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CHEMICAL, BIOLOGICAL, AND METALLURGICAL ASPECTS OF LARGE-SCALE COLUMN LEACHING EXPERIMENTS FOR SOLUTION MINING AND IN-SITU LEACHING

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ABSTRACT

It is well known that the variations in temperature, chemical reactivity, and reaction rates which are presumed to occur and which in some cases have been shown to occur in a leach dump or similar solution mining activity are difficult to duplicate or model on a small laboratory scale, and the degree to which such large-size metallurgical processes can be scaled is not well established. In the present investigations the design criteria and sampling methods for large-scale leach tests on low-grade copper waste are outlined and the results of studies of temperature profiles and associated chemical and biological activity in 185 ton (1.63×10^5 Kg) waste samples (approx. 10.75m in height) are discussed. Various simple scaling features are presented, including drain-down characteristics and waste-body neutralization (of acid consuming minerals) for test samples of 7.0, 4×10^2, and 1.7×10^5 Kg. Simple geometrical scaling laws are shown to closely describe the greater part of these processes. Temperature profiles in a large waste body are shown to be related to or correlated with bacterial activity, which is further shown to be related to available oxygen (and oxygen consumption). Aeration of large waste bodies is shown to be correlated with enhanced bacterial activity and leaching rates, and certain features of bacterial catalysis observed in the large-scale tests are corrob-

orated by detailed laboratory studies of bacterial leaching phenomena utilizing the scanning electron microscope. Long-term data profiles showing a relationship between temperature, bacterial activity, oxygen consumption, and variations in Fe^{2+}, Fe^{3+}, Cu concentration, pH and Eh in the waste body are presented along with the variation of these parameters.

INTRODUCTION

The importance of bacterial activity in the sulfide copper leaching process was first recognized in the recovery of copper in the Rio Tinto (Razzell and Trussel[1]), and since then it has been convincingly demonstrated at least on a laboratory scale that leach solutions inoculated with an appropriate microorganism are much more effective in solubilizing copper from a mineral-bearing waste than those solutions which do not contain any measurable bacterial population or bacterial activity. Although the evidence supporting the effects of bacterial catalysis is overwhelming at the laboratory scale, there has been little direct evidence of the effect of bacterial catalysis in large leach dumps and similar large-scale processes. While Bhappu, et al.[2] have, for example, attempted to determine the extent of bacterial activity within a leach dump, there have been no detailed correlations of leach dump bacterial activity with temperature profile, elemental concentrations (of Fe^{2+}, Fe^{3+}, and Cu) indicative of the efficiency of the copper leaching process; as well as oxygen available within a leach dump. As a result of possible temperature, concentration, and oxygen profiles within a leach dump, and the effect these parameters would be expected to have upon bacterial activity, or the effect bacterial activity may have upon these parameters, it is difficult to characterize such effects in a large leach dump or similar large-scale solution mining operation by studying small-scale, laboratory column tests. There is in general a dearth of information relating to the ability to scale small column leach tests to large leach dump operations or to model such operations, and it is difficult if not impossible to effectively duplicate temperature and biochemical reaction profiles which are expected to occur in a large leach dump, in small-scale, laboratory column tests of only a few hundred kilograms of waste rock.

Madsen and Groves[3], who recently completed a long-term, large-column (7.9×10^3 Kg) leaching experiment on a low-grade 0.25% copper (chalcopyrite) waste, concluded that enhanced leaching and leaching rates which occurred after aerating the waste body were due primarily to bacterial catalysis. However, there were no direct observations of bacterial population or activity within the waste body, and there was also no attempt to systematically evaluate the changes in solution chemistry, or to determine associated variations in the waste body temperatures with leaching time and aeration. Cathles and Apps[4] have recently developed a dump leaching model which illustrates the importance of air convection and temperature profiles.

The present experiments were devised in an attempt to overcome some of the shortcomings outlined above. In particular, the waste columns tested were considered to be representative of a small leach dump or unit of a large dump or heap because they were able to hold nearly 190 tons (1.7×10^5 Kg) of waste; having a height in the 3.08m-diameter waste column of approximately 10.5m. In addition, the test columns were designed to allow for access to the leach solution as it permeated the waste body, and for periodic sampling of waste particles in order to monitor bacterial population and activity at locations within the waste column; and thermocouples were embedded within the waste body at various locations to allow the temperatures to be measured and corresponding temperature profiles to be determined. The application of simple scaling laws in conducting drain-down experiments and the neutralization of an acid-consuming waste was also tested in the present experimental program. Finally, laboratory studies of bacterial catalysis utilizing the scanning electron microscope were undertaken in an effort to elucidate the detailed nature of the associated microorganisms and their apparent role in the leaching process; particularly pyrite and chalcopyrite surface reactions (corrosion rates).

EXPERIMENTAL METHODS AND DESIGN CRITERIA

The large tanks utilized in these experiments are converted liquid oxygen storage dewars measuring roughly 40 ft (12.3m) in overall height and 10 ft (3.1m) in inside diameter. The inside jacket wall is a 304 stainless steel separated from the outer carbon-steel wall by 1 ft (0.3m) of perlite insulation. Currently two tanks in an array of four have been loaded with low-grade waste and tested for varying periods. Prior to loading, drains were cut into the bottom of each tank and provision for aeration made. An air distribution system consisting of a 5 ft. square 1 in. schedule-80 PVC pipe with 0.125 in. holes spaced 1 in. apart was placed on the flat tank bottom. A 1m high quartzite bed (+2") was placed upon the air distributor and the waste rock loaded by hand (hand placed) to a depth of approximately 0.3m. One tank was loaded with Kennecott Chino Mines Division (CMD) Santa Rita waste rock (~ -4"). The total copper was 0.36% with non-sulfide (acid soluble) copper accounting for 0.14% and the balance disseminated as chalcocite and chalcopyrite; with the chalcopyrite representing < 0.1 wt %. The pyrite/chalcopyrite ratio was determined to be 10.6. The rock matrix was a quartz monzonite which required little detectable neutralization.

Four access ports were cut into the sides of the leach tanks corresponding to irregular depths in the loaded waste body, and alternated at 90° from one another along a diameter. Following the initial hand loading of the waste, the next 3.6m depth was loaded by elevator. Upon reaching the first port level, an array of instruments were installed consisting of a half-section pipe array to collect solution, a lysimeter (porous-cup collector for solution sampling) located in the center of the tank, and at least 2 thermocouples near the center of the waste body; with one acting as a backup. This routine was continued until the waste was fully loaded and all access ports installed. The waste rock was hand loaded to a depth of roughly 0.3m over the instruments installed at each level.

A second tank was loaded in a similar manner with Duval-Sierrita waste (~ -6") with a total copper content of 0.34% of which

0.03% was non-sulfide (acid soluble) copper, and the balance chalcopyrite. The pyrite/chalcopyrite ratio of this rock was determined to be 4, and the matrix, unlike the Kennecott rock, contained a considerable carbonate concentration (~ 2%) to be neutralized. In the loading of the Duval waste, a system of funnels (10 in. diameter) filled with screens and alumina balls were used in place of the pipe half-sections to collect solution at locations within the waste body. In addition, an array of gypsum moisture blocks was placed at various locations (at the gravel bed/waste body interface near the bottom and at each access port level). While in the Kennecott waste body, rock samples were removed periodically by augering through the solution collector opening at the access ports, a separate 2 in. fitting was provided for this purpose in the Duval tank. Each access port was provided with fittings to allow oxygen content to be measured. The top of each tank was vented through a 1 in. opening where oxygen content and air flow could be measured. An air compressor fitted with pressure control valves and a flow meter was used as a source of air (oxygen) through the air distributor at the bottom of the gravel bed.

The solution distributor at the top of each tank consisted of a 1 in. stainless steel shaft through a reservoir connected to the center of an arm extending 9.5 ft. across the inside diameter, with 1/16 in. holes in the upper side at decreasing spacing from the center shaft. The shaft itself was slotted to allow a permanent solution head in the reservoir, and was rotated with a high-torque motor at 1 rpm. The solution circuits normally contained approximately 5000 gal. (22.7×10^3 ℓ) of solution. The circuit itself consisted of a 10,000 gal. stainless steel holding tank (gravity fed from the waste column), a cascade scrap-iron launder, and an array of surge tanks or holding tanks between the launder and the waste column.

Figures 1 and 2 illustrate the principal features of the instrumentation systems at the access ports, the solution circuit, and overall views of the facility described above. Figure 3(a)

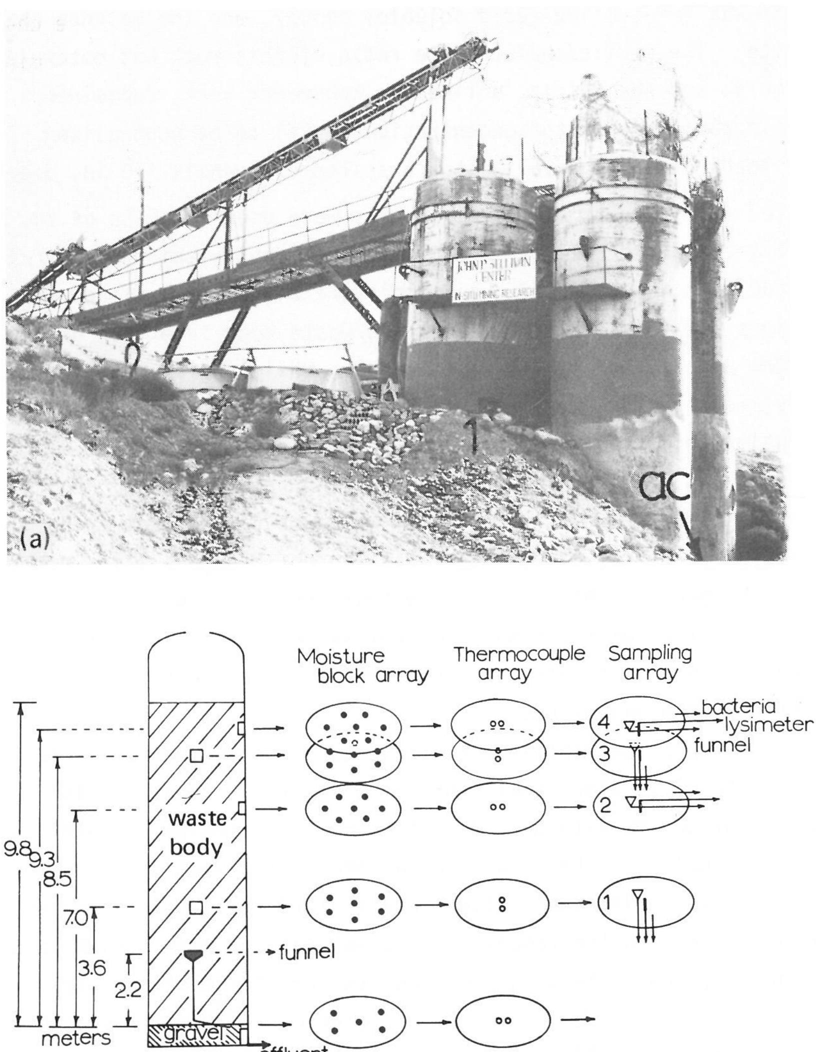


FIG. 1: Large leach tank facility and sampling and instrumentation design. (a) Overall view of leach-tank facility, (b) Duval-Sierrita waste-body sampling and instrumentation design features. Locations and arrays of moisture blocks and thermocouples are shown. The sampling arrays at access ports allow for the extraction of rock particles for determining bacterial population and activity, and solution extraction either from funnel collectors or a lysimeter. The lysimeter consists of a porous cup through which solution is drawn under vacuum and then expelled under pressure. Access port 1 is shown at (1) in (a) while (ac) denotes the air compressor.

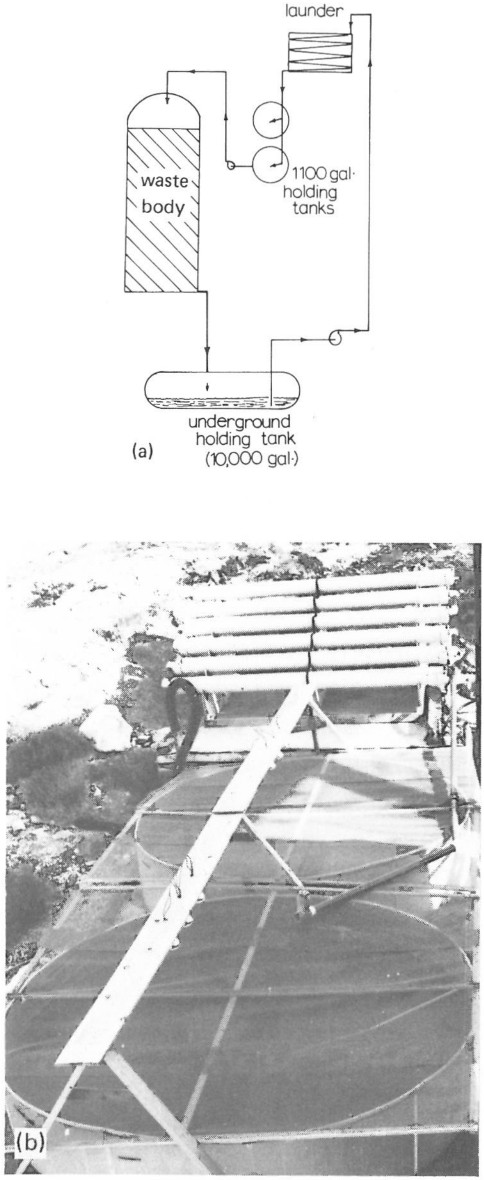
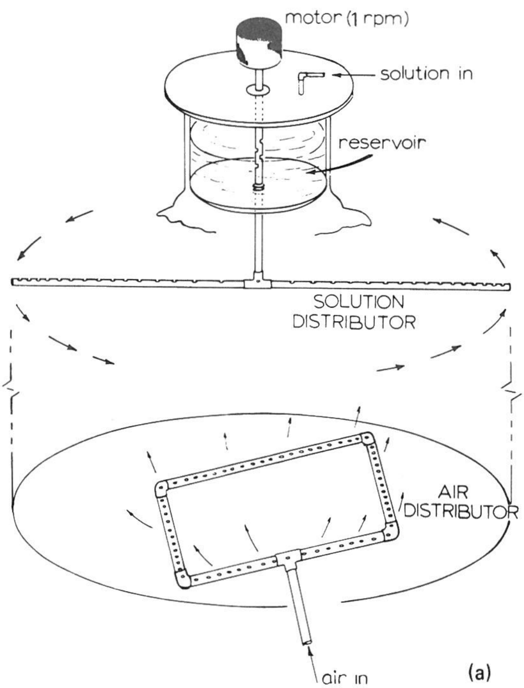


FIG. 2: Solution circuit schematic (a) and full view of launder and holding tanks shown in Fig. 1(a) (b).

illustrates schematically the essential features of the air and solution distributors described above, while Fig. 3(b) compares the drain-down characteristics for the scaled tests. Figure 4(a) and (b) illustrate the waste-body permeabilities throughout the waste column in the dry and solution-saturated states determined by measuring the pressure difference from inlet to outlet at constant flow rate. It is obvious from Fig. 4(a) and (b) that the waste-body permeabilities were considerably different, primarily as a result of differences in particle size distribution and waste body integrity. The variation in permeability is caused by the difference in packing. The waste packed to a greater packing density near the bottom as compared with the top of the waste body as a consequence of the loading technique.

Simultaneous with the loading and initial testing of the Duval-Sierrita waste, smaller column tests were devised to function in a scale test of the large-column leaching experiments. A 0.38m diameter and a 0.10m diameter column were loaded with 405 and 7 Kg of rock sized approximately on a scaled basis by multiplying the large tank maximum rock size (6 in.) by the ratio of the scale column diameter to the large column diameter. It was noted that the ratio column diameter/column height was smaller by a factor 2 for the smaller columns when compared to the large column, and this factor was taken into consideration when scaling the solution and air flow rates. The drain-down characteristics of the smaller columns from a solution-saturated condition were also scaled in terms of initial drain-down flow rate for the large column, and the ratio of retained fluid to saturated fluid volumes determined. These data are listed for comparison in Table 1. Figure 3(b) illustrates the experimentally determined drain-down characteristics for the Duval-Sierrita waste in the large column and the smaller, scaled columns.

Copper and iron analyses were determined by atomic absorption spectroscopy and ferrous iron (Fe^{2+}) by titration methods. Oxygen concentrations were determined at the large waste body access port



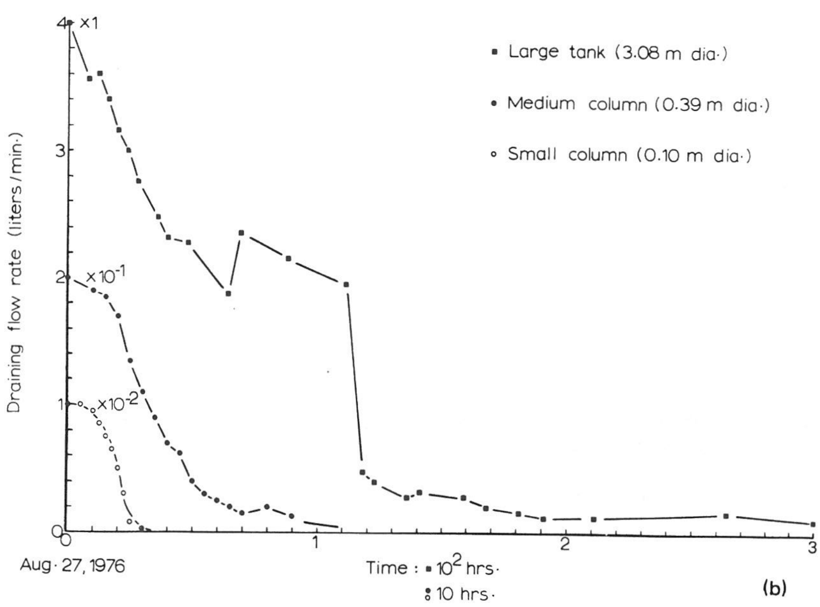


FIG. 3. (a) Schematic view of the solution and air distributors in the large waste columns. (b) Initial drain-down characteristics for the large Duval waste body and the associated, smaller scaled columns.

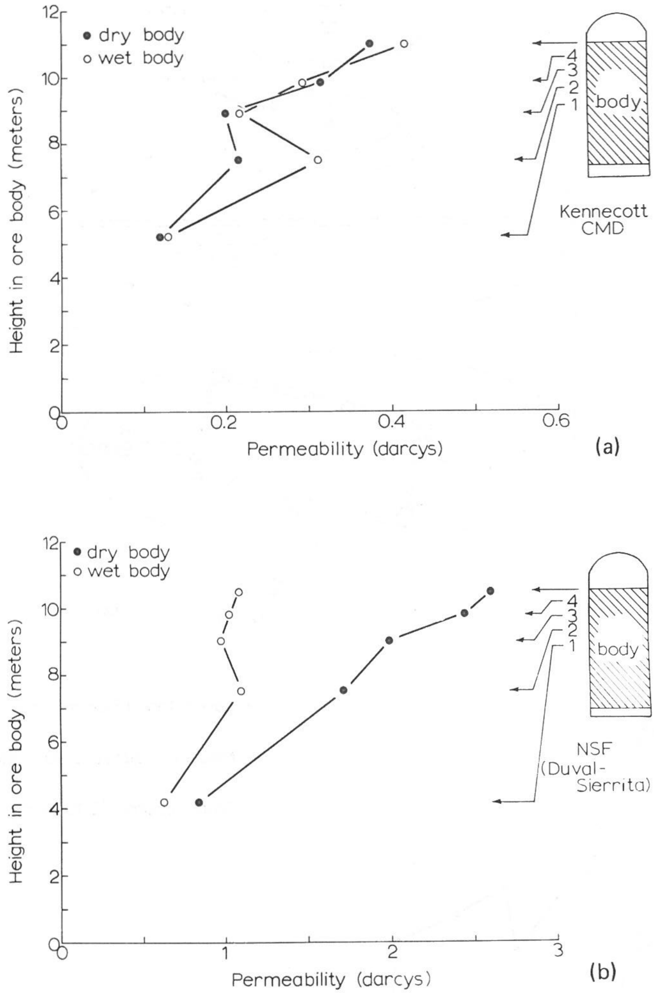


FIG. 4. Waste body permeabilities in the dry and solution-saturated conditions. (a) Kennecott, CMD (Santa Rita) waste body, (b) Duval-Sierrita waste body. The permeability unit, darcy, is equivalent to a fluid flow of 1cc/s-cm^2 (1 centipoise viscosity under a pressure gradient of 1 atmos./cm).

TABLE 1

Duval-Sierrita Waste Scale-Test Data

Test Parameter	Large Column	Medium Column	Small Column
Diameter, d (meters)	3.05	0.38	0.10
Height of Rock, h (meters)	9.75	2.64	0.69
d/h	0.290	0.145	0.145
Maximum Rock Size (in.)	6	0.75	0.20
Initial Drain Rate (ℓ/min.)	4	0.20	0.10
Initial Air Flow Rate (ℓ/min.)	17.5	0.875	0.044
Initial Solution Onfluent (ℓ/min.)	2	0.100	0.005
Retained/Saturated Solution	0.77	0.72	0.68
Waste Body Weight (Kg)	1.7×10^5	4.05×10^2	7

locations using a commercial oxygen meter. Bacterial populations in solution and in the waste body were determined by a most probable number method (MPN) for Thiobacillus ferrooxidans. In addition, laboratory studies involving both Thiobacillus ferrooxidans and a high-temperature microbe (Brierley and Brierley[5]) were conducted to obtain quantitative evidence for bacterial catalysis and to

determine more specifically the characteristics of bacterial attachment, the need for attachment, the site of attachment, and the degree of surface corrosion and the effect of bacterial catalysis on surface corrosion and reaction of FeS_2 and $CuFeS_2$ phases. Direct observations of bacterial phenomena and catalytic activity were made by systematic observations in a Hitachi HHS-2R scanning electron microscope operated at 25 kV, and fitted with an Ortec energy-dispersive X-ray analysis facility. Specific sulfide phase regions and other mineral inclusions in the waste particles were in fact determined by X-ray spectroscopy in the scanning electron microscope.

RESULTS AND DISCUSSION

Figures 5 and 6 illustrate some of the preliminary scale data and neutralization characteristics for the Duval-Sierrita waste. The initial inundation for the Sierrita test was made with Socorro

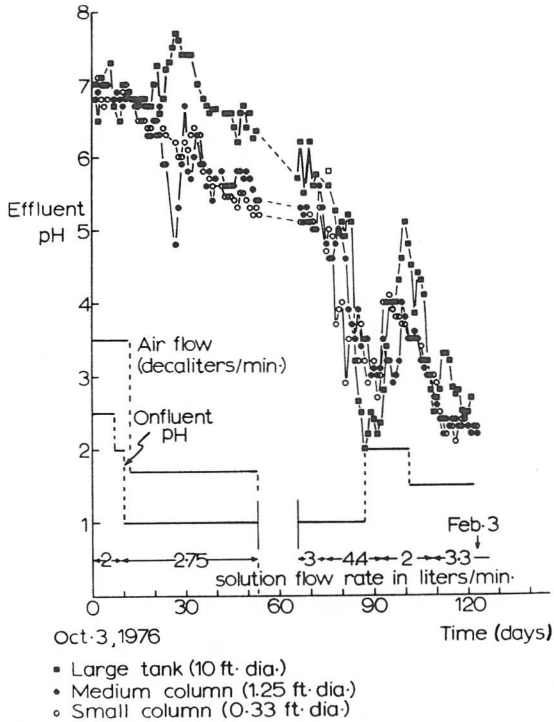


FIG. 5. pH data showing neutralization of the Duval-Sierrita waste rock. The efficiency of the scaling experiments is somewhat apparent.

well water acidified initially to a pH of 2.0. Subsequent solution onfluent pH adjustments and air flow schedules are indicated in Fig. 5. While the initial solution contained no measurable T. ferro-oxidans population, the waste body prior to solution inundation was observed to contain evidence of bacteria, which, as demonstrated in the data of Fig. 6(b) increased in the waste body during the neutralization period. However, temperatures even at the end of 120 days were not above 10°C, and the copper in solution averaged only 50 ppm; indicative of no significant bacterial catalysis or leaching.

The Kennecott-Chino Mines waste, by comparison with the Duval-Sierrita waste, was inundated with CMD tail-water which contained

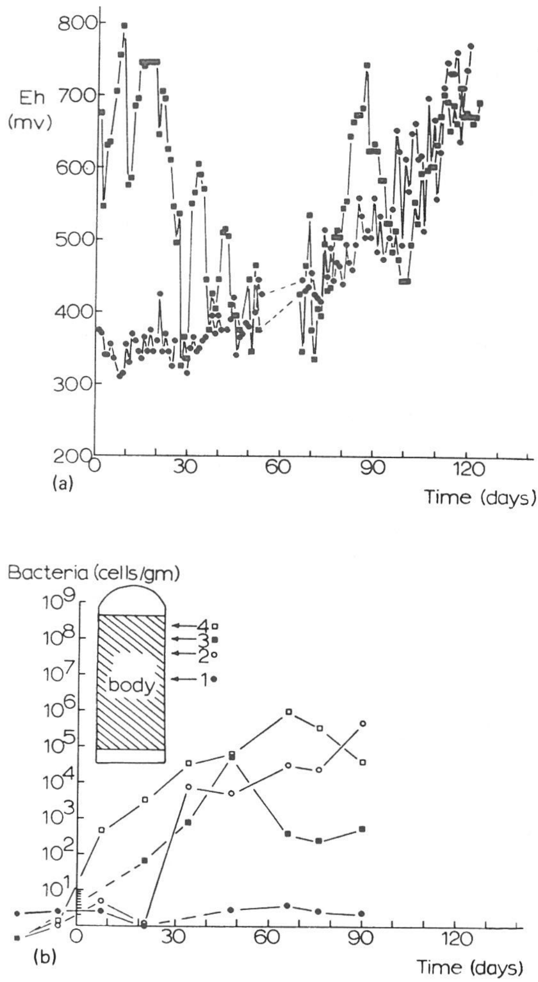


FIG. 6. (a) Eh data for the large and medium-scaled Duval-Sierrita waste bodies corresponding to the neutralization period shown in Fig. 5. (b) Bacterial population profiles (Thiobacillus ferrooxidans) in the large waste body corresponding to the neutralization period shown in Fig. 5.

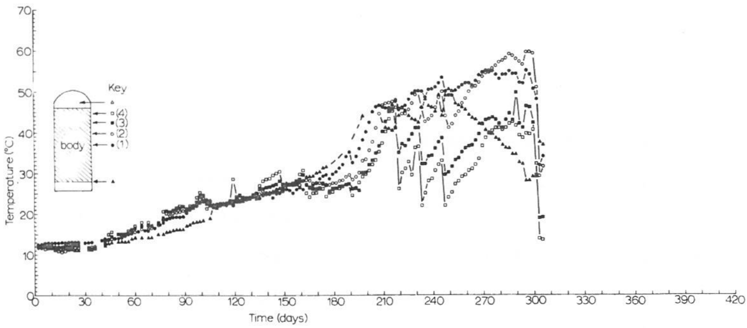
appreciable numbers (> 10^5 organisms/cc) of bacteria (Thiobacillus ferrooxidans). Unlike the Duval waste, the Kennecott waste required little neutralization, and slightly more than 10% of the total copper (as non-sulfide copper) was extracted during the initial solution inundation. Following the initial inundation, a schedule of solution cycles and aeration rates (including solution rest periods) was followed based in large part on the dump leaching computer model developed by Cathles and Apps[5]. One of the principal reasons for the test was to obtain experimental data to support the model predictions.

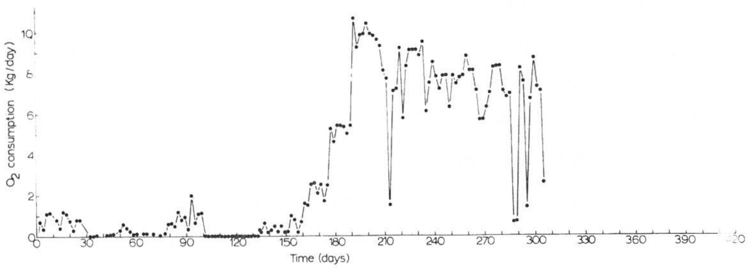
Figure 7 illustrates the temperature profiles, oxygen consumption and levels, bacterial count, and copper extracted and solution flow (flush) schedules for roughly the first 300 days of leaching the Kennecott-CMD waste body. It is of particular interest to note that bacterial population, that is the population of Thiobacillus ferrooxidans, began to decline at approximately 45°C, the upper limit for known strains of the microbe (Marchlewitz and Schwartz[6]). While, as shown in Fig. 7, there is some very small decline in oxygen consumption with the decline in the Thiobacillus ferrooxidan population, the oxygen consumption remains high, and the temperature continues to increase. The maximum temperature attained within the waste body was 60°C, and at the final flush [not shown in Fig. 7 (#14)], the temperatures were dramatically quenched, and tended to stabilize thereafter between a maximum (not shown) of 35-40°C; indicative of some equilibrium condition. There was some evidence during bacterial sampling and analysis at the optimum temperatures that a high-temperature microbe was present within the rock, but no detailed evidence was obtained. However, it is well known that the optimum temperature for sulfide leaching catalyzed by Thiobacillus ferrooxidans is 35°C, and that biological oxidation appears to cease around 55°C (Bryner, et al.[7]). It is difficult to believe the acceleration of waste body heating and the associated reactions (particularly with regard to copper solubilization during solution flushes) could be the result of only chemical oxidation, and a high temperature microorganism would seem to be the only logical alternative.

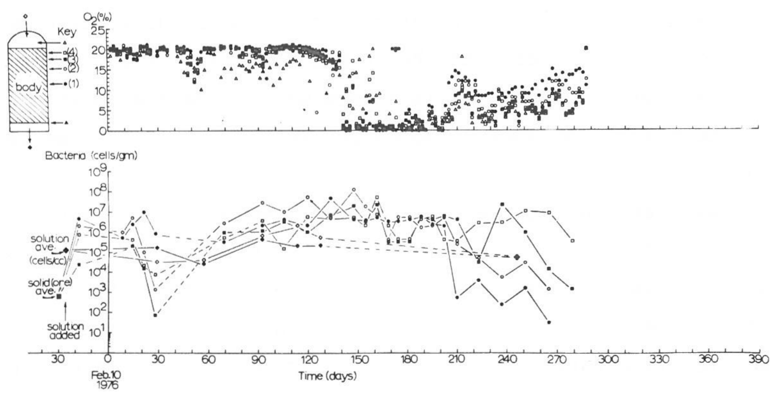
Figure 8 shows several examples of the flush data following cyclic leaching of primarily non-sulfide copper up to and including flush 6 (Fig. 7). It can be observed in Fig. 8, with reference to Fig. 7, that effluent copper increased with waste-body temperature, pH decreased, and the conversion of Fe^{2+} to Fe^{3+} appeared to increase in the same proportion. The high proportion of Fe^{3+} in the flush effluent, and the increase in the level of Fe^{3+} and the Fe^{3+}/Fe^{2+} ratio with progressive flushes and a corresponding increase in waste-body temperature would seem to be indicative of bacterial oxidation. Furthermore, the large difference between the Fe^{3+}/Fe^{2+} ratio in flushes 10 and 12 might be due in part to the accelerated bacterial catalysis associated with a high-temperature microbe.

Figure 9 shows several samples of the data for solution samples extracted from the various waste-body locations at access ports as shown in Fig. 1(b). It is of interest to compare the waste-body pH and Cu levels and trends with the composite data of Fig. 7. The temperature optimum in Fig. 7 is indeed well correlated with a soluble Cu maximum or increasing Cu concentrations at most waste-body locations.

Duval-Sierrita rock sections having a mesh size of -4 to 6 (~ 3-5mm) were randomly selected from crushed waste material representative of the various column leaching tests and polished flat on one side. The polished samples were washed in distilled water and ultrasonically cleaned. Figure 10 illustrates the dissemination of FeS_2 and $CuFeS_2$ (phase regions) within the matrix. The veinlet structures were generally much more characteristic of the Sierrita material while the separate inclusions of Fig. 10(c) were the overwhelming feature of sulfide dissemination in the Kennecott-CMD waste material. Rock particles containing sections as shown typically in Fig. 10 were placed in sterilized culture media (Berry and Murr[8]) adjusted to a pH of 2.3 in separate flasks; some inoculated with bacteria and others maintained as sterile controls. The high-


Key
(4)
(3)
(2)
(1)
body
Temperature (°C)
Time (days)


O_2 consumption (Kg/day)
Time (days)


Key
(4)
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body
O_2(%)
Bacteria (cells/gm)
solution ave. (cells/cc)
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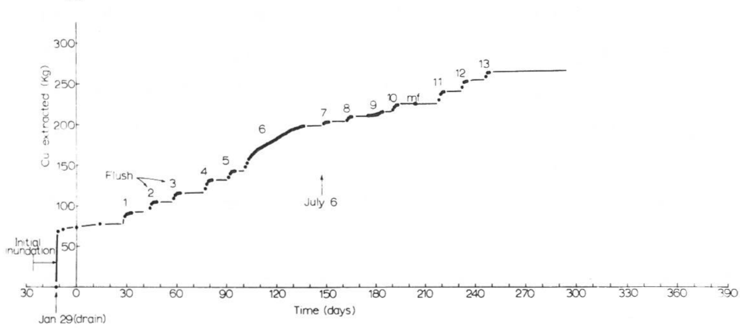
Time (days)


Cu extracted (Kg)
Initial inundation
Flush
July 6
mf
Jan 29 (drain)
Time (days)

temperature microbe inoculated was a Sulfolobus-like organism; maintained at 55°C during static leaching. Samples were removed periodically for examination in the scanning electron microscope and solution samples were withdrawn at the same time for analysis. Figure 11 illustrates some examples of the results obtained. In Fig. 11(a) and (b), the quantitative difference in Fe^{2+} and Fe^{3+} is observed to be consistent with those observed in the data of Fig. 8 (particularly flush 12). Furthermore, Fig. 12(a-d) clearly illustrate the role of bacteria in catalyzing reactions at the FeS_2 surfaces in the conversion of Fe^{2+} to Fe^{3+} as recently described in detail by Berry and Murr[8]. Figure 13(a) and (b) illustrate the morphologies observed for the high-temperature microbes and the Thiobacillus ferrooxidans.

While Fig. 13(a) and (b) are representative of bacterial attachment for each microorganism, it has been found, in contrast to the conclusions of Duncan and Drummond[9] for example, that Thiobacillus ferrooxidans do not generally attach to the mineral surface but when they do attach, they attach selectively; only on FeS_2, $CuFeS_2$, or other sulfide (e.g. Cu_2S) phase surfaces. This feature has already been demonstrated for the high-temperature microbe by Murr and Berry[10]. Moreover, bacterial attachment, while it does occur, is neither necessary nor sufficient for catalysis.

FIG. 7. Kennecott-Chino Mines waste rock leaching data. Temperature in the waste body, oxygen consumption (and levels) and bacterial population appear to be well correlated and indicative of the role of bacterial catalysis. The numbers in the graph showing cumulative copper extracted indicate periods of solution application or leaching (flushes). The initial solution inundation and draindown occurred at the point denoted "solution added" in the graph showing O_2% and bacterial population. The temperature-quench effects of the solution flushes, particularly at higher waste-body temperatures, are readily observable on comparing the top and bottom graphs. Note that the "body" designated in the keys is the waste rock.

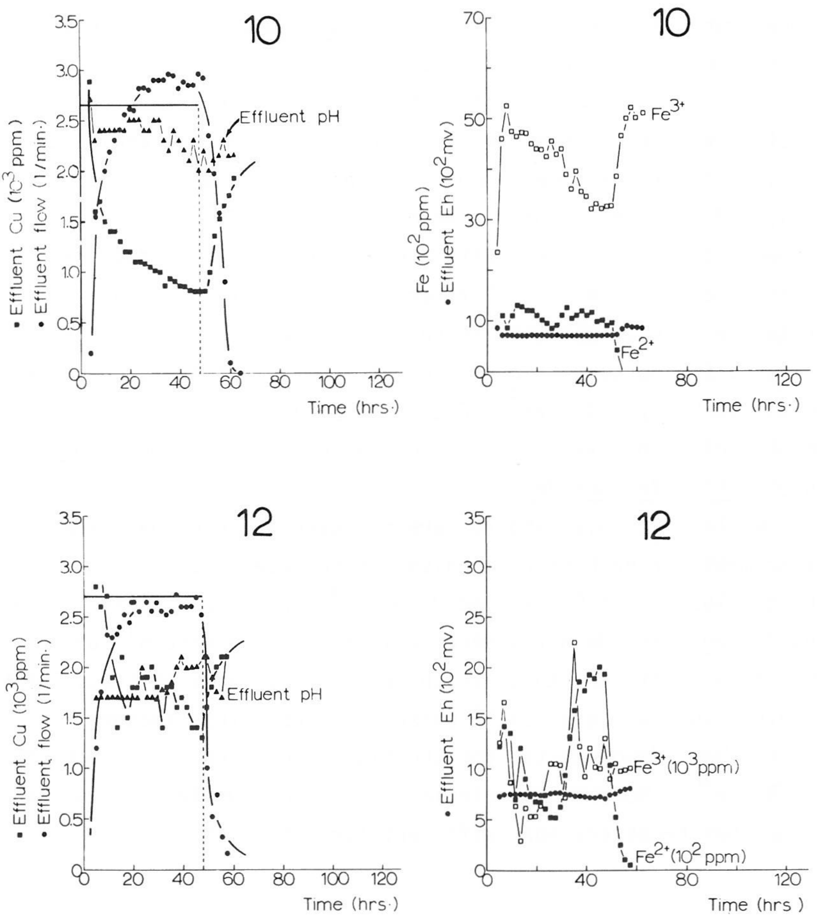


FIG. 8. Examples of solution flush data corresponding to Fig. 7. Each normal flush lasted 48 hrs. as shown. Effluent Cu and Fe^{2+} and Fe^{3+} were analyzed continuously along with Eh and pH at 2 hr. intervals.

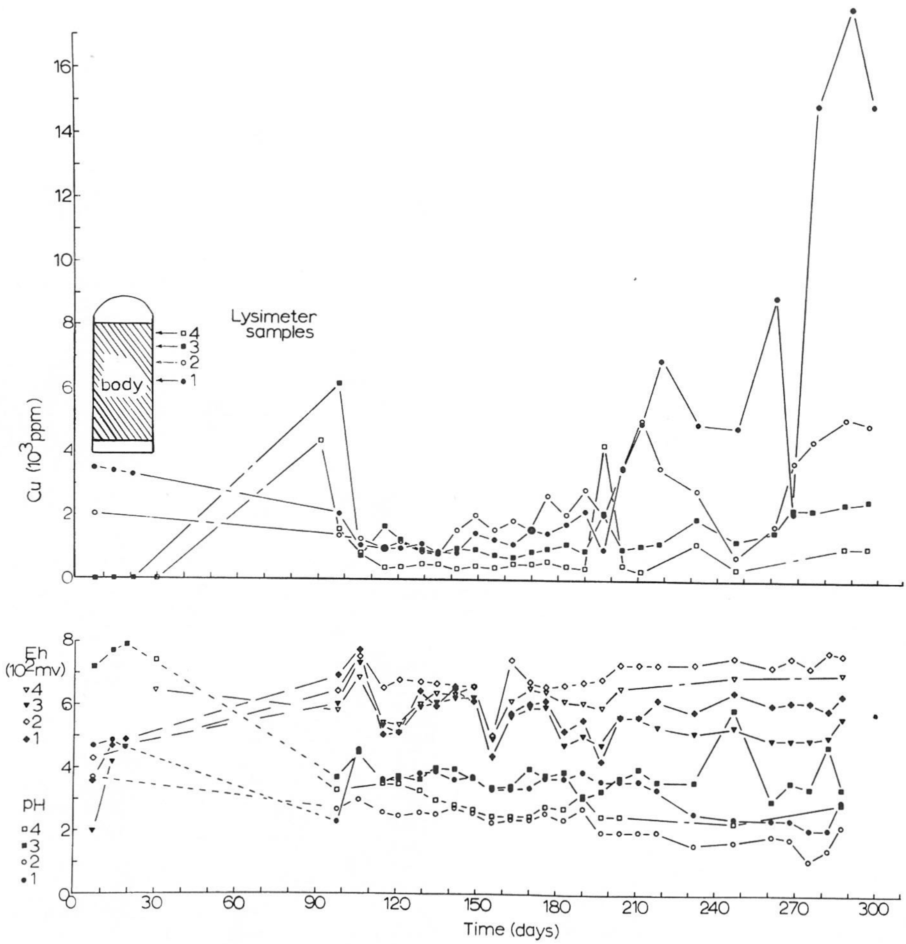


FIG. 9. Cu, Eh, and pH profiles within the Kennecott-CMD monzonite waste column (body). Solution samples were withdrawn from the lysimeters embedded within the waste rock (body) at locations indicated.

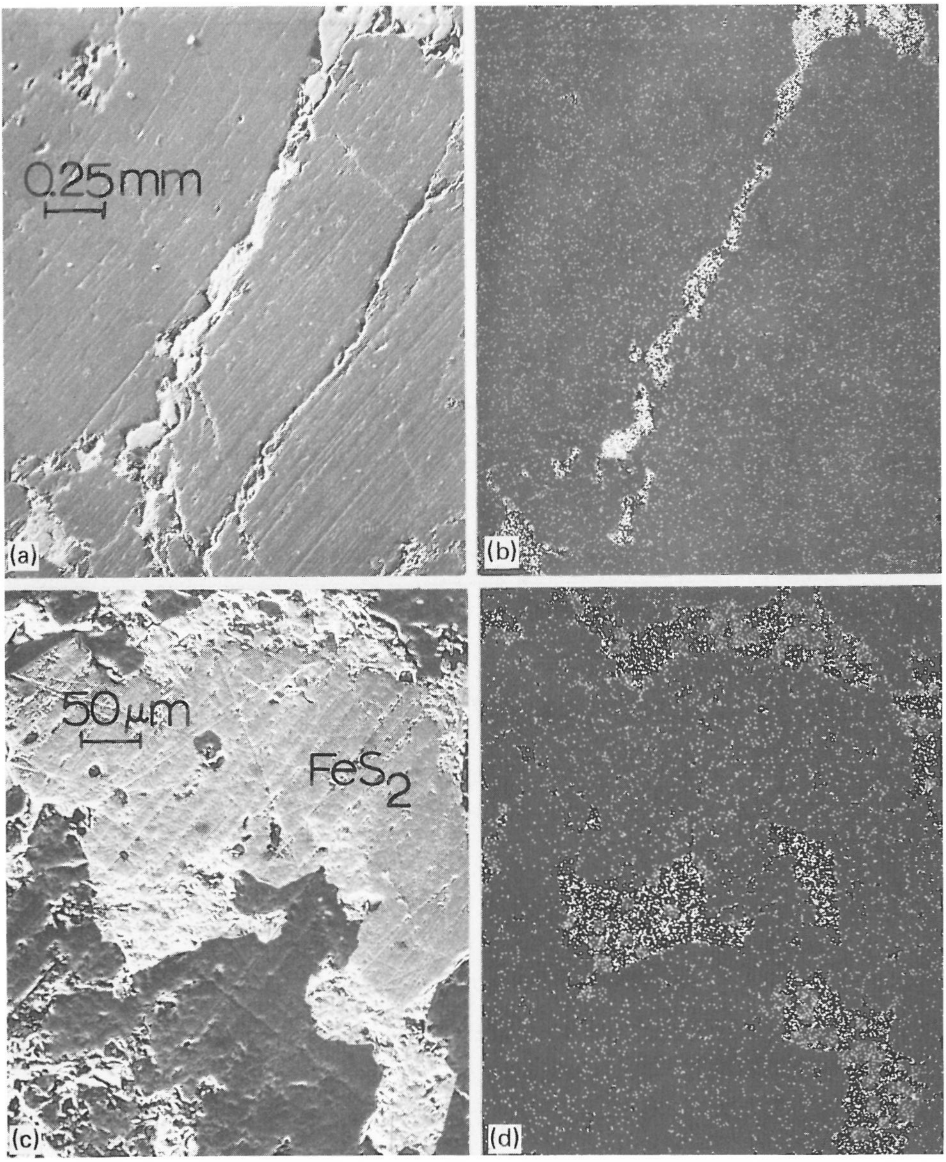
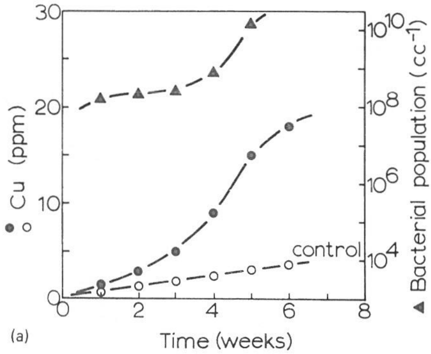
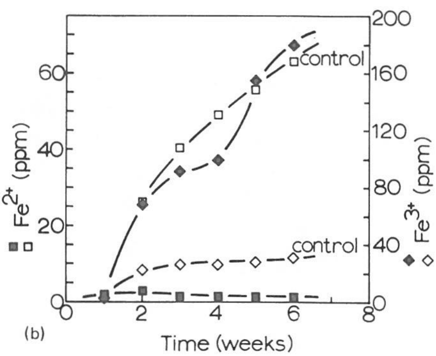


FIG. 10. Examples of sulfide phase dissemination in the Duval-Sierrita waste rock. (a) Veinlet typical of intermixed FeS_2 and $CuFeS_2$, (b) Cu-Fe X-ray map of (a), (c) Intermixed FeS_2 and $CuFeS_2$ phases, (d) Cu X-ray map of (c) showing $CuFeS_2$ regions in relation to FeS_2 regions.



(a)



(b)

FIG. 11. (a) Quantitative analysis of flask samples of Duval-Sierrita waste showing Cu in solution and a correlation of rate of solubilization with bacterial population; (b) Comparison of Fe^{2+} and Fe^{3+} levels in the same flask samples as (a). The control samples were not inoculated while the test samples were inoculated with a high-temperature, Sulfolobus-like microbe.

SUMMARY AND CONCLUSIONS

The results of this investigation, while incomplete, are encouraging. Certainly the results of Madsen and Groves[3] are illustrated by the present experiments, and the effect of oxygen concentration in the leach dump as it influences bacterial concen-

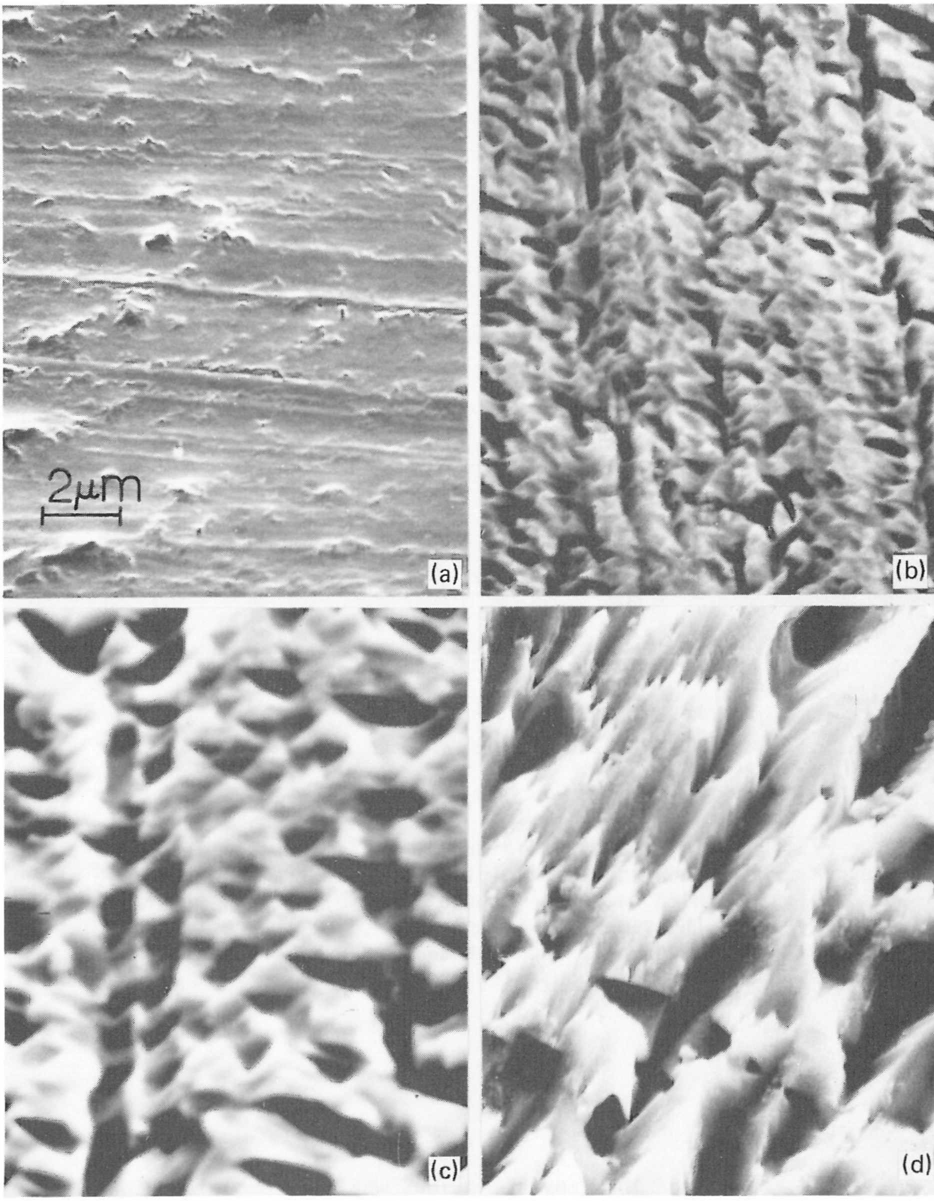


FIG. 12. Surface corrosion of FeS_2 in the high-temperature microbe solution at the same magnification. (a) Surface prior to leaching, (b) after 2 weeks, (c) after 3 weeks, (d) after 6 weeks.

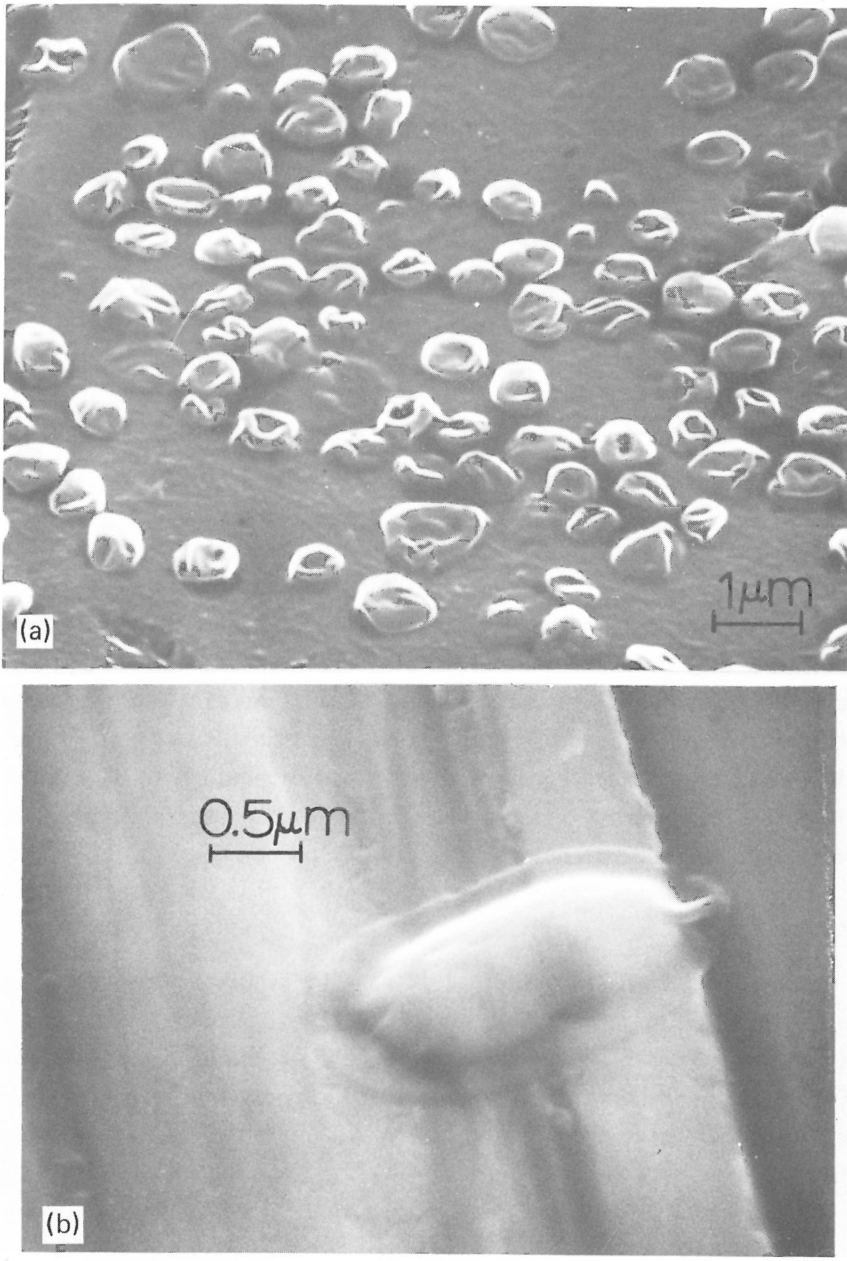


FIG. 13. (a) High-temperature microbes attached selectively to the surface of FeS_2; (b) Thiobacillus ferrooxidan on $CuFeS_2$ surface. Note flagellum and apparent biomatter upon which the organism appears to be situated.

tration is shown to be important. The correlation of temperature, bacterial concentration, and oxygen profiles in a large waste body have been demonstrated, and the utility of large waste-body leaching experiments has been overwhelmingly demonstrated. While the scaling data presented is incomplete, and not yet representative of the leaching of copper, a reasonably close correlation and overlap of data has been established in the neutralization of a waste body with regard to solution effluent pH and Eh. There is some hope that this is indicative of the fact that simple scaling laws can be effective in defining certain specific experimental leaching parameters. However, it is doubtful that those features of leaching which are strongly temperature dependent can be scaled adequately unless the temperatures can be controlled. It would seem unreasonable to expect that temperature profiles such as those shown in Fig. 7 could be duplicated in any laboratory column experiment. Finally, although the results, particularly those relating to bacterial concentration, provide additional insight into the role of bacteria in the leaching process, they also are indicative of certain areas which can yield even more meaningful results. In particular, it might be suggested that bacterial activity is temperature limiting (Fig. 7). If this is indeed true, then there are clearly new directions to be followed in enhancing leaching rates and efficiencies, and these must involve more detailed understandings of bacterial phenomena.

ACKNOWLEDGMENTS

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