Leaching of CuFeS$_2$ by Aqueous FeCl$_3$, HCl, and NaCl: Effects of Solution Composition and Limited Oxidant
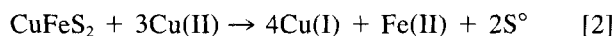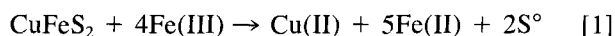
M. L. O'MALLEY and K. C. LIDDELL

Batch leaching experiments were performed in which the initial amounts of chalcopyrite and ferric chloride were selected to ensure that the oxidant was significantly depleted over the course of an experiment. Solution samples were analyzed for Cu(II) and Fe(III) by visible spectrophotometry and for total copper and total iron by atomic absorption, making it possible to measure changes in the solution component concentrations as leaching progressed. For selected samples, the solution potential was also measured. In all experiments, the Cu(II) concentration passed through a maximum and, simultaneously, the Cu(I) concentration increased very sharply. An acceleration in the total rate of leaching was normally observed at the same time. Early in a leach, the solution potential was too high for the reduction of Cu(II) to Cu(I) to take place; at the time of the increase in the overall leaching rate, however, the solution potential dropped sharply during a span of a few hours, reaching a value low enough that reduction of cupric ion became possible. The amount of Cu(I) present at the completion of a leach was dependent on the total chloride concentration of the system. The highest Cu(I)/Cu ratios were observed in systems with the highest chloride concentrations. The ultimate extent of CuFeS$_2$ leaching was dependent on the initial FeCl$_3$ and total chloride concentrations; the FeCl$_3$ was virtually completely consumed and the total chloride concentration controlled the extent to which Cu(II) was reduced by reaction with chalcopyrite.

I. INTRODUCTION

CHALCOPYRITE is both the most abundant copper sulfide and the most refractory. Much work has been done on the leaching of chalcopyrite by ferric chloride[1-13] since Sullivan recognized the value of ferric chloride as a possible lixiviant over fifty years ago. In this early work, it was found that for equal ferric concentrations, FeCl$_3$ leaching was considerably faster than Fe$_2$(SO$_4$)$_3$ leaching.[1] More recently, a number of investigators have reported the activation energy for FeCl$_3$ leaching (e.g., References 6, 8, and 10). The effect of the ferric chloride concentration on the leaching rate has been studied[9,10,13] and the role of the total chloride concentration has been discussed.[8] The influence of FeCl$_2$ on FeCl$_3$ leaching has also been considered.[5,6,13] Except for some of the work by Haver and Wong,[3,4] these later studies were conducted at constant oxidant concentrations. Haver and Wong, however, chose conditions such that the Fe(III) and Cu(II) concentrations were very low at the termination of the experiments. Simultaneously, the Cu(I) concentrations were high.

Cupric leaching systems[14-17] have not received as much attention but, as discussed below, the two systems are closely linked. The following reactions are generally accepted as representing the leaching of chalcopyrite in FeCl$_3$ and CuCl$_2$:

$$CuFeS_2 + 4Fe(III) \rightarrow Cu(II) + 5Fe(II) + 2S° \quad [1]$$

$$CuFeS_2 + 3Cu(II) \rightarrow 4Cu(I) + Fe(II) + 2S° \quad [2]$$

Cu(II), one of the products of Eq. [1], can itself leach the mineral.

Further complicating this situation is the redox reaction between aqueous copper and iron

$$Fe^{3+} + Cu^+ = Fe^{2+} + Cu^{2+} \quad [3]$$
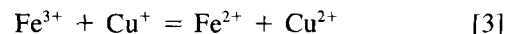
Because of this reaction, the concentrations of ferric and cupric ion cannot be varied independently. In Eq. [3], Fe^{2+} and Cu^{2+} are strongly favored thermodynamically.[18] The ability of each of the metal valences to complex with chloride ion must also be considered. Univalent copper complexes very strongly with chloride; complexation by divalent copper and by di- and trivalent iron is less pronounced. One effect of the simultaneous redox and complexation equilibria is that the Cu(I)/Cu(II) ratio is strongly dependent on the chloride concentration.[18] High Cu(I) concentrations are possible only in solutions with high ligand concentrations and are favored by low concentrations of Fe(III).

To investigate in detail the formation of Cu(I) during FeCl$_3$ leaching of chalcopyrite, batch leaching experiments were undertaken in which the reaction was allowed to proceed until there were no further concentration changes. The initial oxidant (Fe(III) in this work) was the limiting reagent. A method was developed to analyze the leach liquor for Cu(II) and Fe(III);[20] total Fe and total Cu concentrations were also determined. In selected experiments, the potential of solution samples was also determined voltammetrically using a gold electrode. In addition, concentrations of individual solution species were calculated using an equilibrium speciation model.[18] Using this combination of techniques, changes in the solution chemistry were followed in detail throughout each experiment.

The naming of complexes in this paper follows the Stock system;[19] e.g., divalent copper is designated by Cu(II) and is comprised of the species Cu^{2+}, CuCl$^+$, CuCl$_2°$, CuCl$_3^-$, and CuCl$_4^{2-}$.

M. L. O'MALLEY, Graduate Student, and K. C. LIDDELL, Associate Professor, are with the Department of Chemical Engineering, Washington State University, Pullman, WA 99164-2710.

II. METHOD

The effects of the initial FeCl₃, HCl, and NaCl concentrations, temperature, particle size, and rotation speed were investigated.

All chemicals used were of reagent grade. Deionized water was used to prepare all solutions.

The mineral samples were Transvaal chalcopyrite of +99 pct purity (Wards Natural Science Establishment, Inc.). Examination of samples by powder X-ray diffraction and electron microprobe did not reveal any other mineral phases. A wet chemical analysis was also done. Within experimental error, the mineral contained Cu:Fe:S in 1:1:2 molar ratios. The chalcopyrite was hand-picked to remove obvious impurities (quartz), then ground. The ground material was dry sieved, washed with acetone, and wet sieved. The acetone was allowed to evaporate and the sieving and washing were repeated. Samples were kept in a desiccator.

The reactor was a glass vessel with a Teflon agitator and ports for addition of solids, removal of solution samples, and purging with a nonreactive gas. This was suspended in an oil bath which had a temperature control good to ±0.5 °C.

To prepare for an experiment, 4 l of solution containing FeCl₃, HCl, and NaCl were brought to temperature in the reactor. As the solution was stirred, the system was thoroughly purged with nitrogen; a sample of CuFeS₂ was then quickly added. Throughout each leach, the reactor was purged continuously.

To investigate the possibility of nonoxidative leaching of chalcopyrite by HCl and NaCl, experiments were also done in which CuFeS₂ was pretreated in solutions containing only HCl and NaCl; after at least a 12-hour period, solid FeCl₃ was added to the reactor in an amount sufficient to make the solution 0.1 mol/dm³ in FeCl₃. In every other respect, the procedure for these experiments was identical to that followed in the others.

Samples of the liquor were taken at intervals and stored under nitrogen. Cessation of leaching could be detected visually by observing the disappearance of color from the solution; leaching was continued for several hours and additional samples were analyzed to confirm that reaction had stopped. In replicated experiments, the errors in the ultimate concentrations and the time until leaching ceased were ±10 pct.

Following leaching, samples of the solid were examined by scanning electron microscopy. The only solid phases detected were chalcopyrite and elemental sulfur. There was no evidence of significant pitting or preferential leaching along grain boundaries.

Preliminary experiments indicated that above 500 rpm initial leaching rates and total reaction times were independent of agitation speeds. Subsequent experiments were therefore made at 550 or 600 rpm. Experiments with different particle sizes (between 38 and 75 μm) and different solid loadings (24 or 48 g) indicated that the initial rate of change of the total Cu concentration is directly proportional to the total particle surface area.

The speciation model of Kimura et al.[18] was modified to include the Cu(II)-Cl complex formation constants of Ramette and Fan[21] and used to calculate the speciation of most solution samples. Concentrations of 24 species were calculated. The model requires the input of total iron, total copper, total chloride, acid, and salt concentrations. The acid, salt, and total chloride concentrations are set by the initial conditions of each leach, but large changes occur in both the total copper and total iron concentration during leaching. The measured total copper concentration was used directly as input and the total iron concentration was found by assuming equimolar amounts of copper and iron were leached. Comparison of measured and calculated Cu(II) and Fe(III) concentrations showed very good agreement up to the detection limits of the analytical procedure.[20]

III. RESULTS AND DISCUSSION

The concentration changes occurring with time in a typical leach show several features that are uncommon in mineral reactions. In Figure 1 the Fe(III) concentration fell steadily and dropped to unmeasurably low levels after 33 hours, indicating nearly complete reduction of the FeCl₃ lixiviant. The Fe(II) and total iron concentrations rose, reaching maximum values at the end of the experiment.

The behavior of copper, however, was qualitatively different; the Cu, Cu(I), and Cu(II) concentrations are plotted in Figure 2. The experiment may be considered to have three stages. In the first, there was essentially no univalent copper in the liquor: The Cu concentration increased steadily, though the slope of the concentration vs time plot slowly decreased; Fe(III) was the major oxidant present. During the second stage, there was a dramatic increase in the Cu(I) concentration. Concurrently, the Cu(II) concentration declined. The overall rate of leaching increased markedly, as shown by the increase in the slope of the total Cu concentration vs time curve between 25 and 30 hours. Concentrations of Fe(III) and Cu(II) were roughly equal during this stage. In the third and final stage, the overall reaction process stopped. The Fe(III) concentration had declined to undetectably low levels, and the Cu(II) concentration, while still measurable in this experiment, also had decreased considerably. The distribution of the species in each oxidation state was essentially constant throughout a leach, although the relative amounts of the various valences
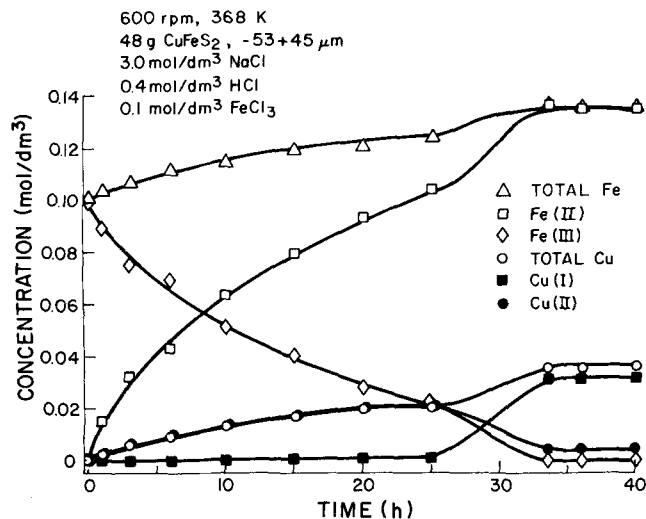

600 rpm, 368 K
48 g CuFeS₂ , -53+45 μm
3.0 mol/dm³ NaCl
0.4 mol/dm³ HCl
0.1 mol/dm³ FeCl₃

△ TOTAL Fe
□ Fe (II)
◇ Fe (III)
○ TOTAL Cu
■ Cu (I)
● Cu (II)

Fig. 1 — Changes in concentrations of Cu, Cu(I), Cu(II), Fe, Fe(II), and Fe(III) during batch leaching. Initial conditions as shown.
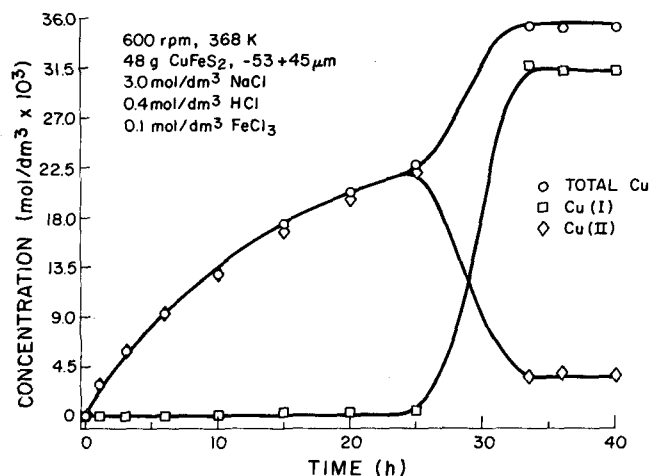
Fig. 2—Changes in concentrations of Cu, Cu(I), and Cu(II) for the same experiment shown in Fig. 1. Vertical scale expanded to show better the shapes of the curves.

Table I. Effect of Total Chloride Concentration on the Ultimate Cu(I)/Total Cu Ratio*

Concentration (mol/dm³)				
FeCl₃	HCl	NaCl	Total Cl	Cu(I)/Cu
0.1	0.4	0	0.7	0.133
0.1	0.4	0.4	1.1	0.456
0.1	0.4	1.5	2.2	0.668
0.1	0.4	3.0	3.7	0.900
0.1	0.4	4.5	5.2	0.890
0.1	0.4	4.5	5.2	0.920
0.1	1.5	0.4	2.2	0.546
0.1	3.0	0.4	3.7	0.911
0.1	4.5	0.4	5.2	0.920

*All experiments performed at 368 K, with 48 g of $-53 + 45$ μm CuFeS₂ in 4 l of solution.

changed greatly. Solution speciation effects therefore do not account for the abrupt change in the Cu(I)/Cu(II) ratio.

Because leaching by cupric chloride is faster than leaching by equal concentrations of ferric chloride, the buildup in the Cu(II) concentration is difficult to explain. A rapidly reacting intermediate would ordinarily be expected to be so quickly consumed that it does not build up in solution. Voltammetric measurements indicate that a change in potential is the likeliest explanation for the observed delay in the reaction of Cu(II). With initial concentrations of 0.1 mol/dm³ FeCl₃, 1.5 mol/dm³ HCl, and 0.4 mol/dm³ NaCl, for example, the solution potential at the beginning of leaching was 0.727 V (vs SHE) at ambient conditions. Over a period of 27 hours the potential decreased to 0.637 V. While Fe(III) can be reduced to Fe(II) at these potentials, Cu(II) cannot be reduced to Cu(I). The slow downward shift in potential speeded up significantly between 27 and 41 hours, when the potential dropped by an additional 0.175 V. During this stage of leaching, the lowered potential made reduction of Cu(II) possible. As a result, Cu(II) rapidly reacted with CuFeS₂, the overall rate of leaching increased, and the Cu(I)/Cu(II) ratio went up sharply. Previously, Jones and Peters[5] had investigated the mixed potential of CuFeS₂ electrodes in solutions of ferric and ferrous chloride; it is difficult to relate their work to the results of this study, however, because of the differences in solution compositions and in the properties and preparation of the mineral.

The leaching system apparently did reach heterogeneous equilibrium. A small amount of fresh mineral was occasionally added to the reactor at the end of a leach. No changes in solution concentrations were observed after such an addition, even in cases where detectable Cu(II) was present.

As indicated by the data in Table I and Figure 3, the total chloride concentration has a very strong influence on the Cu(I)/Cu ratio obtained at the cessation of leaching; this ratio, however, is not related to either the initial rate or the total leaching time. The ultimate Cu(I)/Cu ratio was only 0.133 in a system with 0.7 mol/dm³ total chloride but was around 0.9 in systems with 3.7 or 5.2 mol/dm³ chloride. At intermediate values of the total chloride concentration, there may be some difference in the effects of HCl and NaCl.
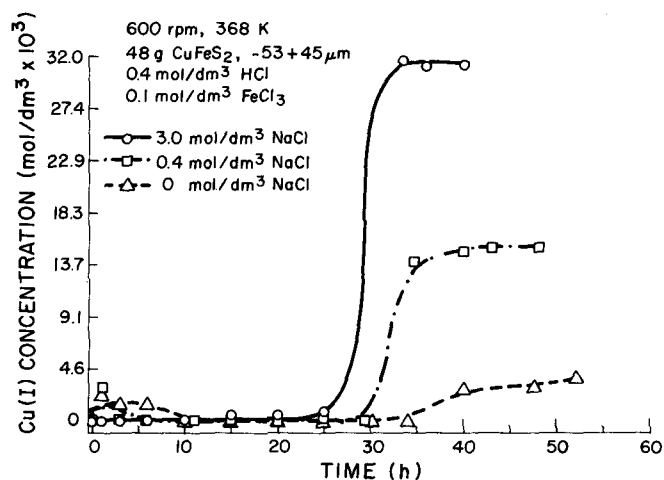

Fig. 3—Effect of total chloride on the Cu(I) concentration between 0.0 and 3.0 mol/dm³ NaCl. All curves 0.4 mol/dm³ HCl, 0.1 mol/dm³ FeCl₃, 368 K, 48 g of $-53 + 45$ μm CuFeS₂.

With FeCl₃, HCl, and NaCl concentrations of 0.1, 0.4, and 1.5 mol/dm³, respectively, the ultimate Cu(I)/Cu ratio was 0.668; the ratio was 0.546 in an experiment with 0.1, 1.5, and 0.4 mol/dm³ FeCl₃, HCl, and NaCl. The species distribution did not differ significantly among experiments containing comparable total chloride concentrations and therefore cannot explain the differences observed between leaching in media high in HCl and those high in NaCl (see below).

The total amount of copper leached was also found to depend quite strongly on the total chloride concentration. In every experiment, the Fe(III) was virtually completely consumed. Although the extent of leaching by the Cu(II) produced by Fe(III) leaching and the ultimate Cu(I) concentration depend on the total chloride concentration, there is apparently no correlation with the concentration of any particular chloride compound (HCl, NaCl, or FeCl₃) or with the concentration of the free chloride ion (Cl⁻). Up to about 3 mol/dm³ total chloride, leaching by Cu(II) increased with the ligand concentration; above 3 mol/dm³ chloride, leaching by divalent copper was independent of the chloride concentration and stopped when about 90 pct of the Cu(II) had been reduced.

Table II. Activation Energies for Leaching of Natural CuFeS₂ by FeCl₃

Concentration (mol/dm³)			Temperatures	E_a	
FeCl₃	NaCl	HCl	(K)	(kJ/mol)	Source
0.1	0	0.3	303 to 373	42 ± 4	7
0.1	0	0.3	328 to 368	66 ± 27	11
0.1	3.0	1.0	356 to 369	83	9
0.2	0	0.2	348 to 369	62	9
0.1	3.0	0.4	338 to 368	68.1 ± 13.2	this work

With initial leaching conditions of 3.0 mol/dm³ NaCl, 0.4 mol/dm³ HCl, 0.1 mol/dm³ FeCl₃, 600 rpm, and 48 gm of −45 + 38 μm chalcopyrite in 4 1 of solution, the initial rates were found between 338 and 368 K. The activation energy was 68.1 ± 13.2 kJ/mol. Table II presents some previously-reported values, several of which are in very good agreement with this work. All of the values tabulated are high enough to be considered in the reaction controlling regime. Table III lists various leach conditions studied along with the initial rates (at $t = 0$) for these runs, corrected for surface area by an assumed sphericity of 0.655.[22]

Rate data were analyzed statistically to determine how the rate depends on the various concentrations during the initial stage of leaching. Results of this statistical procedure are considered to be valid for the period during which 10 to 15 pct of the mineral is leached; particle size changes are negligible over this interval. The best fit rate expression for the first 10 to 15 pct of CuFeS₂ leaching was found to be

$$\text{rate} = \frac{k_1 C_{\text{FeCl}_3}^{0.40} C_{\text{HCl}}^{0.23}}{1 + k_2 C_{\text{FeCl}_3}} \quad [4]$$

For temperatures between 75 and 95 °C, and 0.05 to 3.0 mol/dm³ FeCl₃, 0.4 to 4.5 mol/dm³ HCl, and 0 to 4.5 mol/dm³ NaCl, the constants k_1 and k_2 are 2.13 × 10⁴ and 331; the rate is in mol Cu/cm²-s. Details of the statisti-

Table III. Initial Leaching Rates at 368 K, with Various Lixiviant Concentrations

Concentration (mol/dm³)			Initial Rate (mol Cu/cm²-s, × 10¹⁰)	Initial Rate Constant × 10⁹
FeCl₃	HCl	NaCl		
0.05	0.4	3.0	4.14	1.69
0.1	0.4	0	5.93	1.84
0.1	0.4	0.4	6.37	1.98
0.1	0.4	1.5	2.57	0.797
0.1	0.4	3.0	6.30	1.95
0.1	0.4	4.5	5.26	1.63
0.1	1.5	0.4	12.4	2.84
0.1	3.0	0.4	10.2	1.99
0.1	4.5	0.4	26.1	4.64
0.14	0.4	3.0	5.77	1.56
0.5	0.4	0	5.53	0.901
1.0	0.4	0	7.01	0.865
3.0	0.4	0	11.3	0.899

*Units are $\dfrac{\text{mol Cu/cm}^2\text{-s}}{(\text{mol FeCl}_3/\text{dm}^3)^{0.40}(\text{mol H}^+/\text{dm}^3)^{0.23}}$

See Eq. [4].

cal analysis have been given in Reference 23. Further modeling work on the later stages of reaction is in progress.

HCl and NaCl additions not only have different effects on the initial rate, but also differ in their influence on the total leaching time and the shape of the leaching curve. In contrast to high salt concentrations, high acid concentrations decrease the overall leaching time significantly. Pairs of experiments were done with the same oxidant and total chloride concentrations but different acid and salt concentrations (3 mol/dm³ NaCl and 0.4 mol/dm³ HCl, 4.5 mol/dm³ NaCl and 0.4 mol/dm³ HCl, 3 mol/dm³ HCl and 0.4 mol/dm³ NaCl, and 4.5 mol/dm³ HCl and 0.4 mol/dm³ NaCl). The same amount of copper was leached in all cases. The 3 mol/dm³ HCl, 0.4 mol/dm³ NaCl leach was complete in about one-half of the time required for the 3 mol/dm³ NaCl, 0.4 mol/dm³ HCl experiment. Even more dramatic were the 4.5 mol/dm³ cases where the 4.5 mol/dm³ HCl, 0.4 mol/dm³ NaCl run took one-tenth the time of the 4.5 mol/dm³ NaCl, 0.4 mol/dm³ HCl leach. In the latter case, an acceleration in leaching was not discernible; a maximum in the Cu(II) concentration was measured, however.

There is apparently no published information on the effect of HCl on CuFeS₂ in the absence of an oxidant. Therefore, runs were performed that did not initially contain oxidant to determine whether acid or salt alone leached CuFeS₂ or caused any change in the characteristics of the solid. Leaches were done at 368 K with 48 g of −45 + 38 μm CuFeS₂, one initially with 3 mol/dm³ NaCl and 0.4 mol/dm³ HCl, and another at 0.4 mol/dm³ NaCl and 3.0 mol/dm³ HCl. After pretreatment periods of 12 and 14.5 hours, respectively, sufficient solid ferric chloride was added to produce a 0.1 mol/dm³ initial oxidant concentration.

In the 3 mol/dm³ NaCl, 0.4 mol/dm³ HCl pretreatment, neither copper nor iron was extracted from the ore before the addition of oxidant (see Figure 4). No inflection in the leaching curve was observed. This behavior is similar to that observed in normal leaching experiments (no pretreatment) at 4.5 mol/dm³ NaCl. From $t = 0$ onward, this leach curve can virtually be superimposed on that of a normal experiment with 4.5 mol/dm³ NaCl.

In contrast, when ore was added to the 3 mol/dm³ HCl, 0.4 mol/dm³ NaCl solution, a reaction producing H₂S began almost immediately. Analysis of the liquor (Figure 5) showed significant amounts of Fe(II) in the solution before the addition of oxidant; no Fe(III) or Cu was detected. The copper did not begin to leach until the addition of ferric chloride. The copper was then extracted more quickly than the iron until approximately equimolar amounts of Cu and Fe had been leached. When compared with a normal leach (Figure 6), much higher rates of copper extraction were observed following the pretreatment and addition of oxi-
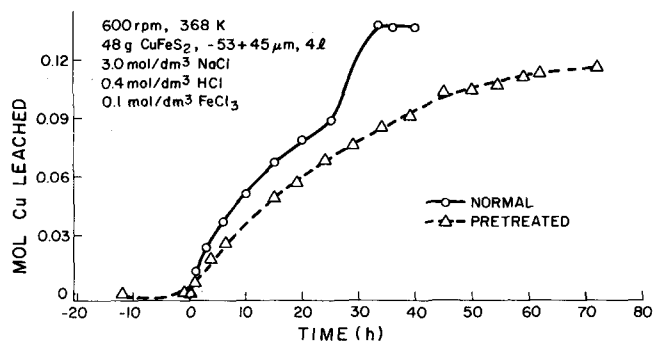
Fig. 4—Effect of 12 h pretreatment in 3 mol/dm³ NaCl and 0.4 mol/dm³ HCl. FeCl₃ concentration 0.1 mol/dm³ at time = 0, 368 K, 48 g of −53 + 45 μm CuFeS₂.
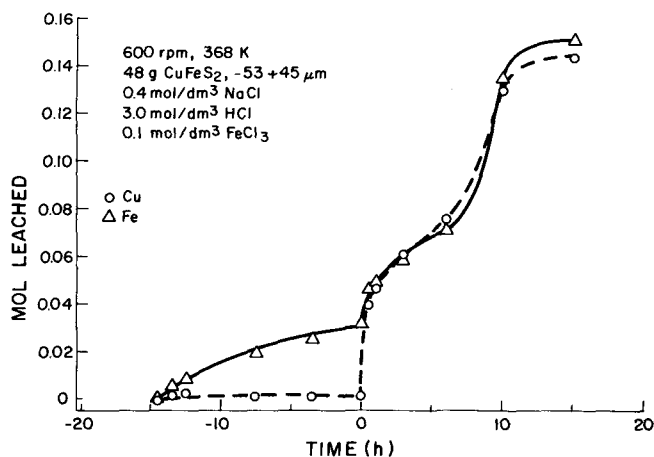

Fig. 5—Effect of pretreatment in 0.4 mol/dm³ NaCl and 3.0 mol/dm³ HCl. FeCl₃ concentration 0.1 mol/dm³ at time = 0, 368 K, 48 g, −53 + 45 μm CuFeS₂.
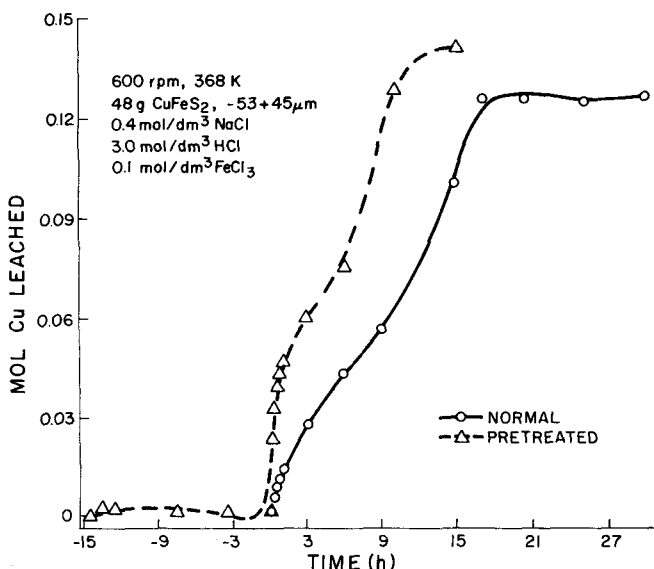

Fig. 6—Effect of 14.5 h pretreatment in 0.4 mol/dm³ NaCl and 3.0 mol/dm³ HCl on the dissolution of Cu from CuFeS₂. FeCl₃ concentration 0.1 mol/dm³ at time = 0, 368 K, 48 g of −53 + 45 μm CuFeS₂.

dant. The acid pretreatment may have formed an iron deficient layer which would be susceptible to attack by the oxidant. This slow acid attack may explain why an increase

in acid concentration slightly increases the rate of chalcopyrite leaching.

IV. CONCLUSIONS

Leaching of chalcopyrite by ferric chloride in a batch reactor has been studied using a combination of analytical and modeling techniques that permit determinations of the various solution species concentrations. The ultimate Cu(I)/Cu ratio and the ultimate extent of CuFeS₂ leaching depended on the total chloride concentration. In every experiment performed, the Fe(III) was virtually completely consumed; the Cu(II) formed by ferric leaching also reacted with the mineral, but to an extent determined by the chloride concentration. In solutions with moderate to high chloride concentrations ($\gtrsim 3$ mol/dm³), about 90 pct of the Cu(II) was reduced to Cu(I) by the cessation of the leaching process. At lower chloride concentrations, the extent of the Cu(II) reaction was proportionately less.

A rate increase was usually observed in the leaching curves in the middle of an experiment. Determination of the solution component concentrations indicated that the increase in the slope of the Cu concentration vs time curve was accompanied by a maximum in the Cu(II) concentration and a sigmoidal increase in the Cu(I) concentration. Prior to these events, the solution potential decreases gradually but is too high for Cu(II) to be reduced to Cu(I); the sharp increase in the Cu(I) concentration is accompanied by a further drop of several tenths of a volt in the solution potential.

Experiments were performed using varying concentrations of ferric chloride, HCl, and NaCl. A power dependence of 0.3 to 0.4 was found for the trivalent iron, which is in very good agreement with others reported.[9,10,11] The hydrogen ion concentration had only a slight effect on initial rates but was found to decrease the total leach time. Once leaching had begun, Fe(II) had a slight inhibitory effect on the overall leaching rate.

Chalcopyrite was found to be leached by solutions containing only HCl and NaCl; the soluble products of this reaction were Fe(II) and H₂S.

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