O}_2$  and to compare the results with the pressure leaching of a pure enargite concentrate in order to elucidate the reason for the rate enhancement observed.

## 2. Experimental

### 2.1. Materials

The primary material used in the experimental work was an enargite concentrate with a high content of pyrite, which was prepared from large particles (about 11" in size) with enargite and pyrite mineralization from El Indio Mine (Barrick Corp.). This material was crushed manually to a size of approximately 5 mm, and a pre-concentrate was obtained by hand sorting the sulfide rich particles. This pre-concentrate was ground in an agate mortar to a size smaller than 150  $\mu\text{m}$ , and the particles smaller than 38  $\mu\text{m}$  were removed by sieving. The material  $-150 + 38 \mu\text{m}$ , was then concentrated by flotation followed by magnetic separation, to obtain a final pyrite rich enargite concentrate. This enargite–pyrite concentrate was classified into size fractions using USA sieve series with apertures: 106, 75, 53 and 38  $\mu\text{m}$ . The chemical analysis of the size fractions is shown in Table 1. The mineralogical components of various size fractions was also determined using a SEM-based automated mineralogical analysis systems (QEMSCAN), and the results are presented in Table 2. As can be seen in the table, the enargite and pyrite content of the various size fractions do not change significantly.

A second essentially pure enargite concentrate was prepared from large enargite crystals also from El Indio Mine. This material was crushed and ground by the same procedure described earlier, and

**Table 1**  
Chemical analysis of the enargite–pyrite concentrate.

Size fraction	Cu, %	As, %	Fe, %	S, %
$-150 + 106 \mu\text{m}$	26.5	10.1	19.0	39.7
$-106 + 75 \mu\text{m}$	27.6	10.5	18.6	40.1
$-75 + 53 \mu\text{m}$	27.2	10.9	17.1	39.0
$-53 + 38 \mu\text{m}$	29.4	11.3	17.7	39.2

washed with distilled water to eliminate any soluble copper species present. The chemical and mineralogical analysis of the  $-75 + 53 \mu\text{m}$  size fraction of this material, which was used in this research, showed 44.4% Cu, 18.4% As, 34% S and 0.2% Fe. The mineralogical analysis indicated 96.2% enargite, and the main impurities were gangue minerals. The pyrite content was 0.4%.

### 2.2. Apparatus and procedure

Pressure leaching experiments were conducted in a batch 2-liter capacity titanium autoclave reactor, which comprises, a variable speed mechanical agitation system, an electric heating mantle and a cooling coil to circulate cold water through the reaction chamber. The autoclave was also equipped with a PID temperature controller, a gas system to provide oxygen overpressure and a sampling device to extract samples of the leaching solution.

The experimental procedure consisted in preheating 1 l of leaching solution to about 85 °C and then to add 2 g of the solid sample. Then, the autoclave was sealed and heated to the temperature specified for the test. Once at the set temperature, oxygen was introduced, and the system was allowed to react at a constant total pressure. At the end of the experiment, a sample of the leach solution was taken and the autoclave was rapidly cooled to near ambient temperature by circulating cold water through the cooling coil. After filtering the pulp, the solids were washed with acidified water and dried at 65 °C. The liquid samples were analyzed for copper and, in some cases, for arsenic and iron by atomic absorption spectroscopy. Since, according to the mineral composition presented in Table 2, enargite was the dominant copper containing mineral in the concentrate, the fraction of copper in solution gives an accurate value of the fraction of enargite dissolved.

## 3. Results and discussion

Most of the experiments were carried out with the enargite–pyrite concentrate, to study the main variables that could affect the pressure leaching of this material: i.e. stirring speed, concentration of sulfuric acid, particle size, oxygen partial pressure, temperature, and time. Some experiments were also carried out using the pure enargite concentrate, in order to compare the leaching behaviour of this material with the behaviour of the enargite–pyrite concentrate.

### 3.1. Effect of the stirring speed

Preliminary experiments were carried out to determine the effect of the stirring speed on copper dissolution using the enargite–pyrite concentrate. The results of experiments carried out in the range 400 to

**Table 2**  
Mineralogical analysis of the enargite–pyrite concentrate.

Size fraction	Enargite, %	Pyrite, %	Chalcopyrite, %	Covellite, %	Gangue, %	Others
$-106 + 75 \mu\text{m}$	57.6	39.1	1.0	0.3	1.8	0.2
$-75 + 53 \mu\text{m}$	56.3	39.7	0.4	2.1	1.4	0.1
$-53 + 38 \mu\text{m}$	58.9	36.7	0.9	1.9	1.3	0.3

800 rpm indicated that 700 rpm was sufficient to suspend completely the solid particles and to distribute effectively the oxygen in the leaching solution. There was no difference in the rate of copper dissolution obtained for both 700 rpm and 800 rpm agitation at 170 °C, indicating that there were no oxygen diffusion limitations in the system. A rate of agitation of 800 rpm was used in subsequent experiments to assure independence of this variable.

### 3.2. Effect of sulfuric acid and oxygen concentration

The effect of the initial sulfuric acid concentrations on copper dissolution was studied in the range of 10 to 40 g/l of  $H_2SO_4$  at 180 °C with the other variables kept constant. The results are presented in Fig. 1, where we can see that the sulfuric acid concentration in the range studied had little effect on the rate of copper dissolution from the enargite–pyrite concentrate. This result is not unexpected since the main roles of the acid in the leaching system are to protonate the arsenic (V) and sulfur (IV) species produced by the leaching and to avoid the hydrolysis and precipitation of the ferric iron. Thus 20 g/l was the standard acid concentration in subsequent experiments.

At this point it is worth to note that the dissolution curves in Fig. 1 have a distinctive S-shape, indicating that the rate of dissolution of enargite is initially slow and increases progressively with leaching time. This shape of dissolution curve was observed in various degrees in all the experimental conditions in the pressure leaching of the enargite–pyrite concentrate.

The effect of partial pressure of oxygen on the leaching of the enargite–pyrite concentrate was studied in the range 345 to 1034 kPa at 180 °C and the results are illustrated in Fig. 2, where we can observe that an increase in  $O_2$  partial pressure produces an appreciable increase in the rate of copper dissolution. This means that the rate determining step must be dependent on the oxygen concentration in the solution, which is proportional to the oxygen partial pressure for any given condition. In addition, the characteristic S-shape of the dissolution curve is more noticeable for the lower partial pressure of oxygen of 345 kPa. This could be due to a slower rate of oxidation of the ferrous to ferric ions at low oxygen concentration, since the ferric ions in solution are believed to be the main oxidizing agent for enargite (see later discussions).

### 3.3. Effect of particle size

Leaching tests were carried out using samples of different particle sizes. The copper dissolution obtained at 180 °C and 689 kPa of

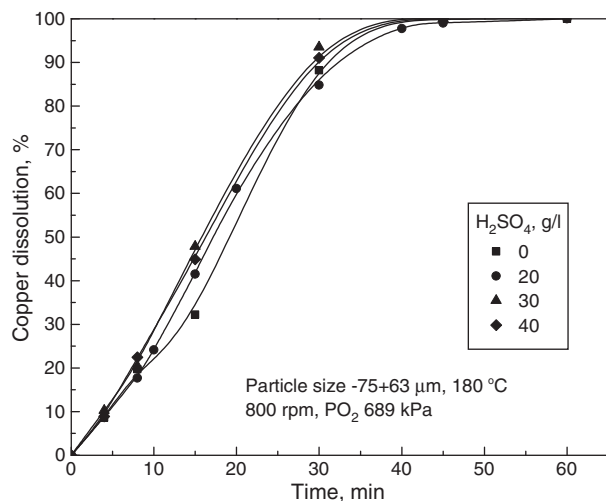


Fig. 1. Effect of sulfuric acid on the copper dissolution from enargite–pyrite concentrate for the conditions given in the figure.

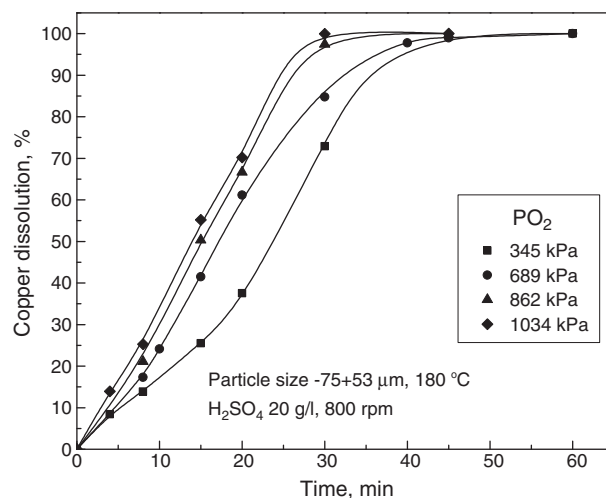


Fig. 2. Effect of oxygen partial pressure on the copper dissolution from enargite–pyrite concentrate.

oxygen partial pressure for various particle sizes are shown in Fig. 3. As seen in this figure, when the size of the solid particles increases, the leaching rate of the enargite decreases significantly. This effect could be due to the decrease in the surface area of the solid sample as the size of the particles increases; however, the slower dissolution for the larger particle sizes could also be partly due to a low amount of iron in solution because of a slower leaching of the pyrite in the coarser samples.

### 3.4. Effect of temperature

To determine the effect of temperature on the enargite leaching, various experiments were executed in the temperature range of 160 to 200 °C. Not surprisingly, Fig. 4 shows that temperature has a large effect on the rate of enargite dissolution. Moreover, the present rates of dissolution are notably faster than the rates reported by Padilla et al. (2008) on pressure dissolution of pure enargite. Nonetheless, the large effect of the temperature on the leaching rate indicates that the dissolution reaction is controlled by a chemical or electrochemical step and not a mass transfer process.

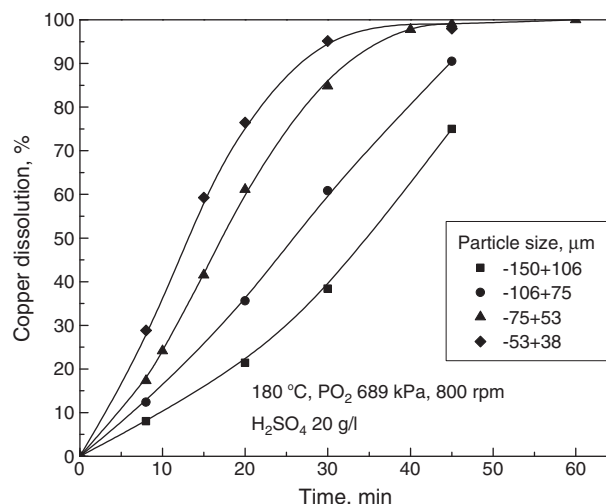


Fig. 3. Effect of particle size on the copper dissolution from enargite–pyrite concentrate.

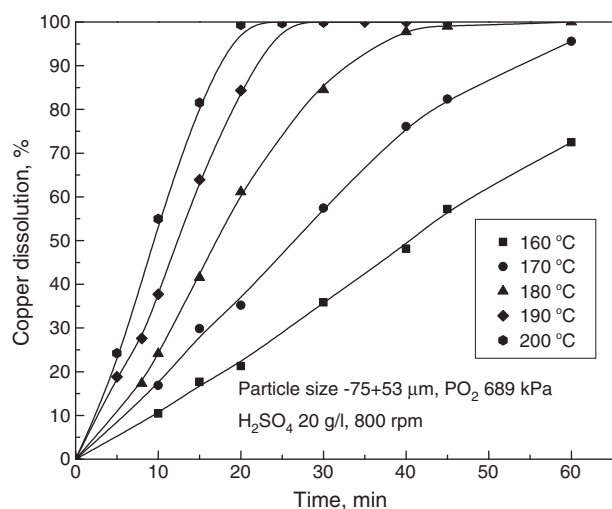


Fig. 4. Effect of temperature on the copper dissolution from enargite-pyrite concentrate.

### 3.5. Arsenic and iron dissolution

Generally, at all the temperatures the dissolution of arsenic was equal to the dissolution of copper at short leaching times as shown in Fig. 5 (within the range of experimental error), which is expected since both copper and arsenic dissolve from enargite. However, as seen in the figure, the rate of arsenic dissolution becomes progressively slower than copper dissolution with time. This is mainly attributed to some precipitation of the arsenic in the solution as an iron(III)-arsenate compound.

The dissolution of iron was also determined as a function of temperature. Since pyrite is the only iron containing mineral in the sample, the iron dissolution corresponds to the pyrite dissolution. It was found, that under all the conditions tested pyrite dissolved faster than enargite as seen clearly in Fig. 6, which compares the iron dissolution curves (continuous lines) with the copper dissolution curves (dotted lines) for experiments obtained at 160, 180 and 200 °C. But the iron content in solution also decreases at longer leaching times because of the precipitation of ferric arsenate compounds as discussed earlier.

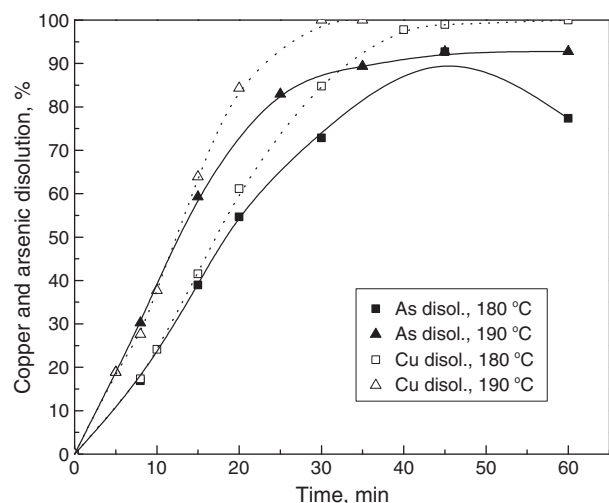


Fig. 5. Dissolution of arsenic and copper from enargite-pyrite concentrate at 180 and 190 °C.

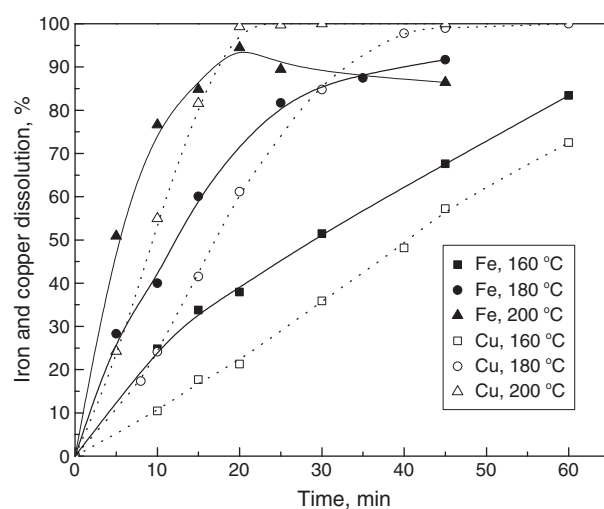


Fig. 6. Dissolution of iron and copper in the pressure leaching of enargite-pyrite concentrate.

### 4. Mechanism and kinetic considerations

The rapid pressure leaching rates observed for the enargite in the presence of pyrite in the concentrate could be explained by two alternative mechanisms:

- The formation of a galvanic couple between enargite and pyrite, with pyrite as the cathodic surface which would lead to an accelerated anodic dissolution of the enargite.
- The generation of ferric ions during the leaching which dissolved enargite faster than oxygen.

The first mechanism was proposed by Rivera-Vásquez and Dixon (2009) to explain the higher reactivity of enargite in the presence of pyrite at atmospheric pressure. However, the experimental results obtained in this research suggest that a galvanic interaction is unlikely to play a major role in the pressure leaching of the enargite-pyrite concentrate. This is supported by the fact that the pyrite dissolution from this material was found to be faster than the enargite dissolution, which is the opposite result of what one would expect if the pyrite were protected cathodically. As a matter of fact, it is well established that when there is a galvanic interaction between two sulfides, the dissolution of the anodic mineral is enhanced while the dissolution of the cathodic mineral is retarded (Murr, 1980; Paramguru, 2002; da Silva et al., 2003). The dissolution rates of pyrite from the enargite-pyrite concentrate found in this research are close to the dissolution rates of pure pyrite determined by Long and Dixon (2004) under similar experimental conditions. Thus, it is clear that pyrite dissolution was not retarded by the presence of enargite as it should do if a galvanic interaction were operative.

On the other hand, it is well known that oxygen can readily oxidize ferrous to ferric ions under pressure leaching conditions, and the oxidation rate depends on the temperature, and the oxygen and ferrous ions concentrations (Vračar and Kerović, 1997; Ruiz et al., 2009). In the present experiments, the ferric to ferrous ratio in the leaching solution should be high, especially at the higher temperatures and at high oxygen partial pressure. This fact and the result that the enargite dissolution rate increases with time when the iron in solution also increases, indicates that the ferric ions generated in the system was the main oxidizing agent for enargite dissolution.

To verify the role of the dissolved iron on the rate of enargite leaching, experiments were carried out using the pure enargite concentrate sample size -75 + 53  $\mu\text{m}$  which was leached with 20 g/l of sulfuric acid with and without additions of 0.22 g/l Fe added as ferrous sulfate. It should be pointed out that 0.22 g/l of  $\text{Fe}^{2+}$  is about 65% of the total

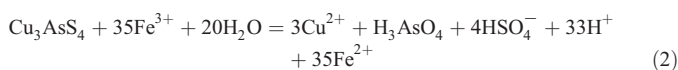


iron that could be dissolved from two grams of the enargite–pyrite concentrate. The results of these tests are shown in Fig. 7, where the results of the leaching of enargite–pyrite concentrate under the same experimental conditions are also included for comparison purposes.

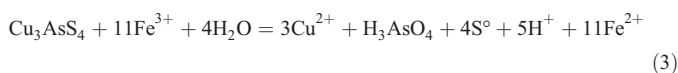
One can clearly see in Fig. 7, that the copper dissolution from the pure enargite sample is very slow as compared to the dissolution of copper from the enargite–pyrite concentrate. On the other hand, when pure enargite is leached with addition of ferrous sulfate in the solution the copper dissolution was even faster, particularly at short leaching times. This result confirms that the ferric iron is the main oxidant of enargite during the pressure leaching of enargite–pyrite concentrate.

To re-affirm this finding a second set of leaching experiments was carried out using a sample prepared by mixing pure enargite with pure pyrite (70% enargite–30% pyrite) both with particle size  $-53 + 38 \mu\text{m}$  and similar results were obtained i.e. both the addition of pyrite and ferrous sulfate accelerate the dissolution of enargite.

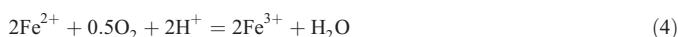
Therefore, in the pressure leaching of the enargite–pyrite concentrate the enargite dissolves by the following main reaction:



At the lower temperatures, a fraction of the sulfide sulfur could be oxidized to elemental sulfur, according to the reaction:



These reactions are coupled with the reoxidation of the ferrous ions to ferric ions by oxygen according to:



It is well known that for monosized particles reacting uniformly with a liquid phase with linear kinetics a plot of the function  $1 - (1 - X)^{1/3}$  (where X is the fraction of solid reacted) versus time at any given condition would give a straight line, and the rate of reaction could be obtained from the slope of the line. Since the function  $1 - (1 - X)^{1/3}$  accounts for the decrease in the interfacial area of the solid particles as they shrink uniformly, the slope (tangent) of the curve obtained from this type of plot at any given time will give the instantaneous rate of reaction even if the kinetics is not linear.

The experimental data on the copper dissolution from the enargite–pyrite concentrate for various temperatures was plotted as

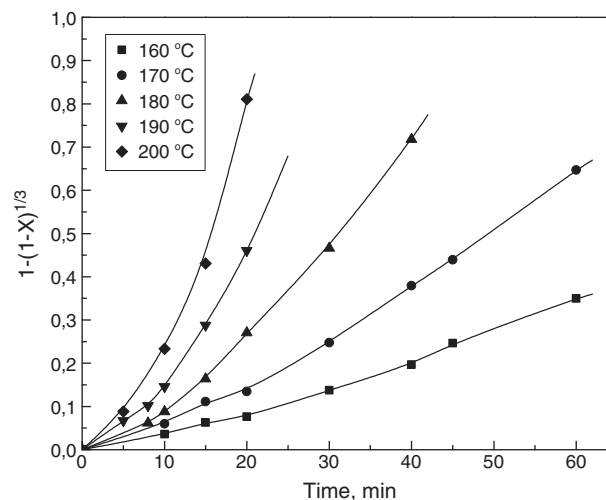


Fig. 8. Fitting of linear kinetic equation of the copper dissolution data at various temperatures for the data shown in Fig. 4.

$1 - (1 - X)^{1/3}$  versus time and presented in Fig. 8. As seen in the figure, the leaching kinetics of enargite in the presence of pyrite is not linear and the slope of the curves increases significantly with leaching time. This result is consistent with the leaching of enargite occurring by reaction (2) if the rate of this reaction were a function of the ferric ions concentration. Earlier investigators have indicated that in the leaching of enargite with ferric ions, the dissolution rate is a power function of the ferric concentration for ferric concentrations lower than 11 g/l (Dutrizac and MacDonald, 1972), therefore, it is likely that this dependence is also true on the conditions of the present research.

On the other hand, the kinetics data on iron dissolution from the enargite–pyrite concentrate (Fig. 6) fits well the linear kinetic equation. Therefore, the pressure leaching of pyrite in the presence of enargite appears to follow a linear kinetic. Finally, in Fig. 9 one can also observe good linear kinetics with the pressure leaching of pure enargite with or without the addition of ferrous ions in the leaching solution compared to the non-linear enargite–pyrite data. This result is expected because the dissolution occurs under a constant ferric ions concentration. In the case of pure enargite leached in absence of iron in solution, the kinetic is also linear, which agrees with the earlier findings reported by Padilla et al. (2008).

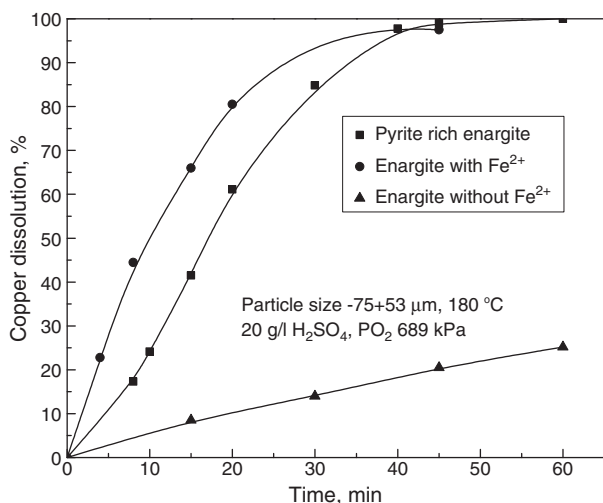


Fig. 7. Copper dissolution from pure enargite with and without 0.22 g/l  $\text{Fe}^{2+}$  addition and from enargite–pyrite concentrate.

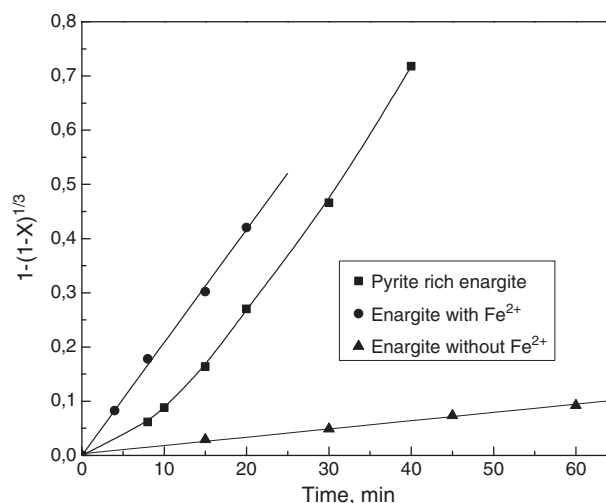


Fig. 9. Fitting of linear kinetic for the leaching of enargite–pyrite concentrate and pure enargite with and without 0.22 g/l  $\text{Fe}^{2+}$  addition for the data shown in Fig. 7.

Modeling of the leaching kinetic of enargite in the presence of pyrite for prediction purposes requires additional experimental data which is currently underway.

## 5. Conclusions

From the experimental data on the acid pressure leaching of enargite–pyrite concentrate and pure enargite sample the following can be concluded:

- The pressure leaching of enargite in the presence of 40% w/w pyrite is considerably faster than the dissolution of pure enargite and all the copper can be dissolved in 15 min at 200 °C depending upon other conditions.
- Temperature, oxygen partial pressure and particle size had significant influence on the rate of enargite dissolution from the enargite–pyrite concentrate, while the sulfuric acid concentration had little effect.
- Ferric ions generated in the leaching, by the oxidation of ferrous ions by oxygen, have been found to be the major oxidant and catalyst for enargite leaching at these high temperatures, rather than galvanic coupling with the pyrite.
- Linear kinetics of shrinking mono-sized particles was observed for the dissolution of pure enargite with or without addition of ferrous sulfate to the leaching solution. Whilst the rate of dissolution of enargite in the presence of pyrite increases substantially with time as ferric ion builds up in solution.

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