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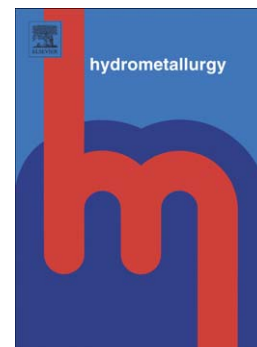
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The Effect of Sulphate Ions and Temperature on the Leaching of Pyrite.

III. Bioleaching

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Abstract

The effect of the total sulphate concentration on the rate of pyrite dissolution was investigated in batch, bioleach tests on a high-grade concentrate at 35°C, 50°C and 65°C.

Good microbial activity was achieved for concentrations up to 42 g/L sulphate. However, higher concentrations resulted in decreased ferrous oxidation kinetics, with the solution potential profiles showing “lag periods” at the highest sulphate concentration.

The bioleach results showed that the rate of pyrite dissolution decreased markedly above an average sulphate concentration of 55 g/L for all three temperatures, with the adverse effect on the dissolution kinetics being more pronounced at 50°C and 65°C. As a result of this, increased temperature has no beneficial effect on the rate of dissolution at concentrations of sulphate above 55 g/L.

High extents of pyrite dissolution (90% to 98%) were achieved at all three temperatures irrespective of sulphate concentration, with almost identical extents of sulphide oxidation.

Rates of pyrite dissolution under bioleaching conditions agree well with those derived under similar conditions from abiotic dissolution experiments and electrochemical measurements. This important observation confirms the so-called indirect mechanism

for the bioleaching process in the case of pyrite and also that the mixed potential model can be used to quantitatively describe both abiotic and biotic dissolution of pyrite in acidic solutions.

Keywords: bioleaching, dissolution, potential, pyrite, sulphate, temperature

1. Introduction

Chalcopyrite (CuFeS_2) is one of the most refractory copper sulphide minerals especially in acidic ferric sulphate solutions at ambient temperatures. Heap bioleaching at high temperatures presents an opportunity to achieve improved copper recovery from low-grade, primary copper sulphide ores with high chalcopyrite content.

This application requires operation in such a way that the heap temperature is increased sequentially from mesophilic (ambient to 45°C), through moderately thermophilic (45°C to 60°C) to thermophilic levels ($> 60^\circ\text{C}$), where it is maintained for some time (Dew *et al.*, 2011). The success of such a strategy will depend, amongst other things, on sufficient levels of available sulphide sulphur (S^{2-}) in the ore, as well as adequate oxidation to sulphate in relatively fast, exothermic reactions. Pyrite (FeS_2), if available, serves as a suitable source of sulphide sulphur in the ore; however, the oxidation of the mineral produces sulphate ions which will build-up in the leaching solution.

Zheng *et al.* (1986) reported that the rate of pyrite oxidation decreased with increased total sulphate concentration, regardless of whether sodium sulphate or sulphuric acid was used. Their finding is also in agreement with earlier reported work (Sasmojo, 1969; Smith and Shumate, 1970). The former authors ascribed this to inhibited

electron transfer and thereby limiting adsorption to the mineral surface, because of iron(III) sulphate complexation. In another study, the rate of oxidation by hydrogen peroxide in acidic sulphate solutions was found to decrease with increasing sulphate concentration (Antonijevic *et al.*, 1997).

This paper, which forms Part III of a series of papers (Nicol *et al.*, 2012), presents the results obtained from an investigation into the effect of sulphate ion concentration on the rate of pyrite dissolution under batch, bioleaching conditions at 35°C, 50°C and 65°C.

2. Experimental

2.1. Pyrite Sample

A sample of a high-grade, pyrite concentrate was wet-screened to produce about 1.5 kg of material at +25-38 µm. This was air-dried, de-lumped through a 300 µm screen, blended and finally divided into 100 g samples with a small, rotary splitter.

Chemical analysis showed 0.080% Cu, 44.4% Fe, 49.1% S²⁻, 0.16% S⁰ and 0.66% Si from which a pyrite content of 93.5% has been estimated.

2.2. Inoculum Build-Up and Leaching Procedures

Inoculum solutions containing 35°C, 50°C and 65°C microbial cultures were prepared by introduction of concentrated biomass (from active, maintenance cultures) into nutrient solution containing 0.2 g/L ammonium sulphate, 0.1 g/L magnesium sulphate and 0.1 g/L potassium hypophosphate. Thereafter, a test reactor was filled with appropriate amounts of nutrient solution, aluminium sulphate (if applicable), additional magnesium sulphate (if applicable), ferrous sulphate and 250 mL of inoculum to produce 5 L of a solution. Tests were conducted at initial sulphate

concentrations of 3.9 g/L, 42.1 g/L, 78.5 g/L and 119 g/L. All test solutions initially contained 1 g/L iron.

The working solution was saturated with an air / carbon dioxide mixture (21% v/v O₂; 0.35% v/v CO₂) and mixed by agitation (Φ 116 mm, 6-blade, Rushton turbine; 500 rpm) in a sealed reactor fitted with four baffles and a water-cooled condenser.

Temperature control ($\pm 0.5^\circ\text{C}$) was achieved by means of a heated element submerged in the solution. Once the set point temperature was reached, the working solution was adjusted with small amounts of (98%) concentrated sulphuric acid to render an initial acidity of pH 1.8.

A mass of 100 g of solids was introduced into the working solution as soon as the solution potential increased above 600 mV, which constituted the start of the leaching test. In two tests, namely 50°C (119 g/L sulphate) and 65°C (119 g/L sulphate), the solids were introduced at lower potentials, i.e. 562 mV (50°C) and 546 mV (65°C), due to the fact that the solution potential did not increase much above these values over the monitored period.

Process parameters such as acidity (pH), solution potential (redox), temperature, total soluble iron, inlet gas flow rate, as well as the carbon dioxide and oxygen concentrations of the inlet and outlet gasses were monitored daily during both inoculum build-up and leaching phases.

After completion of each test, the bioleach slurries were filtered and the filter cakes washed with distilled water. The cakes were then air-dried, weighed, de-lumped with a 300 μm screen, bagged and submitted for chemical analysis.

All solution potential values have been reported against a 3 M KCl, Ag / AgCl reference electrode unless otherwise stated.

3. Results and Discussion

3.1. *Inoculum Build-Up Phase*

Figures 1 to 3 summarize the solution potential profiles at initial sulphate concentrations of 3.9, 42.1, 78.5 and 119 g/L.

35°C

It can be seen that at the lower two sulphate concentrations, good microbial performance in terms of iron-oxidizing capacity has been achieved. This is reflected by rapid increases in the respective potentials to 689 mV (3.9 g/L sulphate) and 664 mV (42.1 g/L sulphate) after only 1 day of operation. In contrast, the test with 78.5 g/L sulphate resulted in a much slower rate of ferrous oxidation with the potential reaching 630 mV after 4 days while the test at the highest sulphate concentration shows an initial “lag period”, with the potential increasing slowly from 398 mV to 448 mV over 12 days, after which it rapidly increased to 673 mV.

Therefore, although high potentials were eventually achieved in all tests, it is evident that the rate of microbial-assisted ferrous oxidation at 35°C is adversely affected by the sulphate concentration.

50°C

The tests at the lowest three sulphate concentrations all show good microbial performance with the respective potentials increasing rapidly to high values of 692 mV (1 day), 664 mV (0.2 days) and 652 mV (1 day), respectively.

The potential of the test at the highest sulphate concentration increases from 373 mV to 484 mV over the first day of operation showing different initial microbial behaviour than that conducted at 35°C. Thereafter, the potential increases at a much slower rate to 562 mV over the next 7 days (similar to the 35°C test). However, unlike in the case of the 35°C test, no sudden increase in potential is observed at a later stage

and this may be due to the fact that the appropriate or critical number of active cells had not yet been established to facilitate faster kinetics of ferrous oxidation.

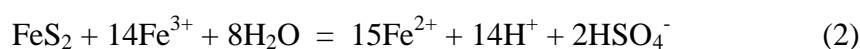
65°C

At the lower sulphate concentrations, rapid increases in potential to 656 mV and 660 mV were observed after 1 day while that at 78.5 g/L sulphate required 3 days to reach 615 mV. At the highest sulphate concentration, the potential increased rapidly from 403 mV to 509 mV over the first day of operation after which it increased at a much slower rate to 546 mV over the next 6 days. This behaviour is very similar to that observed at 50°C.

3.2. *Leaching Phase*

3.2.1. *Solution Potential Profiles*

Figures 4 to 6 show the solution potential profiles during the leaching phase for the tests at various initial sulphate concentrations and temperatures. The starting potentials, which are favourable for pyrite dissolution, decrease sharply on introduction of the solids. This is caused by the reduction of ferric to ferrous ions at the mineral surface according to the reactions:



In these and all subsequent equations, iron(III) is written as Fe^{3+} and iron(II) as Fe^{2+} for simplicity recognizing that the predominant species are not the aquo-ions but the the sulfato-complexes.

The above reaction can also be presented by the coupled, half-cell reactions:

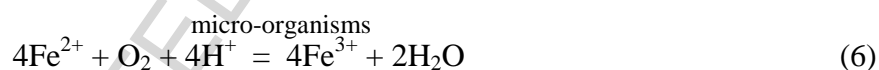




The relative contributions of the two anodic reactions depends on the conditions and the potential., Biological oxidation of elemental sulfur by a non-electrochemical process is also likely which will influence the overall stoichiometry.

Ferrous ion is a product of both the anodic oxidation of the mineral and the cathodic reduction reaction. Thus, the decrease in potential is a result of a decrease in ferric-to-ferrous ion ratio due to the formation of ferrous ion from both half-cell components and reduction of the ferric ion concentration.

Thereafter, the potential for each test increases progressively due to microbial-assisted oxidation of ferrous ions (Equation 6) to reach 700 to 750 mV for the tests at 35°C, 600 to 650 mV for those at 50°C and 625 to 700 mV for those at 65°C.



At 50°C and 65°C, the potential profiles at the two higher sulphate concentrations show a distinct “lag period” which seems to increase with increasing sulphate concentration. The results also show the adverse effect of increased sulphate concentration on the rate of biologically catalysed oxidation of ferrous ions as is evident from the slopes of the potential profiles. The mechanistic reasons for this reduced bacterial activity are not known and not the subject of this paper.

3.2.2. pH Profiles

The variations in pH during each test were approximately similar and one such profile during an experiment is shown in Figure 7. The starting acidity (pH 1.8) for all the tests decreases with time as pyrite is oxidized according to the overall reaction:



The rate of increase in acidity was found to be greater at the lower sulphate concentrations than at the higher concentrations at all three temperatures. This may be ascribed to a decrease in the rate of oxidation of pyrite and / or greater extent of formation of the bisulphate anion within the range pH 1 to pH 2 at higher total sulphate concentrations.

3.2.3. *Solids Residue*

Table 1 summarizes the data obtained from the residues of each test. The extents of iron and sulphide sulphur dissolution were calculated from the mass loss obtained from 1) the weight and 2) analysis for Si as a tie-element with the assumption that negligible Si dissolution occurred during leaching. A comparison of the head and residual Si values show that this is indeed the case.

These results show that, despite the rate of pyrite dissolution being adversely affected by increased sulphate concentration, high extents of pyrite dissolution are still feasible over prolonged leaching periods.

The extents of sulphide dissolution are similar to those of iron which suggest that oxidation of sulphide produces a soluble product as opposed to elemental sulphur which would report to the residue. It is assumed on the basis of the reasonable agreement between the extent of pyrite dissolution and that calculated from the oxygen utilization on the basis of reaction (5) that the product of oxidation is sulphate. Thus, one can also conclude that increased sulphate concentration does not alter the oxidation pathway of sulphide sulphur to sulphate.

3.2.4. *Rates of Pyrite Dissolution*

The rate of dissolution of pyrite was generated from analysis of the oxygen in the gas entering and leaving the reactor assuming the stoichiometry of Equation 5. This data

was converted to extents of reaction by comparison of the calculated final extent of dissolution (from gas analysis) with that obtained by analysis of the head and residue samples for iron. The respective curves for the dissolution of pyrite for the tests at different temperatures are depicted in Figures 8 to 10.

At 35°C, the trends in the rate of dissolution for the various tests are similar to those of the corresponding solution potential profiles. Greater than 90% dissolution was achieved in all cases. While the final extent of pyrite dissolution does not appear to be affected by increased sulphate concentration at this temperature, the rate is lower at the two highest sulphate concentrations compared to that at the lower sulphate concentrations.

At 50°C, the extent of dissolution was greater than 94% in all cases except for the experiment at the highest sulphate concentration for which only 66% dissolution was achieved after 20 days. The curve for the lowest sulphate concentration appears to be anomalous compared to the others as a result of apparent slower initial kinetics. This is accompanied by a slower increase in solution potential over the range 475 to 600 mV when compared to those at higher sulphate concentrations (see Figure 5). Again, the final extents of dissolution for the three tests with the lowest concentrations of sulphate are not affected by an increase in sulphate concentration. However, it is clear from the decreasing slopes of the leach curves that the rate of pyrite dissolution does decrease with increasing sulphate concentration at this temperature.

At 65°C, the tests at the lower sulphate concentrations show similar rates and extents of dissolution and it is clear that the rate of pyrite dissolution decreases with increasing sulphate concentration at this temperature. The dissolution profiles are all in agreement with the corresponding solution potential profiles with obvious “lag periods” at the high sulphate concentrations.

Rates of pyrite dissolution (in mol Fe cm⁻².s⁻¹) were calculated from the difference in the extents of dissolution over the same range of 20% to 64% for each of the three temperatures. The results are presented in Table 2 together with average values for other relevant parameters and also graphically in Figure 11. The detrimental effect of high sulphate ion concentrations on the rate of dissolution is apparent at all temperatures. As mentioned above, the lower rate at the lowest sulphate concentration at 50°C appears to be related to the lower potentials attained in these experiments during the leaching phase as evident in Figure 5. The reason for this is not known. An unexpected result is that the rates of pyrite dissolution at 65°C are significantly lower than those at the lower temperatures at high sulphate concentrations. This has implications for the desirability of operating at high temperatures in the presence of high sulphate concentrations.

3.2.5. Comparison with Electrochemical and Abiotic Dissolution Data

It is interesting to compare the above rates of pyrite oxidation under bioleach conditions with those estimated from the electrochemical methods and controlled potential dissolution experiments under abiotic conditions as described in Parts I and II. It is not possible to exactly match the conditions used in the three techniques but Table 3 summarizes some of the data that allows for a reasonable comparison. Thus, under relatively similar conditions in the fourth row of the table, the rates are very comparable. The higher abiotic rates at 50°C can be ascribed to the higher potentials in these experiments while the lower abiotic rate in the third row is due to a lower potential and the higher abiotic rate in the last row to a higher potential.

The agreement of the rates obtained under bioleaching and abiotic conditions is satisfactory given that the source of the pyrite was different for the two sets of data.

This is further confirmation of the applicability of the so-called indirect mechanism for the bioleaching process in which oxidation of the mineral by iron(III) is the rate determining step.

4. Conclusions

Good microbial activity has been achieved at the lower concentrations (3.9 and 42 g/L) of sulphate resulting in relatively fast kinetics of ferrous oxidation by the various cultures. Increased sulphate concentration of up to 79 g/L has an adverse effect on microbial activity resulting in a decrease in the rate of ferrous oxidation at 35°C and 65°C. At very high sulphate concentrations, the solution potential profiles at all temperatures show the presence of “lag periods” due to (severely) reduced ferrous oxidation kinetics. This may be an indication of inhibition of the growth of all cultures by sulphate ions.

The rate of pyrite dissolution decreases with increasing sulphate concentration at all temperatures with the adverse effect on the kinetics being more pronounced at the two higher temperatures. The results show that a significant decrease in the dissolution kinetics could be expected above an average sulphate concentration of 55 g/L for all three temperatures. Increased temperature does not increase the rate of pyrite dissolution at high (greater than about 50 g/L) sulphate concentrations.

High extents of pyrite dissolution, which range from 90% to 98%, have been achieved at all three temperatures irrespective of sulphate concentration. This suggests that, despite the slower kinetics, effectively complete dissolution of fine-milled, liberated material could be achieved over prolonged leaching periods. Analysis of the residues has confirmed that oxidation of sulphide sulphur results in sulphate as the

predominant product. Increased sulphate concentration does not seem to change the extent of oxidation of sulphide sulphur.

Rates of pyrite dissolution under bioleaching conditions agree well with those derived under similar conditions from abiotic dissolution experiments and electrochemical measurements. This important observation confirms the so-called indirect mechanism for the bioleaching process in the case of pyrite and also that the mixed potential model can be used to quantitatively describe both abiotic and biotic dissolution of pyrite in acidic solutions.

5. References

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Figures

Figure 1 Solution potentials during inoculum build-up phase at 35°C

Figure 2 Solution potentials during inoculum build-up phase at 50°C

Figure 3 Solution potentials during inoculum build-up phase at 65°C

Figure 4 Solution potentials during leaching phase at 35°C

Figure 5 Solution potentials during leaching phase at 50°C

Figure 6 Solution potentials during leaching phase at 65°C

Figure 7 pH values during leaching phase at 35°C

Figure 8 Dissolution of pyrite at 35°C

Figure 9 Dissolution of pyrite at 50°C

Figure 10 Dissolution of pyrite at 65°C

Figure 11 Experimental rates of pyrite dissolution

Tables

Table 1 Mass Loss and Extent of Pyrite Dissolution

Table 2 Experimental Rates of Pyrite Dissolution

Table 3 Comparison of Bio- and Abiotic Rates of Dissolution

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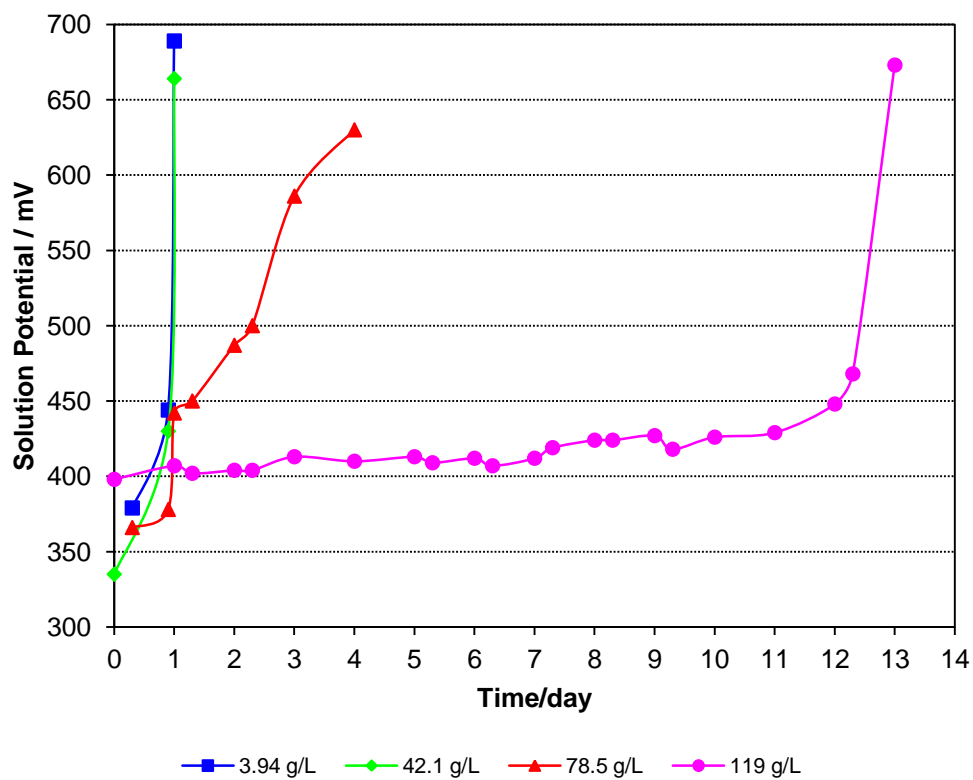


Figure 1

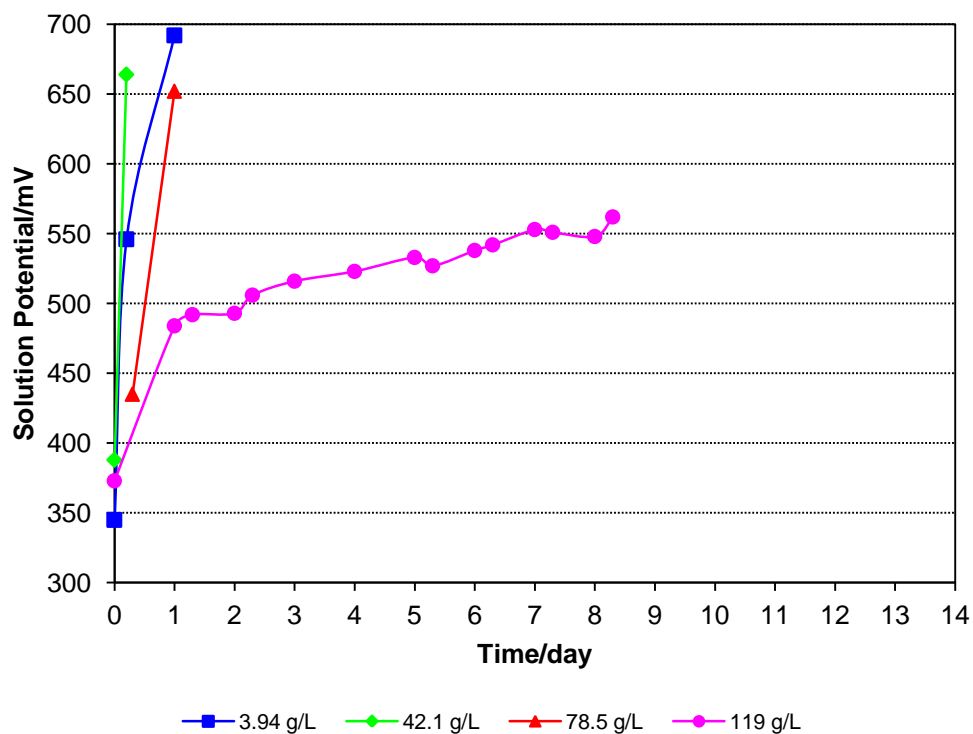


Figure 2

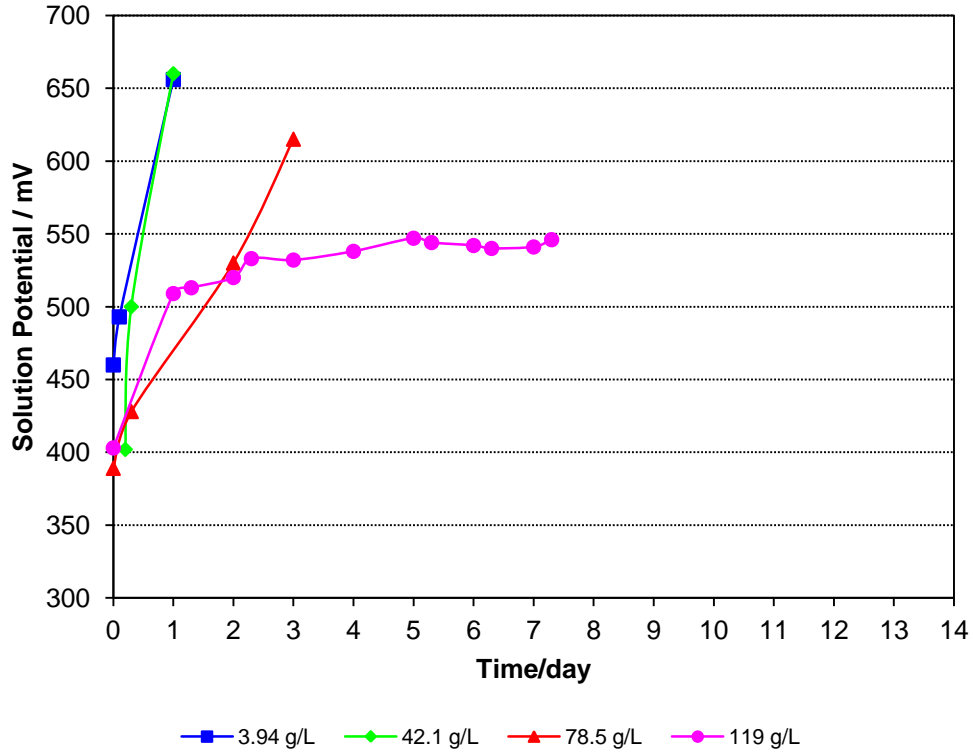


Figure 3

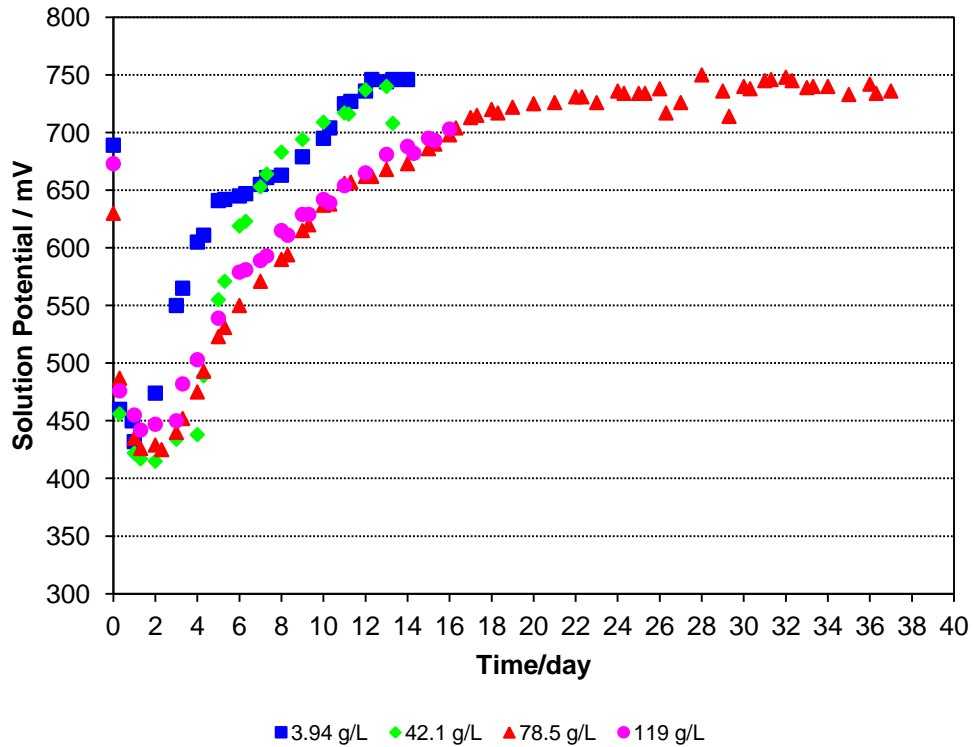


Figure 4

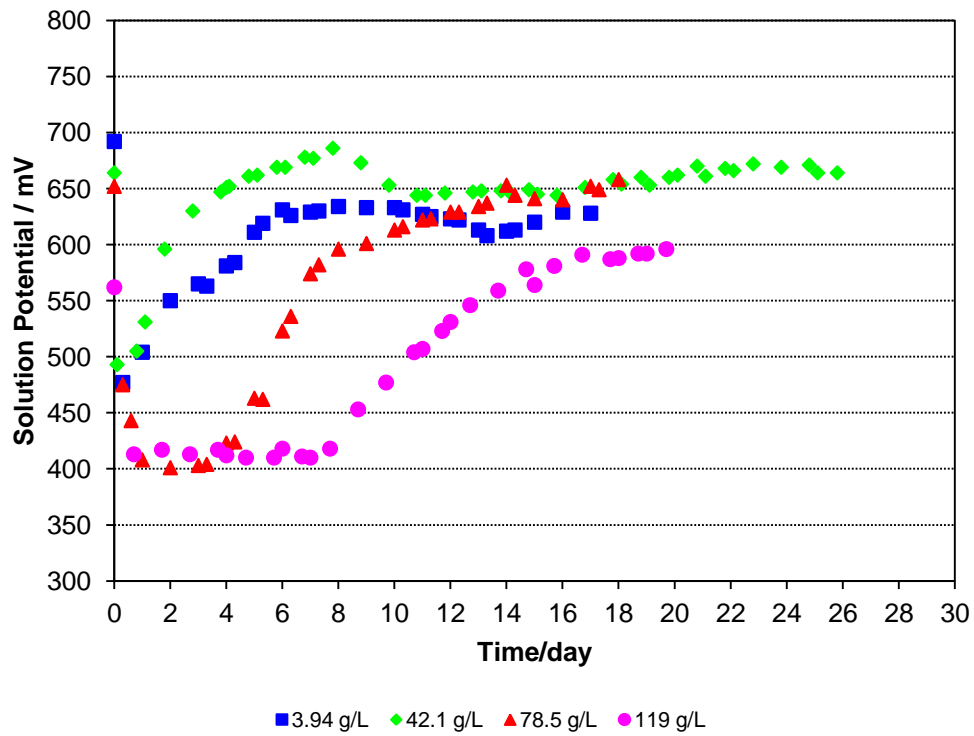


Figure 5

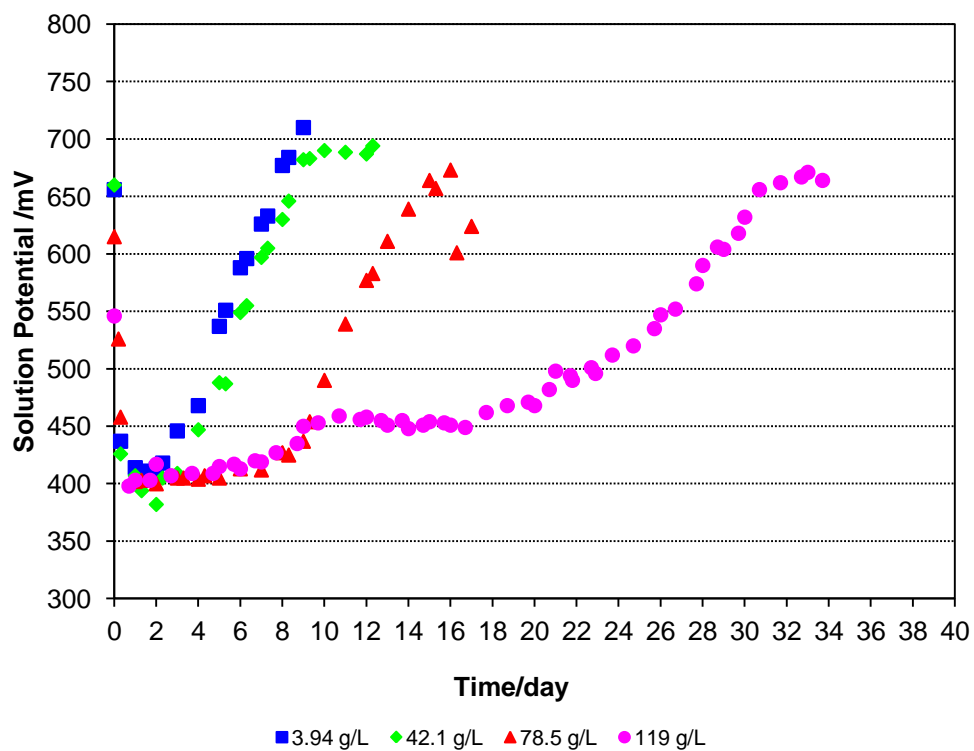


Figure 6

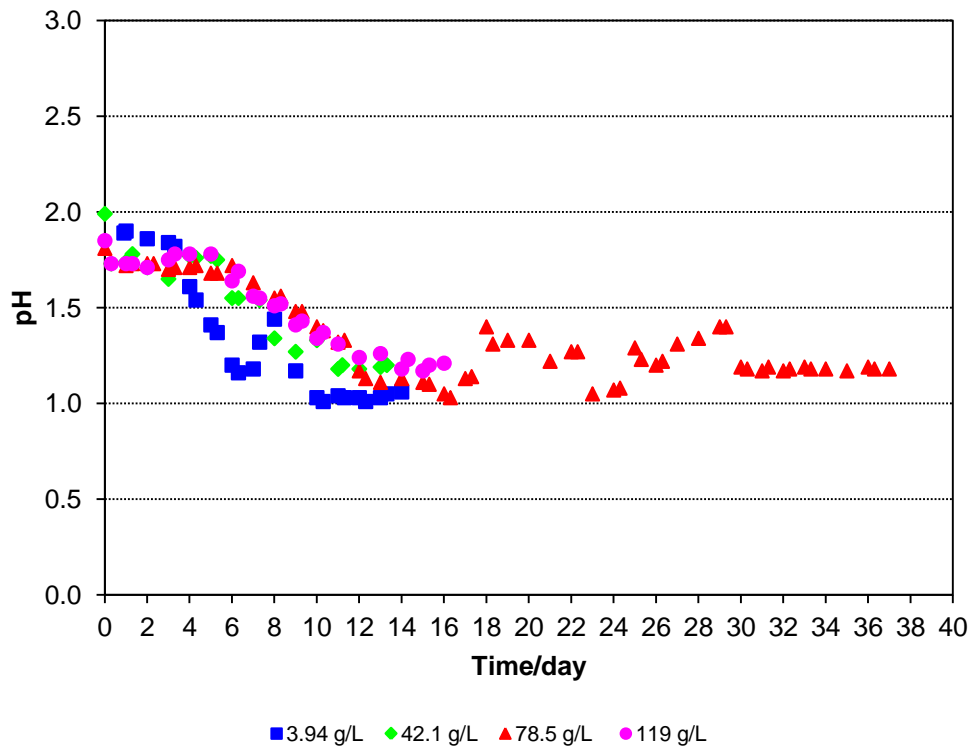


Figure 7

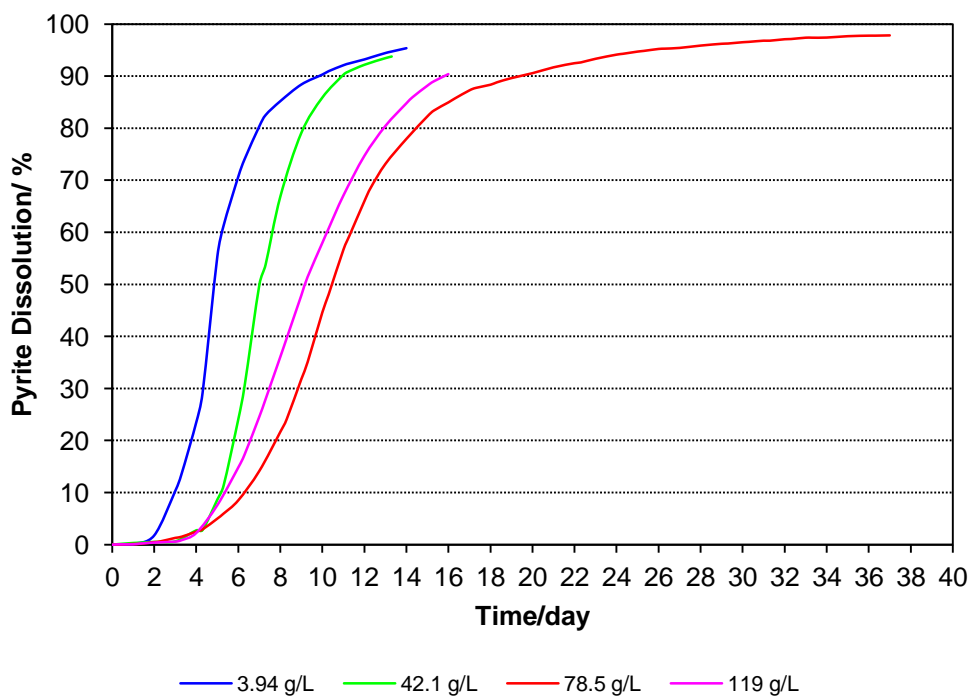


Figure 8

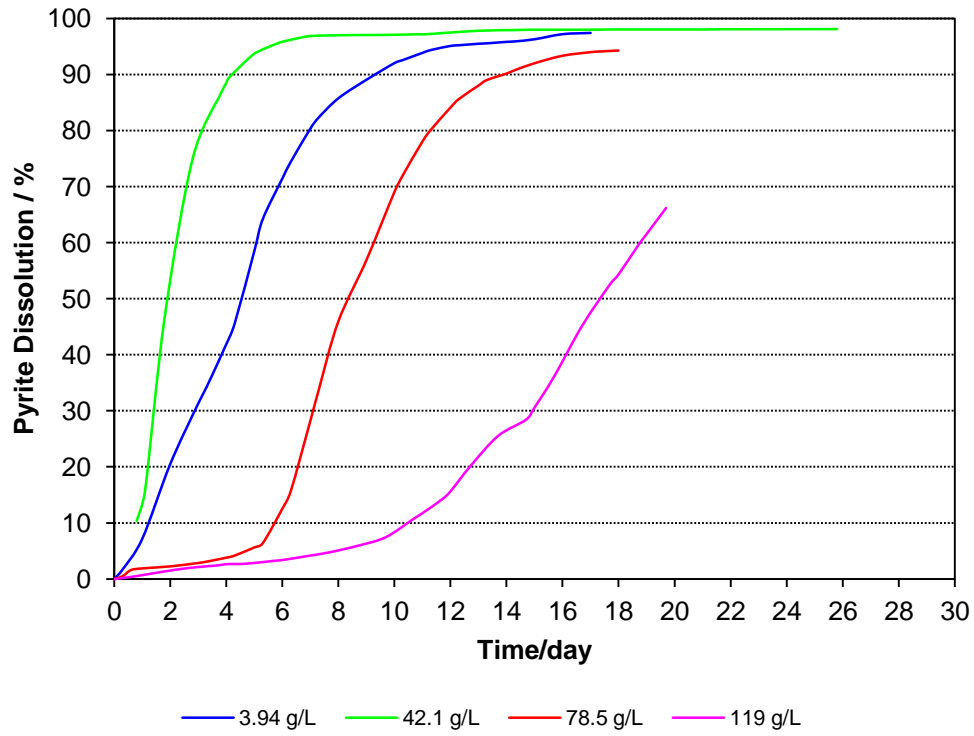


Figure 9

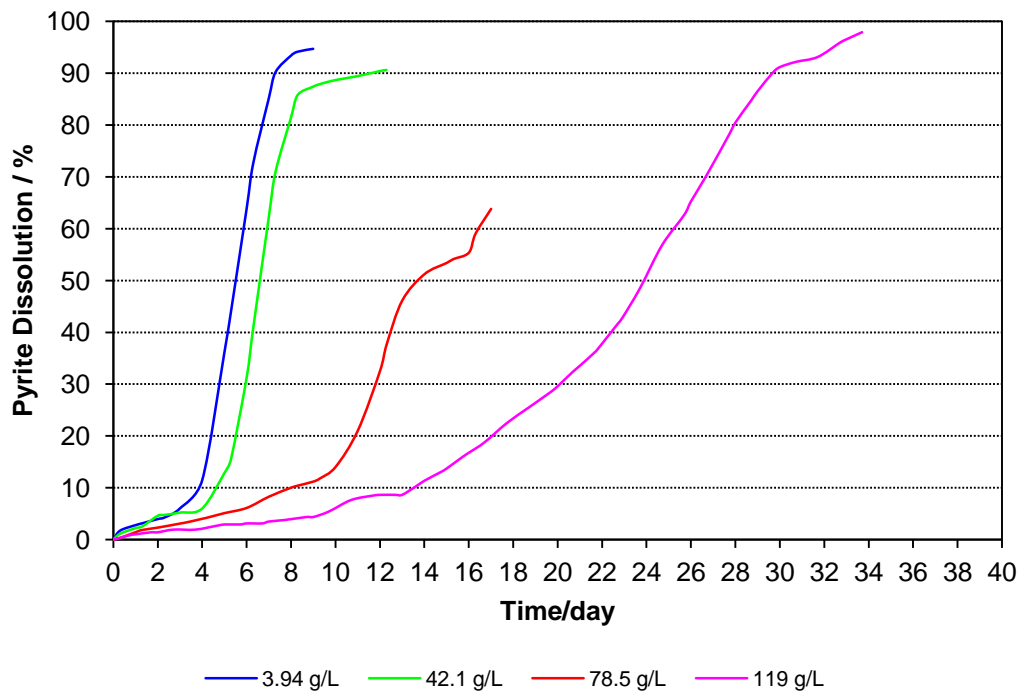


Figure 10

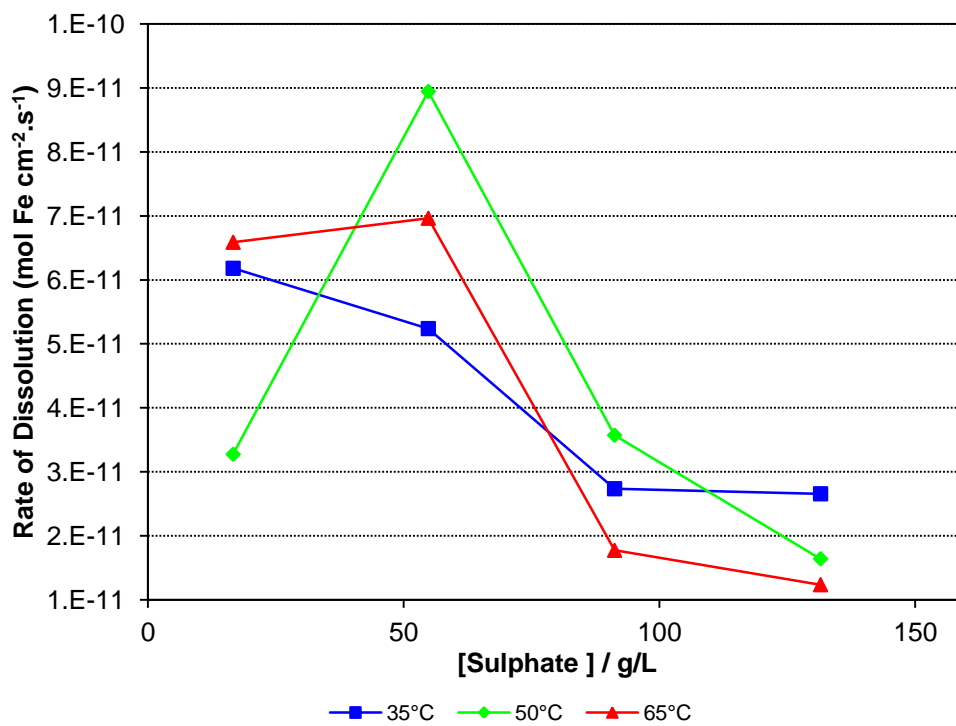


Figure 11

Table 1: Mass Loss and Extent of Pyrite Dissolution

Temp °C	[Sulfate] ₀ g/L	Time days	Mass Loss	FeS ₂ Diss. (%)		Sulphide Diss. (%)	
			%	Mass	Si [#]	Mass	Si [#]
35	3.94	14	92.4	95.3	95	96	96
	42.1	13	90.9	93.8	94	94	94
	78.5	37	94.7	97.8	98	98	98
	119	16	86.9	90.4	90	91	91
50	3.94	17	94.4	97.4	98	99	99
	42.1	26	95.4	98.1	98	100	100
	78.5	18	87.6	94.3	94	98	98
	119	20	63.6	66.2	64	68	65
65	3.94	9	90.6	94.7	94	100	99
	42.1	12	86.3	90.6	90	97	97
	78.5	17	60.8	64.4	65	70	71
	119	34	94.9	97.9	98	99	99

Si as tie-element (assuming negligible Si dissolution)

Table 2: Rates of Pyrite Dissolution

Temperature °C	[Sulfate] ¹ g/L	pH ²	E _{Soln} ³ mV	E ^{Φ4} mV	Iron (g/L) ⁵			10 ¹¹ x Rate Diss. mol Fe cm ⁻² .s ⁻¹
					[Fe(T)]	[Fe(II)]	[Fe(III)]	
35	16.6	1.45	817	672	4.69	0.020	4.67	6.18
	54.8	1.47	842	646	4.69	0.003	4.69	5.24
	91.2	1.35	823	638	4.69	0.004	4.69	2.74
	132	1.46	816	635	4.69	0.005	4.69	2.66
50	16.6	1.35	784	684	4.69	0.19	4.50	3.27
	54.8	1.41	779	656	4.69	0.085	4.61	8.94
	91.2	1.42	779	648	4.69	0.064	4.63	3.57
	132	1.41	770	646	4.69	0.081	4.61	1.64
65	16.6	1.20	740	695	4.69	1.51	3.18	6.59
	54.8	1.43	752	663	4.69	0.45	4.24	6.96
	91.2	1.46	777	654	4.69	0.15	4.54	1.77
	132	1.46	698	651	4.69	1.44	3.25	1.24

- 1) Arithmetic average for the range
- 2) Arithmetic average for the corresponding proton concentrations
- 3) Arithmetic average for the range (V versus SHE)
- 4) Formal potential for the Fe(III)/Fe(II) redox couple (V versus SHE). Data from Part I.
- 5) The total iron concentration calculated from the extent of pyrite dissolution (as obtained from oxygen off-gas analysis after reconciliation with the solids (head and residue) analysis). Ferric and ferrous ion concentrations in the bulk solution were calculated from the total iron concentration, average bulk solution potential and reported formal potential for the Fe(III) / Fe(II) redox couple.

Table 3: Comparison of Bio- and Abiotic Rates of Dissolution

Temperature °C	[Sulfate] g/L	E_{Soln} mV	$10^{11} \times \text{Rate}$ $\text{mol cm}^{-2} \text{s}^{-1}$
50 ¹	42 (55)	835 ³ (779)	32.3 (8.9)
50 ¹	119 (132)	815 ³ (770)	26.9 (1.6)
35 ²	40 (55)	800 (842)	2.8 (6.2)
35 ²	40 (55)	850 (842)	8.1 (6.2)
35 ²	120 (132)	850 (816)	7.2 (2.7)

1) Derived from electrochemical data (Part II)

2) Dissolution in controlled potential agitated reactors (Part II)

3) Pyrite electrode potential

Bioleaching data in parentheses

Highlights – Part III

- Good microbial activity has been achieved at low concentrations (3.9 and 42 g/L) of sulphate resulting in relatively fast kinetics of ferrous oxidation by the various cultures.
- Increased sulphate concentration of up to 79 g/L has an adverse effect on microbial activity resulting in a decrease in the rate of ferrous oxidation at 35°C and 65°C.
- The rate of pyrite dissolution decreases with increasing sulphate concentration at all temperatures with the adverse effect on the kinetics being more pronounced at the two higher temperatures.
- Increased temperature does not increase the rate of pyrite dissolution at high (greater than about 50 g/L) sulphate concentrations.
- High extents of pyrite dissolution, which range from 90% to 98%, have been achieved at all three temperatures irrespective of sulphate concentration.
- Rates of pyrite dissolution under bioleaching conditions agree well with those derived under similar conditions from abiotic dissolution experiments and electrochemical measurements.
- The so-called indirect mechanism for the bioleaching process in the case of pyrite has been confirmed.