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# Indirect oxidative bioleaching of a polymetallic black schist sulfide ore

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ABSTRACT

Biomining of base and precious metal-containing sulfidic ores and concentrates is an important global biotechnology. Currently, mineral bio-processing is carried out in dumps, heaps and stirred tanks located on the land surface. An alternative approach, "deep *in situ* biomining", would solubilise target metals from fractured ore bodies buried deep (>1 km) underground, using a microbially-generated acidic, ferric iron-rich lixiviant. Pregnant leach solutions (PLS) generated are pumped to the land surface where metals are extracted and recovered, ferric iron regenerated in a bioreactor and the oxidised lixiviant re-injected underground. To examine the feasibility of using an indirect bioleaching approach to extract metals from an ore body, a laboratory test was carried out using a polymetallic sulfidic black schist ore (from Talvivaara, Finland) in a non-aerated column reactor, maintained at 30 °C at atmospheric pressure. Following an initial acid leaching phase (using 100 mM sulfuric acid) the ore was subjected to ferric iron-catalyzed oxidative dissolution using a lixiviant generated in a laboratory-scale ferric iron-generating bioreactor (FIGB). During the 16 week time course of the experiment, 93% of manganese, 88% of cobalt, 79% of nickel, 75% of iron, 75% of zinc and 55% of copper present in the ore was extracted. The planktonic microbial populations in the FIGB and those in the PLS were similar, and dominated by *Leptospirillum ferriphilum*, iron-oxidising *Acidithiobacillus* spp. and the archaeon *Ferroplasma acidophilum*.
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## 1. Introduction

Using the microorganisms to accelerate the oxidative dissolution of sulfidic minerals in ores and minerals ("biomining"), has developed into an global biotechnology for extracting and facilitating the recovery of copper and gold, and to a lesser extent of cobalt, nickel and zinc (Rawlings and Johnson, 2007). Although still a niche technology, it has the potential to be used in a variety of contexts where conventional mineral processing is not economically or environmentally feasible, such as low grade ("run of mine") and polymetallic ores, and those with high arsenic contents (Brierley and Brierley, 2001). Since readily accessible, high-grade metal ores are becoming depleted, and demands for base and precious metals are increasing, there is a need to find an economical way to recover metals from materials that are currently perceived as being inaccessible, for example due to their placement deep (>1 km) below the land surface.

Conventional biomining is carried out in dumps, heaps and stirred tanks (Brierley, 2008). Another approach, used both historically and in more recent times to extract additional uranium from otherwise worked out mines, is to use an *in situ* approach, whereby underground mines are periodically flooded to facilitate the microbially-assisted solubilisation of target metals which are subsequently recovered from the pregnant leach solutions (PLS). Uranium is currently being extracted from shallow aquifers using an *in situ* approach, in various parts of the world (Mudd, 2001), though in these situations the "bio" component is probably of minor significance. The proposal to develop and test a "deep *in situ* biomining protocol", is the remit of the European Union Horizon 2020 project "BioMOre" (www.biomore.info). Here the objective is to bioleach base metal ore deposits located at far greater depths (>1 km), where conditions, such as elevated temperatures and pressures, would be much different from those encountered hitherto in *in situ* operations. In addition, ensuring the supply of oxygen and carbon dioxide, which are both essential for growth and oxidation of iron and sulfur by biomining microorganisms, to fractured ore bodies at these depths would not be pragmatic. To get around this problem, an indirect bioleaching protocol has been suggested (Johnson, 2015). This involves delivering an acidic, ferric iron-rich lixiviant, generated in bioreactors (e.g. Livesey-Goldblatt et al., 1977) located at the land surface, to the ore body where it flows through channels opened by hydraulic

*Abbreviations:* FIGB, ferric iron-generating bioreactor; PLS, pregnant leach solution; T-RFLP, terminal restriction enzyme fragment length polymorphism.

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fracturing, oxidising sulfide minerals and solubilising metals such as copper. The PLS generated is reduced (ferrous iron-rich), rather than oxidised as in conventional biomining, and is pumped to the land surface where the soluble copper is removed, e.g., using sulfide precipitation or solvent extraction and electrowinning. Ferrous iron in the raffinate solution is then re-oxidised to ferric in the bioreactor, and the regenerated lixiviant recirculated into the ore body. Indirect mineral bioleaching has been described elsewhere (Schippers and Sand, 1999; Rawlings, 2004) and has the advantage of separating (chemical) mineral oxidation from (biological) ferrous iron oxidation, allowing conditions (such as temperature) for each to be optimised separately. Also, since ferric iron-catalyzed oxidation of sulfide minerals is independent of molecular oxygen, the absence of oxygen in lixiviants percolating through a deep-buried ore body would not preclude the extraction of base metals, though it would mean that there would be no regeneration of ferric iron *in situ*, in contrast to biomining carried out in aerated bioheaps and stirred tanks.

The polymetallic sulfidic black schist which outcrops at the land surface at Talvivaara, Finland, has been processed using heap bioleaching during the past decade (Riekkola-Vanhanen, 2013), though recent problems at the site have caused a curtailment in mining (Bedford, 2015). The ore body contains of quartz (25%), aluminum silicates (38%), iron sulfides (16%), graphite (10%), magnesium-iron silicates (8%) and sphalerite (ZnS)-pentlandite ($(Fe,Ni)_9S_8$)-violarite ($FeNi_2S_4$) (3.2%) (Loukola-Ruskeeniemi and Heino, 1996). The major base metals present are Fe (10–13%), Zn, (0.2–0.5%), Ni (0.2–0.3%), Cu (0.1–0.6%) and Co (0.02–0.04%) (Puhakka et al., 2007; Dopson et al., 2008; Wakeman et al., 2008; Halinen et al., 2009), with nickel being the primary metal targeted for extraction and recovery. This is deported in pentlandite and violarite, pyrite ($FeS_2$) and pyrrhotite ($Fe_{(1-x)}S$). Attempts to produce a high-grade nickel concentrate from the ore were not successful because of its graphite content, and heap bioleaching is carried out using ground ore particles, agglomerated using sulfuric acid. Several independent preliminary studies, carried out in aerated columns, had suggested that Talvivaara ore was amenable to oxidative dissolution in aerated, irrigated heaps (Puhakka and Tuovinen, 1986a,b,c; Riekkola-Vanhanen and Heimala, 1999; Wakeman et al., 2008; Halinen et al., 2009). The exothermic nature of pyrrhotite oxidation, in particular, maintains elevated temperatures (up to ~80 °C) in the insulated heaps, supporting the activities of thermophilic as well as mesophilic biomining microorganisms (Riekkola-Vanhanen, 2013). Dopson et al. (2008), however, reported that bioleaching the ore at extremely low pH (~1.5) resulted in extensive solubilisation of silicon (by acid dissolution of aluminosilicates) which greatly increased the viscosity of percolating liquors and impeded the bioleaching process.

## 2. Materials and methods

### 2.1. Ferric iron-generating bioreactor (FIGB)

A laboratory-scale biofilm reactor was commissioned to generate an acidic ferric iron lixiviant. A 2 L glass reactor vessel fitted with a stainless steel top plate and various inserts (Electrolab, U.K.) was filled sequentially with a layer (~2 cm depth) of acid washed gravel, followed by ~13 cm of acid-washed porous glass beads (8 mm diameter; Poraver-Dennert GmbH, Germany) and a second 2 cm layer of gravel. Six hundred millilitres of a solution containing basal salts and trace elements (Ňancucheo et al., 2016), adjusted to pH 1.6 with sulfuric acid, was added and the reactor vessel sterilised by autoclaving. After cooling, a filter-sterilised solution of ferrous sulfate was added to the vessel, to give a final $Fe^{2+}$ concentration of ~50 mM. The reactor was then inoculated with 14 strains of acidophilic bacteria and archaea, commonly or occasionally found in biomining operations and maintained in the *Acidophile Culture Collection* at Bangor University, UK (Table 1). These acidophiles had been pre-grown in appropriate liquid media (ferrous iron or elemental sulfur for obligately autotrophic species; yeast extract-amended media for facultatively autotrophic and obligately heterotrophic species) and at suitable temperatures (30 °C for mesophilic species and 40 °C for thermo-tolerant species). The temperature of the FIGB was maintained at 35 °C using a FerMac 240 temperature controller (Electrolab) and the bioreactor was aerated (at ~ 1 L/min) with filter-sterilised air. When all of the ferrous iron had been oxidised, the bioreactor was switched to continuous flow mode, using a slightly more acidic (pH 1.5) influent liquor (to minimize jarosite precipitation) and amended also with 100 mM magnesium sulfate to increase the buffering afforded by the sulfate/bisulfate couple. Flow rates into and out of the bioreactor vessel varied between 55 and 400 mL/h, equivalent to hydraulic retention times (HRTs) of between 33 and 5 h. Effluent liquors generated by the FIGB where >99% of the ferrous iron had been oxidised to ferric, were collected and used as lixiviants for indirect bioleaching of Talvivaara ore, as described below.

### 2.2. Acid leaching and indirect bioleaching of Talvivaara ore

A column bioreactor, comprising a Perspex tube (32 cm long and 7.5 cm diameter) filled with 1.375 kg of non-sterile polymetallic ore (2.0–6.7 mm diameter) provided by the Talvivaara Mining Company, was set up in a temperature-controlled (30 °C ± 2 °C) room. Non-sterile sulfuric acid (100 mM; 600 mL) was added to the column, and after three days the column was connected to 5 L inflow and outflow vessels and fresh 100 mM sulfuric acid pumped through continuously for a further 10 days, at a constant flow rate of 55 mL/h (equivalent to a HRT of 11 h). Following this, this influent liquor was changed to the ferric iron lixiviant generated by the FIGB, which contained ~50 mM ferric iron, and had a pH of ~1.8. The same flow rate was maintained as during the acid leaching phase, and indirect bioleaching of the Talvivaara ore continued for a further 103 days. The column reactor was not aerated in any stage of the experiment. Samples of liquids draining the column reactor were withdrawn at regular intervals to determine pH, $E_H$, concentrations of dissolved metals and silicon, and also for microbiological and molecular biological analysis.

### 2.3. Chemical and geochemical analyses

Concentrations of ferrous iron in the effluent liquors were determined using the Ferrozine colorimetric assay (Stookey,

**Table 1**
Acidophilic prokaryotes used to inoculate the ferric iron-generating bioreactor (FIGB).

| Mesophilic acidophilic bacteria | |
|---|---|
| *Leptospirillum* (*L.*) *ferrooxidans*$^T$ | Autotrophic $Fe^{2+}$ oxidizer |
| *Acidithiobacillus* (*At.*) *ferrooxidans*$^T$ | Autotrophic $Fe^{2+}/S^0$ oxidizer |
| *At. ferridurans*$^T$ | Autotrophic $Fe^{2+}/S^0$ oxidizer |
| *At. ferrivorans* strain CF27 | Autotrophic $Fe^{2+}/S^0$ oxidizer |
| '*At. ferriphilus*'$^T$ | Autotrophic $Fe^{2+}/S^0$ oxidizer |
| *At. thiooxidans*$^T$ | Autotrophic $S^0$ oxidizer |
| *Acidiphilium* (*A.*) *cryptum* strain SJH | Heterotrophic $S^0$ oxidizer |
| '*Acidibacillus* (*Ab.*) *ferrooxidans*'$^T$ | Heterotrophic $Fe^{2+}$ oxidizer |
| *Thermo-tolerant acidophilic bacteria* | |
| *L. ferriphilum* strain MT63 | Autotrophic $Fe^{2+}$ oxidizer |
| *Sulfobacillus* (*Sb.*) *thermosulfidooxidans*$^T$ | Mixotrophic $Fe^{2+}/S^0$ oxidizer |
| *At. caldus*$^T$ | Mixotrophic $S^0$ oxidizer |
| '*Ab. sulfurooxidans*$^T$' | Heterotrophic $Fe^{2+}/S^0$ oxidizer |
| Thermo-tolerant acidophilic archaea | |
| *Ferroplasma* (*Fp.*) *acidiphilum* strain BRGM4 | Heterotrophic $Fe^{2+}$ oxidizer |
| *Acidiplasma* sp. strain Fv | Heterotrophic $Fe^{2+}$ oxidizer |

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