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Iron oxidation and jarosite precipitation in a two-stage airlift bioreactor

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ABSTRACT

Continuous iron oxidation and removal of jarosite precipitates from solution at low pH, ambient temperature and pressure was successfully demonstrated with a two-stage airlift bioreactor (ALBR) system. The ALBR system, comprising of two ALBRs, each with its own settler, was operated for iron oxidation and precipitation at room temperature with a mixed culture of mesophilic iron oxidisers. The two-stage reactor design allowed optimization of the overall reactor kinetics by facilitating the growth of two distinct groups of iron oxidizers at low (430 mV vs. Ag/AgCl) and high (517 mV) redox potential in the two reactors, respectively. The influent with pH 1.5 contained (g L^{-1}) 15 Fe^{2+}, 1.5 Cu, 1.5 Ni, nutrients and trace elements. The hydraulic retention time (HRT) was decreased stepwise to evaluate process performance. With the lowest HRTs (8 h in ALBR1 and 10 h in ALBR2), the overall iron oxidation and precipitation rates of the two-stage system were 0.75 ± 0.02 g L^{-1} h^{-1} and 0.15 ± 0.01 g L^{-1} h^{-1}, respectively and overall iron oxidation and precipitation of 94 ± 3% and 18 ± 1%, respectively. The percent of influent Fe, S, Cu and Ni removed as precipitates from settlers were 30.9%, 16.7%, 1.1% and 0.2%, respectively. The precipitates were predominately comprised of (>95%) jarosite with potassium jarosite being the dominant form, followed by hydronium, ammonium and sodium jarosites. In conclusion, the two-stage ALBR system allowed efficient iron oxidation and precipitation of the oxidised iron as well settling jarosite with only minor loss of Cu and Ni via co-precipitation. The process demonstrated performance that is promising for a variety of hydrometallurgical process flow sheets, where it allows iron removal from ferrous solutions without chemical addition and with negligible base metal co-precipitation losses.
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1. Introduction

Ferric iron (Fe^{3+}) is an important low-cost oxidant in hydrometallurgical processing of sulphide minerals (Halinen et al., 2009; Nurmi et al., 2009; Özkaya et al., 2007a,b). The Fe^{3+}-mediated oxidation of sulphides with concomitant metal solubilisation leads to the reduction of Fe^{3+} to ferrous iron (Fe^{2+}). Acidic Fe^{2+} rich solutions are also generated in reductive dissolution and leaching processes imposed on iron oxide minerals which contain base metals (du Plessis et al., 2011; Hallberg et al., 2011). Biological Fe^{2+} oxidation combined with iron precipitation can be achieved at ambient pressure and temperature using iron oxidising microorganisms. Biologically catalysed Fe^{2+} oxidation is preferred over abiotic oxidation, as it is several orders of magnitude faster than abiotic oxidation at low pH (Rawlings, 2002), i.e. below pH 4. Fe^{2+} oxidation consumes acid (reaction 1), whereas the precipitation of ferric iron (Fe^{3+}) as jarosite produces acid (reaction 2).

$$4\,Fe^{2+} + 4\,H^+ + O_2 \rightarrow 4\,Fe^{3+} + 2\,H_2O \quad (1)$$

Jarosite formation is common at low pH (<2) (Gramp et al., 2008) and involves the incorporation of a monovalent cation (A in reaction 2), such as ammonium (NH_4^+), potassium (K^+), sodium (Na^+) or hydronium (H_3O^+) (Gramp et al., 2008).

$$A^+ + 3\,Fe^{3+} + 2\,SO_4^{2-} + 6\,H_2O \rightarrow AFe_3(SO_4)_2(OH)_6 + 6\,H^+ \quad (2)$$

Microorganisms commonly used in iron oxidising bioreactors are *Acidithiobacillus* (*At.*) *ferrooxidans* and *Leptospirillum* spp. *At. ferrooxidans* has been reported to have a higher growth rate at redox potentials below 470 mV (vs. Ag/AgCl), whereas *Leptospirillum* spp. have a greater affinity for Fe^{2+} and are less sensitive to inhibition by Fe^{3+} (Rawlings et al., 1999). A two-stage reactor configuration was recently proposed by du Plessis et al. (2011) for biological Fe^{2+} oxidation and Fe^{3+} precipitation. Partial Fe^{2+} oxidation is achieved in

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the first reactor which is maintained at a relatively low redox potential (below 460 mV vs. Ag/AgCl) favouring the predominance of *At. ferroxidans*. Complete iron oxidation can then be achieved in the second reactor at a higher redox potential that favours *Leptospirillum* spp. Therefore, it is expected that utilising these two different groups of iron oxidisers in a two-stage bioreactor configuration could maximise the overall Fe^{2+} oxidation kinetics, thereby reducing reactor footprint (volume) and hence operational costs (du Plessis et al., 2011). The aim of this work was to demonstrate the transient performance response to imposed process parameters in a two-stage iron oxidising and precipitating airlift bioreactor (ALBR) which could be used for a variety of hydrometallurgical flow sheets where Fe^{3+} as an oxidant or iron removal may be required.

2. Materials and methods

2.1. Microbial inoculum

A mixed culture inoculum of iron-oxidising microorganisms was cultivated in a stirred tank reactor (1.5 L liquid volume) at room temperature (20–25 °C). The medium used for cultivating the inoculum and for continuous flow experiments as an influent was prepared in deionised water (pH 1.5 adjusted with H_2SO_4) and contained (g L^{-1}): 5.9 $CuSO_4 \cdot 5H_2O$, 6.7 $NiSO_4 \cdot 6H_2O$, 0.79 KCl, 0.37 $CaCl_2 \cdot 2H_2O$, 0.3 K_2HPO_4, 0.3 $(NH_4)_2SO_4$, 0.3 $MgSO_4 \cdot 7H_2O$, 1.26 $FeCl_2 \cdot 4H_2O$, 73 $FeSO_4 \cdot 7H_2O$ and 1 mL L^{-1} trace element solution (pH 1.5) containing (mg L^{-1}): 65 $MnSO_4 \cdot H_2O$, 144 $ZnSO_4 \cdot 7H_2O$, 76 $CoSO_4 \cdot 7H_2O$, 31 H_3BO_3 and 10 Na_2MoO_4. The concentrations of selected elements were 15 g L^{-1} Fe^{2+}, 1.5 g L^{-1} Cu, 1.5 g L^{-1} Ni, 0.55 g L^{-1} K, 0.1 g L^{-1} Ca and 1 g L^{-1} Cl. The bioreactor was inoculated with a combination of mesophilic, native, non-proprietary strains of *At. ferrooxidans*, *Leptospirillum (L.) ferrooxidans*, *L. ferriphilum*, *Acidimicrobium ferrooxidans*, *Sulfobacillus thermosulfidooxidans*, *Ferroplasma* sp., and uncharacterised native non-proprietary mixed iron-oxidising cultures.

2.2. Airlift bioreactors

A continuous-flow two-stage ALBR system was set up for iron oxidation and precipitation at room temperature (23 ± 2 °C). The system comprised of two 0.7 L ALBRs, each with its own separate settler (0.65 L) in series for sludge settling and recovery (Fig. 1). The ALBR system was initially started up as a one-stage system, consisting of one bioreactor and one settler only, with the second bioreactor and settler added following on day 10. The ALBRs were aerated with compressed air (1.5–4.5 L min^{-1}). The air flow was increased from 1.5 to 4.5 L min^{-1} on day 17. The reactors did not contain any growth support media, thereby facilitating ease of aeration and mixing, and allowing for jarosite precipitate to be continuously removed from the system. This also allowed the jarosite to act as both precipitation nuclei and help to retain cells within the reactors. Influent was fed with a peristaltic pump into the ALBR1 from which it flowed with gravity to the first settler. The overflow from the first settler was peristalticly pumped to the ALBR2 from which the solution flowed by gravity to the second settler. The effluent from the second settler flowed by gravity to effluent container. The hydraulic retention time (HRT) was decreased stepwise from 45 to 8 h in ALBR1 and from 50 to 10 h in ALBR2 to evaluate process performance (Fig. 2). Sludges from settlers 1 and 2 were recycled back into ALBR1 and ALBR2, respectively, at a flow rate of 100 mL min^{-1}. Excess sludge was intermittently removed from the settlers using a peristaltic pump connected to a high-performance programmable logic controller (PLC) (CompactRio® National Instrument™) and operated using a LabVIEW™ program at a rate of 0–300 mL day^{-1}. The sludge removal was started on days 22 and 38 from the first and second settler, respectively (Fig. 2).

2.3. Sampling

The ALBR influents and effluents were sampled for pH, redox potential, Fe^{2+} and soluble Fe analysis. Biomass present in the reactors was sampled for total cell counts and microbial community analysis. Solid concentrations in the bioreactors and settler underflows (i.e. slurries returned from settlers to ALBRs) were determined by total solids (TS) and total suspended solids (TSS) analyses. Sludge removed from the settlers was collected for analysing the elemental and mineralogical composition of the precipitates, scanning electron microscopy (SEM), and the analysis of particle size distribution, settling characteristics and filterability of the precipitates.

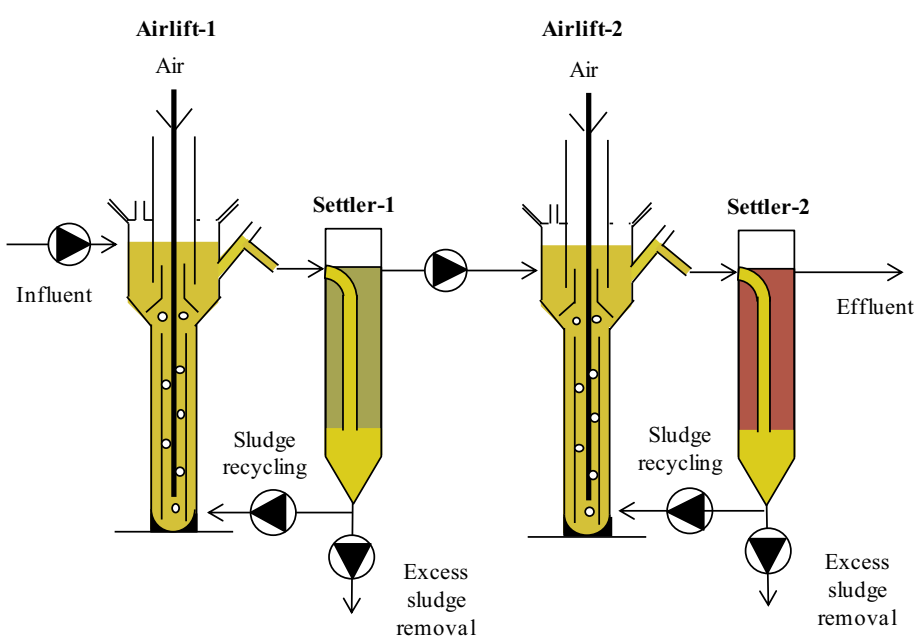

Fig. 1. Schematics for continuous-flow two-stage airlift reactor system.
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