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Role of Galvanic Interactions in the Bioleaching of Duluth Gabbro Copper-Nickel Sulfides

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ABSTRACT

The role of galvanic interactions in the bioleaching of Duluth gabbro copper-nickel sulfides is discussed with reference to

(a) selective leaching of bulk flotation copper-nickel concentrates,
(b) environmental leaching of Duluth gabbro and flotation tailings.

There appears to be a significant, combined galvanic-bacterial oxidation effect on the rate of iron and nickel dissolution from the copper-nickel sulfide ores, bulk flotation concentrates and flotation tailings in the presence of *Thiobacillus ferrooxidans*. Specially adapted nickel and copper tolerant strains of *Thiobacillus ferrooxidans* were found to enhance the nickel and copper dissolutions from bulk flotation concentrates and the nickel-tolerant strains could be used advantageously in selective leaching.

INTRODUCTION

The role of galvanic interactions in the leaching of sulfide minerals has been pointed out by many authors in the past (1-3). The presence of bacteria, *Thiobacillus ferrooxidans*, for example, was found to accelerate the galvanic corrosion of an active mineral phase in a multi-sulfide mineral system (4-5). The differences in the rest potentials between two or more sulfide minerals in intimate contact result in selective leaching of the less noble phase,

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which could prove attractive in the treatment of complex sulfides that are often difficult to beneficiate. A combination process involving initial bulk flotation followed by selective bioleaching of the bulk concentrate had been attempted in the treatment of a complex lead-zinc-copper sulfide ore (6).

The copper-nickel mineralization in the Duluth gabbro of northeastern Minnesota contains chalcopyrite-cubanite, pentlandite and pyrrhotite as the major sulfide minerals with minor amounts of millerite, sphalerite and bornite, finely disseminated in the interstices between, or intergrown with silicates such as plagioclase, olivine and pyroxene. Though this deposit has not yet been commercially exploited, beneficiation studies both on a laboratory and pilot plant scale have indicated practical problems in differential flotation to yield separate copper and nickel rich products (7). The difficulties associated with differential flotation stem from the fact that the major copper mineral is cubanite rather than chalcopyrite; hence, hydrometallurgical processing of bulk copper-nickel concentrates appears to be a logical alternative to be investigated, since direct leaching "in situ" or in "dumps" of the as-mined ore may not be practical due to the higher pH buffering capacity of the gabbro (8), and also due to the potential water pollution problems as this deposit is situated in an environmentally sensitive region.

This work was undertaken in order to study the microbe-mineral interactions of interest to Duluth gabbro copper-nickel sulfides with special reference to the following aspects:

(a) Selective bioleaching of bulk flotation copper-nickel concentrates,

(b) Role of bacteria in acid generation and heavy metal ion release from typical Duluth gabbro ores and flotation tailings in contact with an aqueous environment, from an environmental point of view.

The role of galvanic interactions in the dissolution of copper, nickel, and iron from mixed sulfides involving chalcopyrite-cubanite, pentlandite and pyrrhotite in the presence and absence of *Thiobacillus ferrooxidans* under different experimental conditions is discussed.

EXPERIMENTAL

Active cultures of *Thiobacillus ferrooxidans*, ATCC 13728 grown in 9K media (9), were used in these studies. Leaching tests were carried out in Erlenmeyer flasks arranged on a rotary shaker incubated at 35°C. Uninoculated flasks contained 5 mL of a 2% alcoholic thymol solution as a bactericide. Chemical analyses and size distributions of ores, tailings, and concentrates used in this study are given in Table 1. Fifty gram samples of Duluth gabbro ore, flotation tailings and bulk flotation concentrate, as the case may be, were taken in 200 mL of $9K^-$ media (9K without ferrous ions) for leaching studies.

In all the leaching tests, sample solutions were withdrawn from time to time for analysis and an equal volume of the medium was replaced. Any evaporation losses during leaching were compensated for. Copper and nickel were analyzed through atomic absorption spectrophotometry.

TABLE 1

Chemical Analyses and Size Distributions of Duluth Gabbro Copper-Nickel Ore, Bulk Sulfide Concentrate and Flotation Tailings

	Ore (-10 mesh)	Bulk Conc	Flot Tail
Chemical Analyses:			
% Cu	0.8	12	0.08
% Ni	0.19	2	0.06
% Fe	11.85	30	10.4
% S	1.98	21	0.08
Size Distribution:			
+ 65 mesh	72.0	0.3	7.3
65/400 mesh	22.1	44.9	61.7
-400 mesh	5.9	54.8	31.0

In the investigations to study bacterial attachment to various sulfide minerals contained in the Duluth gabbro, polished sections of the respective minerals present, either alone or in different combinations, were kept immersed in $9K^-$ media containing *Thiobacillus ferrooxidans* and maintained at a pH of 2.0 - 2.5. Blank experiments under similar but sterile conditions in the absence of bacteria were carried out side by side. The specimens were taken out at regular intervals and observed under a scanning electron microscope.

Special strains of the above bacteria tolerant to larger doses of copper and nickel were also developed in the laboratory by repeated successive reinoculation in the medium with a gradual increase in the concentrations of copper and nickel. Such adapted strains of *Thiobacillus ferrooxidans* were used in the selective leaching of bulk copper-nickel concentrates.

For the rest potential measurements with typical Duluth gabbro sulfide minerals such as chalcopyrite, pentlandite and pyrrhotite, representative sections of the minerals were cut out from a piece of a mineralized gabbro specimen and mounted onto lucite tubing. Steady state values were measured in a $9K^-$ medium at pH 2.5.

RESULTS AND DISCUSSION

Galvanic Dissolution in Sulfide Mineral Combinations

The rest potentials of chalcopyrite, pentlandite and pyrrhotite minerals in a $9K^-$ medium maintained at a pH of 2.5 are given in Table 2. Both Pyrrhotite and pentlandite appear to be equally active electrochemically with reference to nobler chalcopyrite. There were marginal variations in the measured rest potentials with different sets of electrodes for both pyrrhotite and pentlandite, both of which exhibited a decrease in potential with time, unlike chalcopyrite. Since cubanite was inseparably associated with chalcopyrite, an electrode of cubanite free of chalcopyrite could not be prepared. Its rest potential, however, is expected to lie close

TABLE 2

Rest Potentials of Duluth Gabbro Sulfide Minerals in $9K^-$ Medium (pH 2.5)

Mineral Electrode	E_{SCE}, mV
Chalcopyrite	+ 250 mV
Pentlandite	+ 180 mV to + 100 mV
Pyrrhotite	+ 120 mV to + 110 mV

to that of chalcopyrite. A sulfide mineral electrode consisting of a mixture of chalcopyrite and cubanite registered a potential slightly lower than that of pure chalcopyrite. Thus, in a chalcopyrite-cubanite-pentlandite-pyrrhotite combination, as present in the Duluth gabbro, preferential oxidation of pyrrhotite and pentlandite may be expected. Such an anodic dissolution would also be influenced by the relative surface areas of the various sulfide constituents, the nature and duration of the contact between them, and the property (pH, conductivity, oxygen availability, etc.) of the surrounding aqueous medium.

Scanning electron micrographs illustrating galvanic interactions in Duluth gabbro sulfides after exposing different mineral combinations for one month in a $9K^-$ medium of pH 2.5 in the absence of bacteria are shown in Figure 1. Figure 1A indicates the leached surface of pyrrhotite which had been kept in contact with a separate piece of chalcopyrite mineral with a similar surface area. Galvanic corrosion of an active pyrrhotite grain in contact with a nobler chalcopyrite grain within the same ore specimen is shown in Figure 1B. No significant galvanic interaction appears to have occurred across a pyrrhotite-pentlandite grain boundary (Figure 1C) since both these minerals lie close to each other in the galvanic series (Table 2). On the other hand, pyrrhotite and pentlandite grains separated by and in intimate contact with a cubanite mineral (Figure 1D) showed corrosion tendencies, while the cubanite surface appeared unaffected.

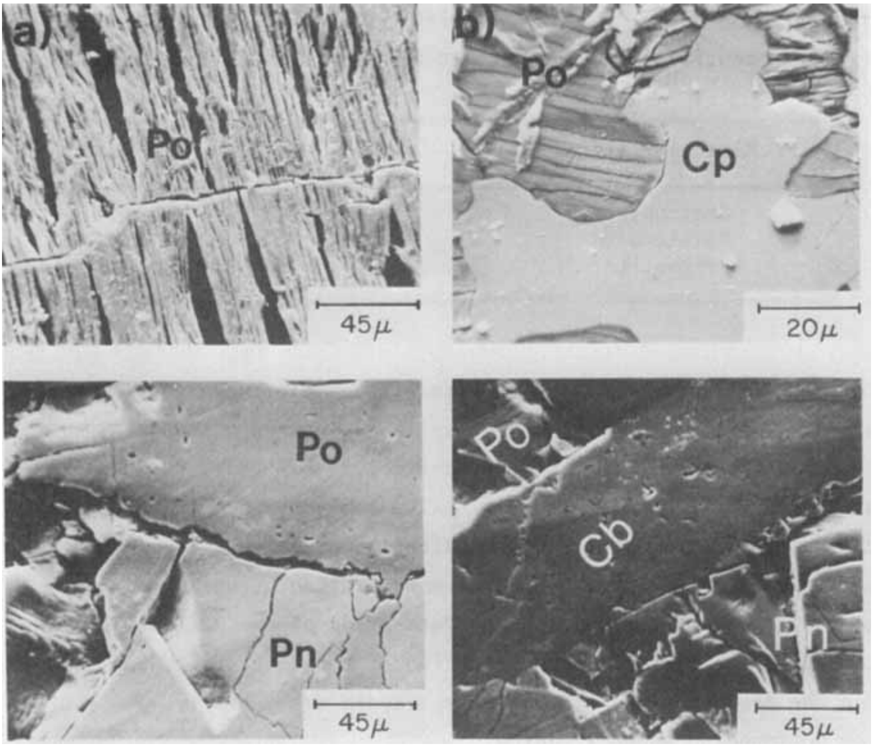

FIGURE 1. SCANNING ELECTRON MICROGRAPHS ILLUSTRATING GALVANIC CORROSION OF ACTIVE MINERAL IN CONTACT WITH NOBLE MINERAL IN THE ABSENCE OF BACTERIA IN 9K MEDIUM AT pH 2.5.

A. PYRRHOTITE (Po) SURFACE KEPT IN CONTACT WITH CHALCOPYRITE (Cp)
B. CORROSION OF PYRRHOTITE GRAIN IN CONTACT WITH A CHALCOPYRITE GRAIN IN THE SAME ORE SPECIMEN
C. PYRRHOTITE-PENTLANDITE (Pn) GRAIN BOUNDARY
D. PYRRHOTITE-CUBANITE (Cb)-PENTLANDITE GRAIN BOUNDARY

Microbe-Mineral Interactions

Thiobacillus ferrooxidans were found to attach themselves tenaciously onto all sulfide mineral substrates used in this study and galvanic dissolution mechanisms were operative in the same manner, as illustrated above. Higher dissolution rates of iron and nickel were observed in the presence of the bacteria than those observed in sterile media. A direct attack mechanism based on microbe-mineral interaction appears to exist in the biooxidation of Duluth gabbro sulfides. Typical scanning electron micrographs illustrating bacterial attachment to different sulfide mineral phases and the nature of the anodic dissolution of the more active mineral phase are shown in Figure 2. Preferential leaching of pyrrhotite and pentlandite in close contact with chalcopyrite could be observed (Figures 2B, 2C and 2D).

Chalcopyrite grains appeared tarnished and passivated. Pentlandite presented an exfoliated appearance on corrosion, and the pentlandite grains could be seen flaking off from the surface. Only limited corrosion of the pyrrhotite in contact with pentlandite and partially surrounded by ilmenite could be seen (Figure 2E). No attempt was made in this work to estimate the cell population on the different sulfide mineral substrates. Although it could not be confirmed whether any preferential bacterial attachment could occur, pyrrhotite grains were observed to contain relatively larger numbers of bacteria in the initial stages of these tests. Also, elemental sulfur along with other surface precipitates could be seen on the leached surfaces of the pyrrhotite and pentlandite.

Preferential dissolution of nickel over copper from the Duluth gabbro in the presence of *Thiobacillus ferrooxidans* could be seen from Figure 3 and such a leaching behavior is of practical significance in the selective oxidation of mixed sulfides. As pointed out above, preferential galvanic dissolution of pyrrhotite and pentlandite in contact with chalcopyrite-cubanite could be expected and the presence of the bacteria would further catalyze the selective dissolution reaction. Also, due to the reasons brought out already, selective bioleaching of bulk copper-nickel concentrates would be more practical than any attempt to leach the as mined ore.

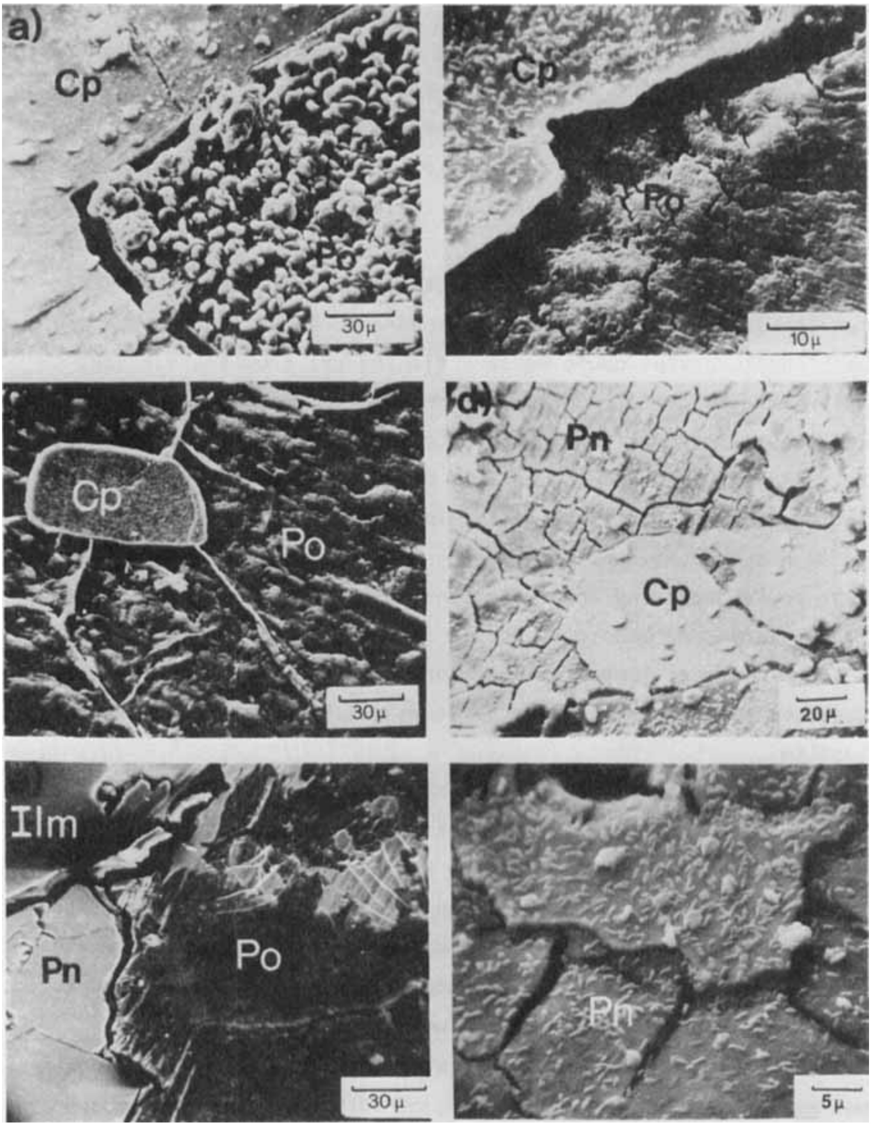

Figure 2

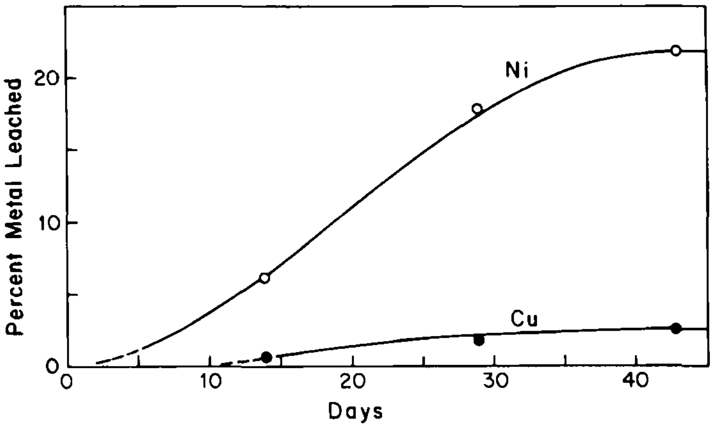

FIGURE 3. BACTERIAL LEACHING OF DULUTH GABBRO ORE.

Selective Bioleaching of Bulk Copper-Nickel Concentrates

In the microbial leaching of flotation concentrates, the bacterial strains used must tolerate higher concentrations of the metal ions so that their activity is not impeded by the successive build-up of dissolved metal ion concentration. Both copper and nickel are known to be toxic to bacterial activity, and therefore it may be essential to develop special strains of the leaching bacteria that can tolerate high levels of these metal ions to en-

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FIGURE 2. SCANNING ELECTRON MICROGRAPHS ILLUSTRATING BACTERIAL ATTACHMENT AND GALVANIC INTERACTION.

A. CHALCOPYRITE (Cp)-PYRRHOTITE (Po) INTERFACE--PYRRHOTITE SURFACE COVERED WITH BACTERIA AND REACTION PRODUCTS
B. CHALCOPYRITE-PYRRHOTITE GRAIN BOUNDARY--GALVANIC CORROSION OF PYRRHOTITE
C. CHALCOPYRITE INCLUSION AND STRINGERS IN PYRRHOTITE--ONLY PYRRHOTITE IS LEACHED OUT
D. SURFACE CRACKING AND EXFOLIATION OF PENTLANDITE (Pn) IN CONTACT WITH CHALCOPYRITE
E. ILMENITE (Ilm)-PENTLANDITE-PYRRHOTITE BOUNDARY--LIMITED CORROSION OF PYRRHOTITE
F. BACTERIAL ATTACHMENT TO EXFOLIATED PENTLANDITE GRAINS

sure efficient and continuous bioleaching of copper-nickel concentrates.

Copper and nickel tolerant strains of *Thiobacillus ferrooxidans* were cultured in the laboratory by repeated, successive reinoculation in the medium with successive increases in the concentrations of copper and nickel. Concentrations of copper and nickel, added as their sulfate salts, were increased in increments of 5, 10, 20, 30 and up to 50 g/L. Microbial activity in flasks containing various concentrations of copper and nickel was frequently tested with respect to ferrous ion oxidation and acid formation. These studies were intended to check the potential applicability of using copper and nickel tolerant strains in the leaching of copper-nickel concentrates and thus were preliminary in nature.

Separately adapted copper and nickel tolerant strains were used in the leaching of bulk copper-nickel concentrates and their leaching efficiency compared with that of unadapted strains. Typical results of leaching tests are given in Figure 4. Nickel-adapted

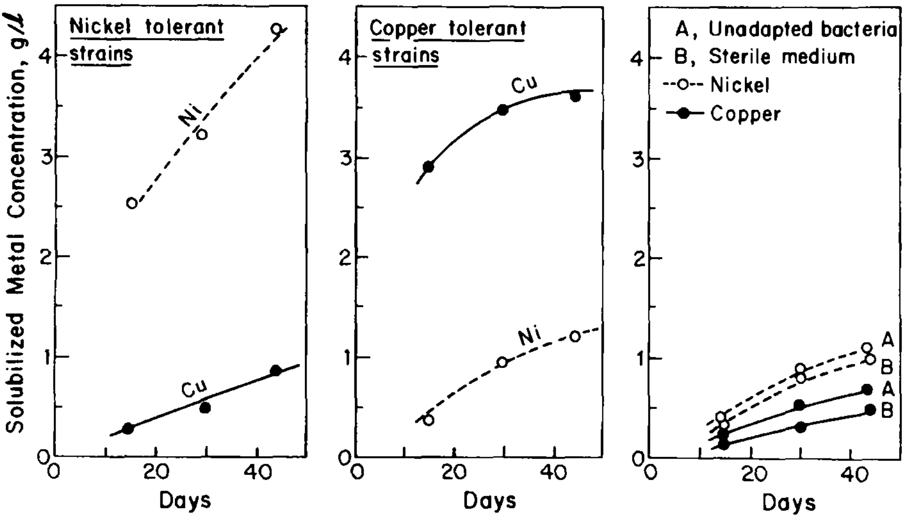

FIGURE 4. BACTERIAL LEACHING OF BULK COPPER-NICKEL CONCENTRATE WITH NICKEL-TOLERANT, COPPER-TOLERANT AND UNADAPTED STRAINS OF *Thiobacillus ferrooxidans*.

strains could extract almost 90% of the nickel within 45 days. Similarly, copper-tolerant strains aided in higher copper dissolution. It appears that unadapted strains of *Thiobacillus ferrooxidans* may not function efficiently in the bioleaching of bulk copper-nickel concentrates. The above studies also indicate that selective bioleaching of nickel from the bulk copper-nickel concentrate may be feasible with specially adapted strains of *Thiobacillus ferrooxidans*, since the lag period in the biooxidation process could be minimized if not completely eliminated, along with increasing the metal tolerance of the bacteria, through such preadaptation.

Role of Galvanic Interactions in the Environmental Leaching of Duluth Gabbro

Since sulfur/iron bacteria of the type *Thiobacillus ferrooxidans* are indigenous to the sulfide mineral system, they serve as living catalysts for mineral oxidation reactions. Such a microbial activity is known to contribute towards acid mine drainage and heavy metal ion release from mines, ore stock piles and tailing ponds. The role of microbial activity in the environmental control of copper/nickel bearing Duluth gabbro is discussed in detail elsewhere (10,11).

Metal ion release and acid generation from Duluth gabbro ore and flotation tailings in the presence and absence of externally added *Thiobacillus ferrooxidans* are illustrated in Table 3. The presence of the bacteria was found to contribute towards more acid production and copper/nickel dissolution from the flotation tailings and the gabbro ore in contact with an aqueous medium. Nickel release into the solution was faster and in all cases took place before copper appeared in solution. Under acidic conditions, nickel concentrations in solution were several times higher than those of copper. As reported in a separate paper (11), copper concentration in solution was found to increase only after most of the nickel has been solubilized. The dissolution tendencies for the copper and nickel were found to be different.

TABLE 3

Metal Ion Release from Duluth Gabbro Ore and Tailings (1 Month Leaching in Shake Flasks, 50 g/200 mL of 9K⁻ Medium, 35°C) (11)

Condition	Material	Initial pH	Final pH	Cu, ppm	Ni, ppm
A. In the presence of *Thiobacillus ferrooxidans*	Tailings	7.2	5.5	2.7	65
		3.4	3.9	240	445
		2.4	3.9	330	444
	Ore	7.1	4.8	7.8	191
B. Sterile	Tailings	7.2	6.3	2.5	36
		3.4	6.5	0	36
		2.4	6.3	0	-
	Ore	7.1	7.3	0	3

Copper:nickel ratios in the gabbro samples as well as the reaction of copper ions with pyrrhotite and pentlandite might be influencing the copper:nickel ratios in solution. A decrease in copper concentration in solution with time for the ore and flotation tailings in contact with the aqueous medium even under acidic pH conditions was observed in a few tests. Exchange reactions involving pyrrhotite and cupric ions of the type

$$Cu^{++} + FeS = CuS + Fe^{++} \qquad (1)$$

could be considered to explain the above observation. Galvanic displacement of dissolved copper in the presence of active minerals such as sphalerite (12) and pyrrhotite may also be possible.

Bio- and Electro-Chemical Mechanisms

There appears to be a significant, combined galvanic-bacterial oxidation effect on the rate of iron and nickel dissolution from the Duluth gabbro sulfides. Figure 5 illustrates such a combined mechanism for the anodic dissolution of pyrrhotite in contact with chalcopyrite. Preferential leaching of the pyrrhotite present adjacent to a chalcopyrite grain in the gabbro sample is shown in

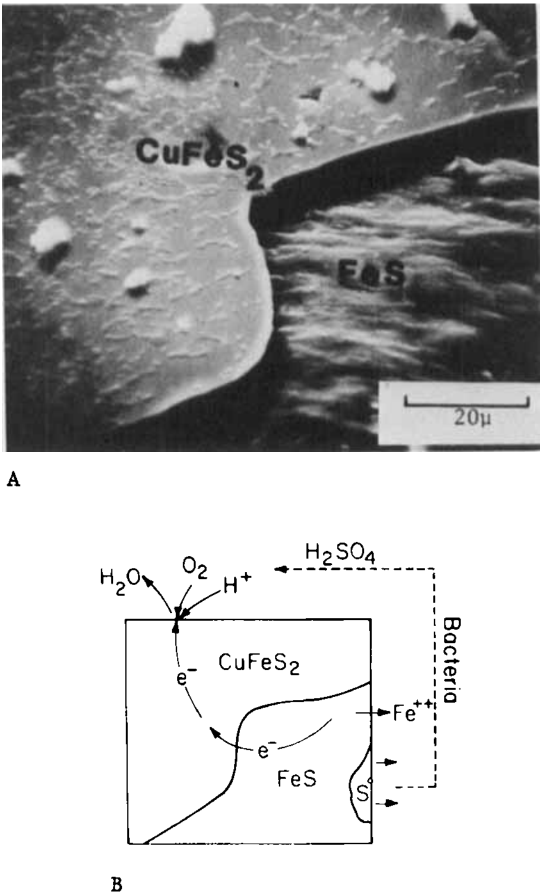

FIGURE 5. MODEL ILLUSTRATING COMBINED BACTERIAL-GALVANIC OXIDATION IN A CHALCOPYRITE-PYRRHOTITE CONTACT.

A. SCANNING ELECTRON MICROGRAPH SHOWING PREFERENTIAL LEACHING OF PYRRHOTITE IN CONTACT WITH A CHALCOPYRITE GRAIN IN THE DULUTH GABBRO

B. ELECTROCHEMICAL AND BIOCHEMICAL REACTIONS AT THE CHALCOPYRITE-PYRRHOTITE CONTACT IN THE PRESENCE OF OXYGEN the scanning electron micrograph 5A. The follow electrochemical reactions are indicated in the model 5B:

$FeS = Fe^{++} + S^{\circ} + 2e$ (anodic dissolution of pyrrhotite) (2)

$O_2 + 4H^{+} + 4e = 2H_2O$ (cathodic reduction of oxygen at chalcopyrite surface) (3)

A similar reaction for the anodic dissolution of pentlandite in contact with chalcopyrite-cubanite could be written as follows:

$$(Fe_xNi_{1-x})_9\ S_8 = 9x\ Fe^{++} + 9(1-x)Ni^{++} + 8S^{\circ} + 18e \qquad (4)$$

In the presence of *Thiobacillus ferrooxidans*, the leaching rate of iron and nickel from the Duluth gabbro in an acidic medium was found to be further enhanced by several folds. A few leaching tests in shake flasks were carried out by mixing chalcopyrite and pyrrhotite concentrates in different ratios in the presence and absence of bacteria and it was observed that the amount of dissolved iron increased 3 to 4 times in the presence of the bacteria as opposed to a sterile medium. With increasing amounts of pyrrhotite in the mixture, the amount of copper dissolved was found to decrease. Although the bacteria oxidized all the sulfide minerals present in the gabbro, nickel and iron were leached out at a faster rate and earlier than copper indicating the significant role played by galvanic interactions. Fe^{++}, $S^{=}$ and S° are energy substrates for the bacteria. Elemental sulfur formed at the mineral surfaces due to oxidation will be subsequently oxidized by the bacteria, resulting in the exposure of fresh mineral surfaces and generation of acidic ferric sulfate lixiviant. Thus, the presence of the bacteria could indirectly enhance the leaching rate. As discussed above, the bacteria also participate in a "direct attack mechanism," disintegrating sulfide mineral lattices through attachment and direct interaction with the substrate. Controversy exists in the literature (4) regarding bacterial attachment to preferred sulfide mineral sites. It is also not well established as to whether the bacteria could directly participate in the selective oxidation of one sulfide mineral in a mixture of sulfides. It is evident from the present study that specially adapted nickel and copper tolerant strains exhibit selectivity in the leaching of nickel and copper, respectively, from bulk flotation concentrates. Tolerance to larger concentrations of copper and nickel appears to be an essential prerequisite for sustained bacterial activity in the leaching of concentrates. For example, while almost 90% of the nickel could be extracted from the bulk concentrate in the presence of nickel-tolerant bacteria, only about 22% could be leached out with unadapted *Thiobacillus ferrooxidans*, after 45 days of leaching in shake flasks. Similarly, the presence of copper-tolerant strains was found to increase the percent extraction of copper from the above concentrate by about six times in comparison with unadapted bacteria (Figure 4). The role of bacteria in the selectivity as well as enhancement of leaching rates thus may not be an indirect one alone. With unadapted bacteria, the leaching rate of either nickel or copper from the concentrate was not much different from that obtained in a sterile acid medium, indicating that bacterial activity did not significantly contribute to leaching and that the galvanic dissolution mechanism was the only predominant factor. With specially adapted strains, a different picture emerges as illustrated above. In the leaching of the copper-nickel ore or the flotation tailings, however, the presence of bacterial activity was found to enhance metal ion dissolution quite significantly. Unlike the leaching of concentrates, metal ion concentrations encountered in the leaching of low grade gabbro materials are smaller and may not attain higher toxicity levels, impairing bacterial activity.

CONCLUSIONS

Galvanic interactions play a significant role in the dissolution of nickel and copper from Duluth gabbro sulfides. The presence of *Thiobacillus ferrooxidans* in the leaching system was found to enhance the rate of dissolution of iron, nickel and copper from Duluth gabbro ores and flotation tailings. Specially adapted nickel and copper tolerant strains of the above bacteria exhibited better selectivity and higher tolerance towards nickel and copper respectively, in the bioleaching of bulk flotation concentrates.

Selective bioleaching of nickel as opposed to that of copper from the Duluth gabbro bulk flotation concentrates appears to be more favored and practical due to the following reasons:

(a) The concentrates contain six times more copper than nickel.

(b) Leaching of pentlandite in the presence of a larger concentration of chalcopyrite-cubanite would be favored galvanically.

(c) Generally, *Thiobacillus ferrooxidans* appears to be more tolerant to nickel than copper and strains with a higher tolerance limit for nickel than copper can be cultivated in a laboratory (13).

(d) Due to the relatively smaller concentration of nickel in the concentrate, the nickel ion build-up in solution may not exceed the bacterial tolerance limit achieved in the laboratory (13) and as such higher percent extraction for nickel can be expected.

The combined influence of nickel and copper ions on bacterial activity, however, remains to be understood.

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