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COMPARISON OF CHEMICAL AND BIOLOGICAL LEACHING OF SULFIDE TAILINGS

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Abstract: Sulphidic tailings from Finnish Hitura nickel mine and Pyhäsalmi multi-metal mine were leached using sulphuric acid and bioleached. The aim was to recover minor amounts of valuable Cu, Ni, Zn and Mn. Both tailings consisted mainly of iron and magnesium-containing minerals and acid neutralizing minerals. The solution after chemical leaching tests contained mostly iron and magnesium, in Hitura up to 11 g/dm³ Fe and 38 g/dm³ Mg while in Pyhäsalmi 8–9 g/dm³ Fe and 4 g/dm³ Mg. Amount of these metals was 20–100-fold larger than amount of valuable metals, which were typically 100-300 mg/dm³. Problems in chemical leaching were high consumption of acid and poor selectivity. Bioleaching using iron and sulphur oxidizing bacteria was more selective towards the valuable metals. Both in leaching and bioleaching the high concentration of iron and magnesium in solution will make metals recovery challenging.

Keywords: sulfide tailings, chemical leaching, bioleaching, selectivity

Introduction

The tailings from mining and extraction contain metals whose dissolution is an environmental risk. In Finland, mining activities utilise mainly sulphidic ores, containing only minor amounts of neutralizing carbonate minerals. This leads to a situation where sulphidic tailings may oxidize spontaneously in tailing ponds and generate Acid Rock Drainage (ARD). Metal rich in the ARD may leak from pond to surrounding environment, causing heavy stress to large areas (Moncur et al. 2005). The tailings are also considered as sources of valuable metals (Xie et al. 2005, Antonijevic et al. 2008). Yearly in Finland about three teragrams (Tg, million tons) of mining wastes containing valuable metals are stored at dump sites. Currently only 20% of

these wastes are reused. Especially tailings from mineral processing contain valuable metals but they appear in so small concentrations that processing them would be difficult with traditional methods.

Bioleaching is a rapidly growing technique for extraction of various metals from ores and mineral concentrates. Traditionally industrial scale applications have been related to copper extraction from sulphidic minerals, but lately promising results have also been acquired with recovery of gold, uranium, nickel and zinc. As bioleaching is relatively low-cost technology, it may also be utilised for poor ores and mine site wastes, like tailings (Bosecker 1997, Rawlings 2002). Bioleaching is based on certain bacteria's ability to oxidize ferrous iron (Fe²⁺) to ferric iron (Fe³⁺), and/or to oxidize reduced sulphur compounds to sulphuric acid (H₂SO₄). Produced Fe³⁺ and H⁺ can both attack sulphide minerals and release valuable metals to surrounding solution. Due to generation of sulphuric acid, pH of the surrounding solution decreases rapidly to values like 1–3, maintaining valuable metals in oxidized and solubilised form suitable for recovery process (Sand et al. 2001).

This work studies hydrometallurgical and biological methods for processing sulphidic tailings. It focuses on recovery of copper, nickel and zinc from sulphide tailings. Hydrometallurgical and bioleaching test series were done for two mineral processing tailings, which had been taken from the Finnish Hitura and Pyhäsal mine. Hitura is a nickel and Pyhäsalmi is a multi-metal mine, and both are located in North Ostrobothnia. Hitura produces nickel concentrate, which contains small amounts of copper, cobalt, platinum and palladium. Pyhäsalmi produces three different copper, nickel and pyrite concentrates.

Experimental

The purpose of the work was to clarify, which leaching conditions should be used to recover as much as possible of the valuable copper, nickel and zinc metals. Tailings from the Hitura and Pyhäsalmi mines were collected in years 2010–2011 and stored under water. Freeze-dried and homogenized samples were analysed by the ICP-AAS. The results are shown in Table 1. Attention must be paid to the facts that the Hitura tailings contained some zinc and Pyhäsalmi tailings contained little nickel. Sands contained a ten-fold, or greater, amount of iron compared to the wanted metals.

Material	Cu, g/kg	Ni, mg/kg	Zn, mg/kg	Mg, g/kg	Mn, g/kg	Fe, g/kg
Hitura tailing	1.20	2775	70	41.60	1.27	111.00
Pyhäsalmi tailing A	1.56	25	2185	9.64	1.14	205.00
Pyhäsalmi tailing B	0.52	16	738	6.96	0.40	764.00

Table 1. Average metal concentrations in Hitura and Pyhäsalmi tailings

The Hitura tailings consist mainly serpentinite $Mg_3(OH)_4Si_2O_5$ (52.6%), chlorite $(Mg,Fe)_5Al(Si_3Al)O_{10}(OH)_8$ (12.8%),and tremolite or antofyllite $Ca_2(Mg,Fe)_5Si_8O_{22}(OH)_2$ and $(Mg,Fe)_7Si_8O_{22}(OH)_2$, (10.4%). In addition, the Hitura tailings contained calcite $CaCO_3(0.4\%)$ and some sulphide minerals of which pyrrhotite (Fe_{0.83-1}S) is the most common. The Hitura tailings contain acid producing sulphide minerals, but very little neutralizing carbonates. On the other hand, the serpentinite is an alkaline and acid-neutralizing mineral. Pyhäsalmi tailings A and B consist mainly of pyrite FeS_2 (38.4–83.6%) and barite (3.1–19.6%). It contains also some pyrrhotite (2.2%), sphalerite (Zn, Fe)S (0.2%) and chalcopyrite CuFeS₂ (0.1%). The Pyhäsalmi tailings contain a lot of acid-producing sulphates but only little neutralizing carbonates and silicate minerals. As shown in Table 1, the composition of Pyhäsalmi tailings A and B varies greatly, illustrating the heterogenic and time dependent nature of tailings (Toropainen and Heikkinen 2006).

Chemical leaching experiments were done as batch leaching tests with Hitura and Pyhäsalmi tailings A, which contained more valuable metals than Pyhäsalmi tailings B. Constants in experiments were volume of acid 700 cm³, amount of solid 250 g, speed of stirrer 200 rpm and gas flow rate 1 dm³/min. Experiment time was 120 minutes. During the test samples were taken from the solution and the values of pH and redox potential were followed. The 15 cm³ of samples were taken at 5, 15, 30, 60 and 120 minutes from the beginning of experiment. Before sampling stirring and gas purging were stopped and solution was allowed to settle. The solution sample was taken from the surface of the solution, and immediately filtered and stabilized. pH and redox potential values were measured at 0, 5, 10, 20, 30, 40, 60, 80, 100 and 120 minutes. Factorial testing with three variables and three levels was done for both materials. Factors were pH that had levels 0, 1 and 3, temperature at levels 25 °C, 50 °C and 75 °C and redox potential, which was controlled by purging with oxygen, air or nitrogen. Table 2 shows the factor levels and combinations used in factorial testing. Note that for acid concentration the highest level is the highest acid concentration that

Std Order	Run Order	pН	T, ℃	Redox
4	1	0	75	Nitrogen
9	2	1	50	Air
6	3	0	25	Oxygen
2	4	0	25	Nitrogen
10	5	1	50	Air
1	6	3	25	Nitrogen
8	7	0	75	Oxygen
3	8	3	75	Nitrogen
5	9	3	25	Oxygen
7	10	3	75	Oxygen

Table 2. Randomized order of experiments and factor combinations

numerically means lowest pH-value. When the variables had been chosen, the 2^3 test series with center point was planned using the MINITAB 16 software. In Table 2 the column Run Order is the order of tests and Std Order is the order used for the analysis. The same test program of ten tests was done for both tailings. Factorial analysis of the variables was made with the MINITAB 16 statistic calculation software. Three major analyses were conducted. Pareto analysis shows the significance of the factor to the measured response. The main factor analysis shows the effect of each individual factor on the measured response. An interaction plot shows how two factors effect on the response together.

For bioleaching experiments the VTT (VTT Technical Research Centre of Finland) mixed acidophilic culture was cultivated in duplicate 500 cm³ Erlenmeyer flasks with a working volume of 250 cm³. Cultivations were incubated at 28 °C with shaking at 150 rpm. Sterile growth medium contained deionized water (ddH₂O), mineral salts and trace elements (Table 3) and it was supplemented with sterile S⁰ (1% w/v) and FeSO₄ (4.5 g/dm³ Fe²⁺). Vitality of the cultivation was monitored by pH, Oxidation Reduction Potential (ORP) and dissolved Fe²⁺ measurements, and by microscoping (Zeiss Axioskop II and Zeiss Axio Imager). New medium was made every fortnight.

Chemical formula	Concentration, mg/dm ³	Chemical formula	Concentration, mg/dm ³
$(NH_4)_2SO_4$	2160	H ₃ BO ₃	1.4
K ₂ HPO ₄	36	MnSO ₄ ·H ₂ O	1.4
MgSO ₄ ·7H ₂ O	360	Na ₂ MoO ₄ ·2H ₂ O	0.6
Ca(NO ₃) ₂	7.2	CoCl ₂ ·6H ₂ O	0.4
FeCl ₃ ·6 H ₂ O	7.9	ZnSO ₄ ·7H ₂ O	0.6
CuSO ₄ ·5 H ₂ O	0.4	Na_2SeO_4	0.07

Table 3. Concentration of growth medium

Bioleaching experiments were done as batch leaching tests with Hitura and Pyhäsalmi tailings B. As Pyhäsalmi tailings B contained less valuable metals than Pyhäsalmi tailings A, it was seen more suitable for cost effective bioleaching technique. Preliminary bioleaching experiments were done to clarify optimal conditions for scaled up reactor experiments. Bioleaching experiments were conducted in duplicate 250 cm³ Erlenmeyer flasks containing mineral salts medium and trace elements (Table 3) with a total working volume of 100 cm³. Flasks were inoculated with 10% (v/v) VTT's mixed acidophilic culture and incubated at 28 °C with shaking at 150 rpm. Examined parameters were addition of S⁰ (0–1% w/v) and/or FeSO₄ (0–4.5 g/dm³ Fe²⁺), pH fixation with strong H₂SO₄ to pH 2, and pulp density of tailings (1–10% w/v). Progress of bioleaching was monitored by pH, ORP and dissolved Fe²⁺ measurements. Liquid samples were taken to analyse dissolved metals and anions after

experiments. Samples were filtered with 0.45 μ m filter paper and stabilized with strong HNO₃ (0.25 cm³ / 50 cm³ of sample). Tailings were bioleached for 20 days.

Scaled up bioleaching reactors were run in batch mode in 3 dm³ Continuously Stirred Tank Reactors (CSTR) with a total working volume of 2 dm³. Reactors were equipped with mixers and 10 cm diameter propellers. Stirring speed was 100 rpm. The aeration was supplied from the bottoms at 1.5 dm³/min. The air bubble size was reduced with a choke. Temperature of reactors was maintained at 28°C with heated water circulations. The leaching solution contained mineral salts, trace elements, ddH₂O (Table 4) and 10% (v/v) of VTT's mixed acidophilic culture. The experiment with Hitura tailings was supplemented with S⁰ (1% w/v) and FeSO₄ (4.5 g/dm³ Fe²⁺). Also pH was fixed to 2 with strong H₂SO₄. Experiment with Pyhäsalmi tailings B had no S⁰ or FeSO₄ additions, and pH was not fixed. Both tailings were bioleached for 20 days. Progress of bioleaching was monitored by pH, ORP and dissolved Fe²⁺ measurements. Liquid samples were taken to analyse dissolved metals and anions after experiments. Samples were filtered with 0.45 µm filter paper and stabilized with strong HNO₃ (0.25cm³ / 50 cm³ of sample). Also solid reject was analysed. Tailings were bioleached for 20 days.

Results

Chemical leaching tests

The leaching kinetics of different metals was estimated by solution analyses. The recovery of metals was calculated using solution and tailing analyses. The leaching tests showed that dissolution of metals is most efficient in the most acid solutions. Examples of dissolution kinetics of the Hitura tailings are shown in Figs. 1 and 2. Figure 1 shows that iron and magnesium concentrations in the leach solution are much higher than those of valuable metals. This results from the raw material composition, where iron and magnesium compounds dominate. The iron concentrations in the most successful tests were $4-11 \text{ g/dm}^3$ and magnesium concentrations $7-38 \text{ g/dm}^3$. Figure 2 shows that the contents of the valuable metals were typically only a hundredth of the contents of iron or magnesium. The Cu, Ni and Mn concentrations were in the range of $100-300 \text{ mg/dm}^3$ in the most successful experiments.

The recoveries from the Hitura tailings are shown in Table 4. The most successful leaching tests were 7, 3, 4 and 1, even though copper was not recovered in the last one. Table 4 shows that recovery of magnesium is greater than that of other metals. The recovery of iron is often of the same order as the recovery of the valuable metals. No preferential dissolution of valuable metals was found. In tests 1 and 7 with low pH and high temperature, magnesium recoveries were over 100%, which must be an analytical error. Leaching tests with the Hitura tailings dissolved mainly magnesium and iron.

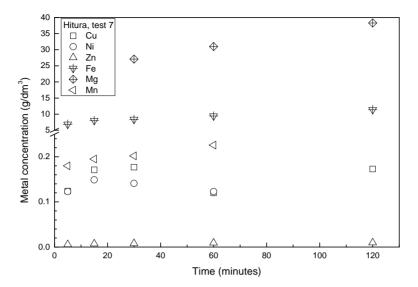


Fig. 1. Metal concentration as function of time in Hitura tailings test 7 (pH = 0, T = 75 °C, O₂)

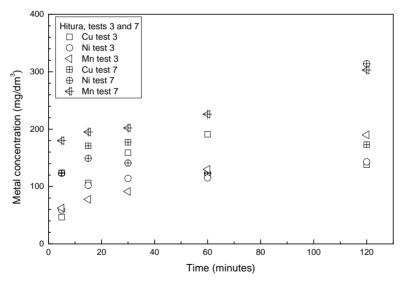


Fig. 2. Dissolution of Cu, Ni and Mn from Hitura tailings in tests 3 (pH = 0, T = 25 °C, O₂) and 7 (pH = 0, T = 75 °C, O₂)

Examples of dissolution kinetics in leaching tests with the Pyhäsalmi tailings are shown in Figs. 3 and 4. Also in these tests most efficient dissolution was found in tests with highest acidity. In the Pyhäsalmi tailings tests iron concentrations were 2-8 g/dm³ and magnesium concentrations 1.5-4 g/dm³ in the most successful tests (Fig. 3). The

valuable metal concentrations were 20-80 mg/dm 3 Cu, 200–500 mg/dm 3 Zn, and 100–200 mg/dm 3 Mn.

Test, RunOrder	рН	T, ℃	Redox	Cu, %	Zn,%	Ni, %	Fe, %	Mg, %	Mn,%
1	0	75	N_2	0.14	25.9	18.6	17.5	173	46.8
2	1	50	Air	0.00	0.08	3.07	0.01	8.83	8.31
3	0	25	O_2	27.7	10.5	12.4	12.8	81.4	35.9
4	0	25	N_2	13.1	8.81	5.85	6.36	38.6	14.0
5	1	50	Air	0.01	0.17	3.36	0.01	7.27	8.37
6	3	25	N_2	0.00	0.02	0.04	0.00	1.89	0.15
7	0	75	O_2	34.7	33.0	27.2	24.6	220	57.3
8	3	75	N_2	0.00	1.03	0.02	0.01	1.23	0.15
9	3	25	O_2	0.01	0.02	0.19	0.00	2.09	0.18
10	3	75	O_2	0.02	0.09	0.10	0.00	3.16	0.29

Table 4. Recoveries from Hitura tailings based on analyses after 120 minutes of leaching

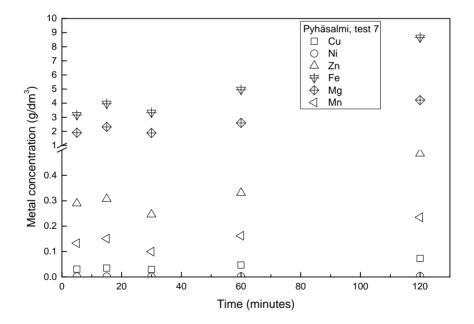


Fig. 3. Metal concentration as a function of time in Pyhäsalmi tailings test 7 (pH = 0, T = 75 °C, O₂)

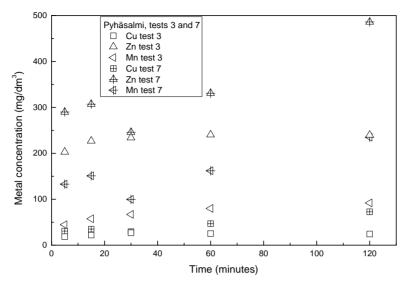


Figure 4. Dissolution of Cu, Zn and Mn from Pyhäsalmi tailings in tests 3 (pH = 0, T = 25 °C, O₂) and 7 (pH = 0, T = 75 °C, O₂)

The recoveries from the Pyhäsalmi tailings are shown in Table 5. The most successful tests were again the tests with highest acidity 7, 4, 3 and 1. Dissolution of valuable metals Cu, Zn and Mn in other tests was very small. The solution concentrations of iron and magnesium were also in Pyhäsalmi tests much higher than concentrations of the valuable metals. In Pyhäsalmi tests iron recovery was often smaller than that of valuable metals but magnesium extraction was again high. The Pyhäsalmi tests showed slightly better preferential dissolution of valuable metals than the Hitura tests.

Test, RunOrder	pН	Т, °С	Redox	Cu, %	Zn,%	Ni, %	Fe, %	Mg, %	Mn,%
1	0	75	N_2	12.8	46.4	32.9	9.03	90.6	41.3
2	1	50	Air	0.34	26.9	17.9	1.07	17.0	13.7
3	0	25	O_2	3.75	26.4	19.7	2.75	36.6	19.4
4	0	25	N_2	3.66	26.7	19.4	2.81	37.1	19.6
5	1	50	Air	0.90	19.7	11.9	0.82	12.9	10.0
6	3	25	N_2	0.00	0.01	0.14	0.00	1.47	0.20
7	0	75	O_2	11.2	53.4	31.1	10.1	105	49.5
8	3	75	N_2	0.00	0.01	0.08	0.00	3.44	0.26
9	3	25	O_2	0.00	0.02	0.18	0.00	1.64	0.23
10	3	75	O_2	0.00	0.02	0.11	0.00	3.31	0.38

Table 5. Recoveries from Pyhäsalmi tailings based on analyses after 120 minutes leaching

The effect of leaching factors were analysed using the Minitab 16 software. The main effects of the Hitura tailings leaching were strong increased dissolution of Cu, Ni and Mn and also of Fe and Mg with increasing acidity and redox potential and low increase with increasing temperature. The increase in acidity had positive interaction with temperature and redox potential for all metals. All tested factors were significant for all metals. The main effects of the Pyhäsalmi A tailings leaching were increased dissolution of Cu, Zn and Mnas well as Fe and Mg with increasing acidity and temperature and low increase or even decrease with increasing redox potential. The increase in acidity had positive interaction with temperature for all metals. Tested factors were significant for Cu, Mn, Fe and Mg but not for Zn, possibly due to highly scattered results.

Bioleaching tests

Figure 5 presents monitored parameters of bioleaching of the Hitura tailings in a 3 dm³ reactor. At the beginning of the experiment, pH tends to rise, mainly due to acid-neutralizing serpentinite. However, after addition of H_2SO_4 (95 cm³ 95% H_2SO_4 / 1 kg of Hitura tailings), biological sulphuric acid production outmatched neutralization capacity and pH decreased to 1.4. At the same time, sulphate concentration rose from 13 to 37 g/dm³, illustrating the activity of sulphur oxidizing bacteria and sulphide mineral dissolution. Dramatic changes in both ORP (E_h) and concentration of dissolved Fe²⁺ illustrate the activity of iron oxidizing bacteria. Dissolved Fe²⁺ concentration is quickly decreased by bacteria.

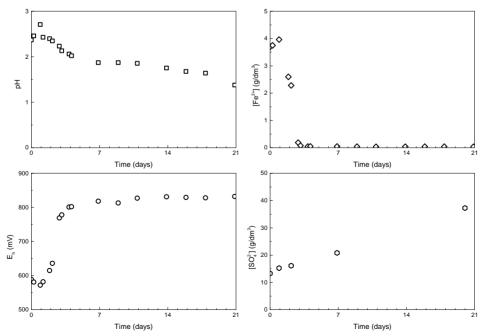


Fig. 5. Monitored parameters in bioleaching reactor of Hitura tailings

The leaching kinetics of different metals was estimated by solution analyses, presented in Fig. 6. Concentrations of all valuable metals were increasing steadily, as predicted. Mg followed also this trend, rising from 0.2 g/dm³ to 3.4 g/dm³. However, concentration of iron remained rather constant at ~ 5 g/dm³, mainly due to addition of FeSO₄ (4.5 g/dm³ Fe²⁺). Table 6 shows the recoveries of metals. The recovery were calculated using solution and tailing analyses.

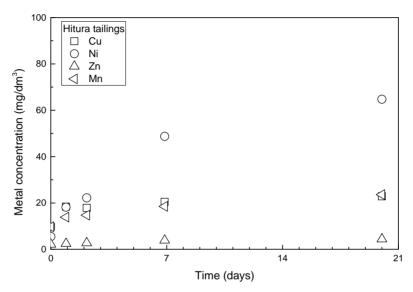


Fig. 6. Metal concentration as function of time in Hitura tailings bioleaching reactor, $S^0 = 1\%$ (w/v), $Fe^{2+} = 4.5$ g/dm³, fixed pH ≤ 2 , pulp density 2.5% (w/v)

Table 6. Recoveries from Hitura tailings based on the analyses after 20 days of bioleaching

Hitura tailings	Cu, %	Zn,%	Ni, %	Fe, %	Mg, %	Mn,%
Recovery	76.5	100 ¹⁾	93.3	_2)	100 ¹⁾	74.3

¹⁾Over 100 % recovery, due to error in ICP analyses

²⁾FeSO₄ added to reactor, iron recovery cannot be calculated

Figure 7 presents monitored parameters of bioleaching of Pyhäsalmi tailing B in the 3 dm³ reactor. pH began to decrease immediately and end up at pH 1.4. At the same time sulphate concentration rose from 3 to 101 g/dm³. This illustrates the activity of sulphur oxidizing bacteria and efficient sulphide mineral dissolution. Dramatic changes in the ORP (E_h) and low Fe²⁺ concentration during the experiment illustrate the activity of iron oxidizing bacteria.

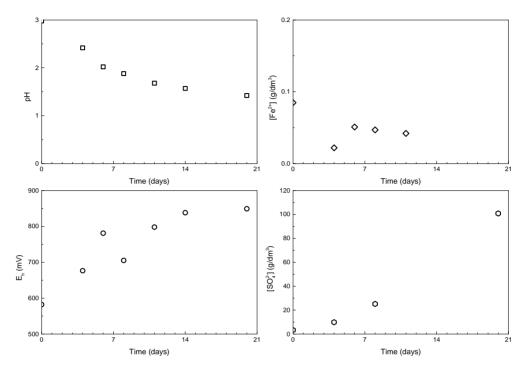


Fig. 7. Monitored parameters in bioleaching reactor of Pyhäsalmi B tailings

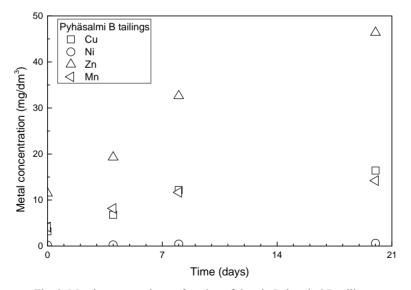


Fig. 8. Metal concentration as function of time in Pyhäsalmi B tailings bioleaching reactor (no chemical additives, pulp density 5% (w/v))

The leaching kinetics of different metals was estimated by solution analyses (Fig. 8). Concentrations of all valuable metals were increasing steadily, as predicted. Magnsium and Fe followed also this trend, rising from 0.07 g/dm^3 to 0.26 g/dm^3 and 0.1 to 6.0 g/dm³, respectively. As FeSO₄ was not added, iron was completely dissolving from the mineral matrix. Table 7 shows the recoveries of metals. The recoverys were calculated using solution and tailing analyses.

Table 7. Recoveries from Pyhäsalmi tailings B based on the analyses after 20 days of bioleaching

Pyhäsalmi tailing B	Cu, %	Zn,%	Ni, %	Fe, %	Mg, %	Mn,%
Recovery	71.3	100 ¹⁾	85.2	17.6	83.3	79.1

¹⁾Over 100 % recovery, due to error in the ICP analyses

Composition of leached tailings

Leached tailings were analysed with ICP-AAS for residual metals. The results are shown in Table 8. The results indicate that bioleaching can be more selective towards the wanted metals.

Table 8. Residual metals in leached tailings

Material	Cu, mg/kg	Ni, mg/kg	Zn, mg/kg	Mg, g/kg	Mn, mg/kg	Fe, g/kg
Hitura tailing (chem. leaching, test 7)	439	1533	62	59.85	412	61.75
Hitura tailing (bioleaching reactor)	369	404	30	75.90	513	119.40 ¹⁾
Pyhäsalmi tailing A (chem. leaching, test 7)	1600	29	1490	8.26	559	163.50
Pyhäsalmi tailing B (bioleaching reactor)	286	9	112	2.96	153	442.00

¹⁾FeSO₄ added to reactor

Discussion

Both tailings gave similar results in the chemical leaching tests. All variables had effect for leaching metals. High temperature and redox potential and low pH gave best results. Both tailings contained minerals that neutralize acid. The tailings had also much iron and magnesium containing minerals. The solution after chemical leaching tests contained mostly iron and magnesium. The Hitura tailing contained up to 11 g/dm³ Fe and 38 g/dm³ Mg and Pyhäsalmi tailing 8–9 g/dm³ Fe and 4 g/dm³ Mg. Due to tailings composition, the amount of these metals was 20–100-fold larger than the amount of valuable metals, which was typically 100–300 mg/dm³. The iron and magnesium compounds dissolve easily and no preferential dissolution of valuable metals was achieved. High acid consumption and high Fe and Mg levels will make valuable metals recovery difficult with conventional methods. Also it must be emphasized that the leached tailings contained still relatively high concentrations of valuable metals making utilisation or disposal challenging.

In bioleaching experiments it was seen that bacteria were able to grow in the presence of both tailings when low pulp densities (2.5-5.0% w/v) were used. The

Hitura tailings contained plenty of acid-neutralizing minerals (e.g. serpentinite), which caused the need for external acid addition alongside the biological acid production. When there were minor amounts of suitable substrates in the Hitura tailings (e.g. FeS_2), S^0 and $FeSO_4$ were added to the reactor. With a 20-day bioleaching, the majority of valuable metals were leached to the solution, but as Fe and Mg levels were magnitudes higher, recovery from solutions is challenging. Pyhäsalmi tailing B was seen as more suitable material for bioleaching technique as it contained lower amount of acid neutralizing minerals. Moreover, the Pyhäsalmi tailing B consisted mainly of FeS_2 , which was used itself as a substrate for iron and sulphur oxidizing bacteria and no S^0 or $FeSO_4$ additions were needed. Valuable metals were leached efficiently and also residual metal concentrations in solid reject were lower. However, the recovery processes will still face challenges due to high Fe and Mg concentrations in produced liquid.

Conclusions

Sulfidic tailings were leached using sulfuric acid and bacteria. The tailings consisted mainly of iron and magnesium containing minerals as well as acid neutralizing minerals. The concentrations of valuable metals like Ni, Cu, Zn and Mn were orders of magnitude lower than those of Fe and Mg. This caused high consumption of acid and poor selectivity in chemical leaching. Bioleaching using iron and sulphur oxidizing bacteria was more selective towards the valuable metals. Even though the reaction rates and valuable metal concentrations in bioleaching were lower than in chemical leaching, the selectivity and lower processing costs make bioleaching more attractive. Both in chemical leaching and bioleaching the high concentration of iron and magnesium in solution will make metals recovery challenging.

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