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Selection of thermophiles for base metal sulfide concentrate leaching, Part II: Nickel-copper and nickel concentrates

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ABSTRACT

The ferrous iron- and sulfur-oxidizing Acidithiomicrobium was the dominant iron-oxidizing bacterium in a mixed culture of moderate thermophiles in a bioreactor with a high concentration of nickel in solution (20 g l^{-1}) and a continuous feed of nickel concentrate. This is the first demonstration specifically indicating that this bacterium could be used for industrial processing of a base metal sulfide concentrate. However, its sensitivity to copper determined that it was replaced by *Sulfobacillus thermosulfidooxidans* in the mixed culture when the feed was a nickel-copper concentrate. The extraction of copper from the nickel-copper concentrate by moderate thermophiles, despite fine grinding of the feed, was relatively poor (50% at 49 °C) compared to that achieved with high temperature archaea (92% at 77 °C) in single, continuous reactors with 5% w/v concentrate feeds and residence times of 2.7 and 2.5 days respectively.
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1. Introduction

It is approximately fifty years since the first investigations of bioleaching of pentlandite concentrates were sponsored by mining companies in Canada during the 1960s. This unpublished work involved shaken flask tests with mesophilic bacteria at 30–35 °C. Subsequently, there were at least two significant developments in the early 1980s towards nickel concentrate bioprocessing. First, an investigation of semi-continuous bioleaching which proposed a possible process flow-sheet and considered some aspects that became critical in later work, such as the influence of particle size (Miller et al., 1986). Secondly, there was the demonstration of enhanced processing rates and yields by the use of thermophilic bacteria and archaea (Norris et al., 1986; Norris and Parrott, 1986). Since then, there has been continuous pilot scale test work that showed higher recoveries of nickel with thermophiles compared to mesophiles (Dew et al., 1999). There have also been nickel-bioleaching pilot plants with processes that were described as ready for commercial implementation: BioNIC® developed by Gencor/Billiton/Mintek and Queensland Nickel Ltd., which processed 300 kg concentrate per day for six months (Miller et al., 1997; Norton et al., 1998; Heinzle et al., 1999), and a process developed by Mintek, which used thermophiles at 70 °C (Neale et al., 2009). The lack of industrial, tank-bioleaching of nickel sulfides despite such research and development has partly reflected a generally more convincing technical and economic case for more conventional processing routes. However, following initial work (Wakeman et al., 2011) and pilot plant development (Neale et al., 2015), industrial processing of a nickel sulfide concentrate in stirred-tank bioreactors has recently been introduced for processing a pentlandite/pyrrhotite concentrate by-product of talc mining.

This report revisits the selection of thermophiles for nickel concentrate leaching, particularly with regard to the relative merits of moderately thermophilic *Sulfobacillus* and Acidithiomicrobium species in this context. It also extends previous work, which used the same moderate thermophiles for continuous leaching of a nickel sulfide concentrate (Cleaver et al., 2007), to investigate the effect of higher concentrations of nickel in solution. Acidithiomicrobium (then referred to as a novel *Acidimicrobium* species) was the dominant iron-oxidizing bacterium in the previous work but the concentration of nickel in solution did not exceed 10 g l^{-1}. *Sulfobacillus thermosulfidooxidans* has also not been reported to tolerate high concentrations of nickel in solution (Watling, 2008).

The performance of high temperature thermophilic archaea is compared to that of the moderate thermophiles in the context of nickel-copper concentrates that could be considered for bioleaching but which contain chalcopyrite, from which copper extraction is usually poor at the lower growth temperatures of moderate thermophiles.

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2. Materials and methods

2.1. Microorganisms

The mixed cultures of moderate thermophiles and high temperature archaea which were used were as described for work with a copper concentrate (Norris et al., 2016). The key moderate thermophiles (Acidithiomicrobium *strain* P2, *Acidithiobacillus caldus* and *S. thermosulfidooxidans*) were used previously in laboratory reactors processing a nickel sulfide concentrate (Cleaver et al., 2007). Nickel tolerance of the *A. caldus* type strain (DSM 8584) was assessed. Previous concentrate leaching with the mixed culture of thermophilic archaea has been reviewed (Norris et al., 2013).

2.2. Mineral sulfide concentrates

Two concentrates were used. The nickel concentrate (particle size d_{80}, −75 μm) contained 10.5% w/w nickel (mostly in pentlandite and violarite) and 26% w/w iron (in pyrite, pyrrhotite, and the nickel sulfides). The nickel-copper concentrate (particle size d_{80}, −88 μm) contained 8.8% w/w nickel (in pentlandite), 4.3% w/w copper (in chalcopyrite and cubanite) and 35% w/w iron. A sample of this concentrate was finely ground (SMD mill grinding by Grinding Solutions Ltd., Truro, Cornwall, UK) to a particle size d_{80} of −9 μm. Samples of this finely ground concentrate were washed separately at different times, with some loss of target metals into solution. The washed sample used for work with high temperature thermophiles contained 7.6% w/w nickel, 4.3% w/w copper and 33.3% w/w iron. The washed sample used for work with moderate thermophiles contained 7.3% w/w nickel, 3.9% w/w copper and 32.5% w/w iron.

2.3. Bioreactors, culture conditions and assays

Temperature controlled (oil-jacketed), baffled reactors were used with culture volumes between 550 and 600 ml. Cultures were gassed (600 ml min^{-1}) with air supplemented with 1% v/v CO_2. Agitation was by overhead motors stirring flat, two-bladed paddles at 400 rpm. Culture medium contained (g l^{-1}) K_2HPO_4 (0.3), $(NH_4)_2SO_4$ (0.6), and $MgSO_4{\cdot}7H_2O$ (0.5). Overflows during continuous operation were through reactors' side arms.

Reactors containing moderate thermophiles at 49 °C were fed with medium from reservoirs through pinch valves which were set to open every four hours, each time delivering one sixth of the required daily flow rate. Medium and the nickel and nickel-copper concentrates were added daily to the reservoirs linked to the pinch valve feed system. The medium feeds were adjusted with sulfuric acid to pH 2.5 with the nickel concentrate and to pH 1 with the nickel-copper concentrate. Where required, the medium feeds were supplemented with either nickel (15 g l^{-1}, added as nickel sulfate) or nickel and copper (15 g l^{-1} and 4 g l^{-1} respectively, added as sulfates).

Reactors containing the high temperature thermophiles at 77 °C were fed with medium adjusted with sulfuric acid to pH 1.3 and the nickel-copper concentrate was added directly twice daily (to a primary reactor when two were operated in series).

Ferrous iron in solution was measured by titration against 5 mM ceric sulfate in 5% v/v sulfuric acid with 1,10-phenanthroline ferrous complex (Fisher Scientific) as indicator. 1 ml samples were used for titration after removal of solids by centrifugation. Metals in solution were measured by atomic absorption spectrophotometry of reactor samples centrifuged to remove solids and of hot acid digested (HCl/HNO_3) solid residues. Cell numbers and biomass volumes were estimated from orifice electrical flow impedance measurements using a CellFacts Particle Size Analyser (CellFacts Instruments).

3. Results and discussion

3.1. Nickel concentrate leaching with moderate thermophiles: Nickel tolerance

The moderately thermophilic culture was maintained at 49 °C in a stirred reactor which was fed with a medium that was supplemented with nickel sulfate (15 g Ni l^{-1}) and a nickel concentrate (5% w/v, 10.5% w/w nickel). The nickel concentration in solution reached 19.4 g l^{-1} without apparent inhibition of the culture at a residence time of 2.7 days. Solid residues were collected from the effluent overflow over three separate 24 h periods after 56, 60 and 66 days continuous operation (25 culture volume turnovers). Analysis of the residues indicated, respectively, 88, 82 and 86% of the nickel had been leached into solution. This slightly enhanced extraction efficiency compared to 80% in previous work with the same concentrate at this temperature (Cleaver et al., 2007) reflected the mineral feed of 5% w/v compared to 10% w/v previously, a smaller particle size feed and more regularly spaced concentrate additions (six times each day compared to three times during nine hour periods of each day in the previous work). Between 61 and 65% of the iron leached remained in solution.

Microscopy confirmed that Acidithiomicrobium and *A. caldus* were the dominant bacteria and this population was maintained without apparent inhibition for the duration of the trial (ten months) with a nickel concentrate and a three day residence time with nickel in solution at about 20 g l^{-1}. It was separately demonstrated that the *A. caldus* type strain could be maintained in serial batch cultures on elemental sulfur with nickel at 20 g l^{-1}, but growth was slower than at lower concentrations of nickel.

3.2. Nickel-copper concentrate leaching with moderate thermophiles: effect of copper

The moderate thermophile mixed culture was maintained in a continuous reactor with a nickel-copper concentrate (5% w/v). The medium feed was supplemented with nickel (15 g l^{-1}) and copper (4 g l^{-1}) to allow observation of the microbial population in the presence of a copper concentration that had previously inhibited the Acidithiomicrobium species (Norris et al., 2016). Apparently stable, continuous leaching of the concentrate occurred with small variations in the nickel concentration in solution which were related to variation in the residence time through unintentional feed rate inconsistency (Fig. 1). Analyses of residues collected over 24 h periods on days 16, 27 and 31 indicated nickel extraction efficiencies of 88, 90 and 88% and copper extractions of 51, 51 and 48% respectively (Table 1). Approximately 76% of the leached iron remained in solution. Nickel extraction from the nickel-copper concentrate was slightly greater than from the nickel concentrate (Section 3.1) which was of a larger particle size (Table 1).

Previous examination of the mixed culture during continuous nickel concentrate leaching used 16S rRNA gene clone bank analysis, 16S rRNA gene dot blots and fluorescent-in-situ-hybridization with 16S rRNA oligonucleotide probes (Cleaver et al., 2007). 16S rRNA gene clone bank analysis and species-specific fluorescent antibodies were used during copper concentrate leaching (Norris et al., 2016). An electrical impedance particle analysis was introduced here which, despite factors that cause the measurements to have some approximations that preclude a precise cell count, has the considerable advantage of providing a simple, real time analysis of the culture profile. The particle analysis did not account

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