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A novel experimental system for the study of microbial ecology and mineral leaching within a simulated agglomerate-scale heap bioleaching system

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ABSTRACT

Heap bioleaching systems are complex, with multiple sub-processes interacting at various scales within the heterogeneous reaction environment. This provides a challenge to determining the growth characteristics of micro-organisms and reaction characteristics of the mineral ore in a representative environment. The experimental system presented in this paper was designed to simulate heap bioleaching conditions using multiple, identically constructed agglomerate-scale mini-column reactors. Ore samples were prepared representatively as grab samples of a larger heap. Particle size distributions and agglomerate masses of the prepared ore samples were shown to be similar within acceptable variance and provided comparable surface areas for microbial colonisation and chemical reaction. The microbial abundance within the whole ore system was determined from effluent sampling for the planktonic population and the systematic and sequential sacrifice of identically operated mini-column reactors to determine the change in the ore-associated microbial population with time. Microbial colonisation and growth rate kinetics were determined from analysis of these populations. The growth curves obtained for the bulk flowing solution and ore-associated populations at the base case operating conditions were reproducible, within a 95% confidence interval.

1. Introduction

The treatment of low-grade, copper-bearing mineral sulphides by heap and dump leaching has emerged as a promising alternative process for metal extraction, responsible for around a quarter of global copper production [1]. Current studies are aimed at understanding the dissolution of low-grade chalcopyrite ores using heap bioleaching [2–5]. Although chalcopyrite has been identified as the most abundant copper-bearing mineral sulphide, low-grade chalcopyrite ores are difficult to treat using chemical dissolution because of the refractory nature of the mineral. Both ferric iron and hydronium ions have been found to leach chalcopyrite, with the rate of these reactions dependent on the ferric to ferrous iron ratio [6], galvanic interactions between mineral sulphides [7,8] and temperatures in excess of 50 °C [9,10].

Despite this, there is a lack of fundamental understanding of the role and interaction of key sub-processes that drive the mineral dissolution reactions. Micro-organisms play a vital role in the regeneration of the leaching agents for mineral sulphide dissolution, which may either be ferric iron or hydronium ions or both, with the rate of microbial oxidation of ferrous iron and sulphur species directly proportional to microbial abundance and activity. Typically, commercial bioheaps experience a significant lag period from the time of heap inoculation until enhanced mineral dissolution rates through microbial activity are observed [2]. This lag period may be due to microbial adaptation to the heap environment, determined by the extent of mineral–microbe interactions through microbial attachment to the mineral surface, microbial growth and colonisation of the heap [11].

In earlier studies of microbial population dynamics within copper mine dumps [12] and pilot scale heap leach columns [13], authors found significant variation in microbial abundance along the depth of the packed bed. At present, however, the PLS microbial concentration is assumed to indicate the microbial abundance and diversity within commercial heaps [14,15]. As a result, understanding of the microbial population dynamics within a heap is limited; with key questions regarding inoculation strategy, the choice of

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designed over natural inoculum and the adaptation and tolerance of inoculum, remaining unresolved [5,11,16].

Table 1 provides a summary of design conditions for selected lab-scale studies focused on microbial attachment and colonisation of low-grade ore in a heap environment. Microbial attachment studies on low-grade ore have been performed at the particle scale, in a biofilm reactor system [17,18], and at the agglomerate-scale, in glass column reactors loaded with geo-coated glass beads [19,20]. Authors observed preferential attachment to sulphide minerals; more specifically, pyrite over chalcopyrite and low-grade ore, and localised attachment to surface defects irrespective of the mineral, for mesophiles *Acidithiobacillus* (At.) *ferrooxidans* and *Leptospirillum* (L.) *ferriphilum* and the moderate thermophile, *Metallosphaera* (M.) *hakonensis*. For *Acidithiobacillus ferrooxidans*, the observed attachment rates were enhanced by enrichment of the culture on sulphur over ferrous iron as a growth substrate and increasing temperature [18,20–22]. Although the aforementioned studies were performed with maximum solution-ore contacting as opposed to an unsaturated environment, these findings may inform inoculation preparation and the role of pyrite in inoculum retention as well as heat generation through acid and ferric production.

Numerous studies have investigated the abundance and diversity of micro-organisms within the whole ore heap bioleaching system; specifically, the characterisation of microbial colonisation associated with the mineral has recently been studied using agglomerated crushed low-grade ore [23–30]. In the study by Tupikina et al. [23], multiple columns were each loaded with *ca.* 5 kg of crushed low-grade chalcopyrite ore which had been agglomerated with 0.1 M sulphuric acid. The reactors were then inoculated with a consortium of micro-organisms prepared from stock tank reactors, operated at temperatures ranging from mesophilic to moderately thermophilic (25–65 °C). In the earlier study [30], the authors monitored the diversity of the microbial populations in the PLS and those associated with the ore, as the temperature within the heap progressed from ambient to 60 °C, by sequentially sacrificing individual columns at pre-determined intervals throughout the experiment. The heating jacket surrounding the glass reactor was assumed to distribute heat uniformly throughout the ore bed, with a negligible effect on the temperature profile from solution and gas transport. A similar column reactor system together with a unique in-bed sampling technique and a larger scale, box reactor configuration (loaded with *ca.* 135 kg of dry ore) were used to study the effect of solution flow dynamics on microbial colonisation of the heap [28,31]. However, ore samples removed using the in-bed sampling technique were not representative of the bulk ore bed, with the removal of *ca.* 200 g of ore from a 4 kg ore bed. The technique was biased towards sampling the lower portions of the bed where fine particles may have accumulated, rather than allowing for random sampling. Collectively, the aforementioned studies of microbial colonisation in bioheaps have highlighted the significant difference in microbial diversity and population abundance between the PLS and ore-associated phases, the importance of the interstitial phase where microbial population and growth appear to be highest and the impact of fluid flow dynamics on the rate and extent of colonisation of a heap. The extension of these studies under fully representative conditions in a manner that facilitates the alteration of local conditions is, therefore, desirable.

Details of the design and commissioning of an experimental system capable of simulating heap bioleaching conditions at the agglomerate-scale, are presented in this paper. The experimental system, including the equipment, experimental approach, sampling protocol and analytical techniques, allowed for independent study of microbial attachment, colonisation of crushed low-grade ore and subsequent microbial growth kinetics in the flowing PLS and ore-associated phases. Validation of the underlying assumptions of the design of the experimental system were undertaken and base case experiments were performed in duplicate to determine the reproducibility of the results obtained. The agglomerate-scale experimental system described in detail in the current paper was used previously for the study of microbial growth rates and colonisation by *At. ferrooxidans*, used as a model micro-organism, on low-grade chalcopyrite ore [27].

2. Proposed experimental approach

Heap bioleaching systems may simply be described as unsaturated packed beds in which micro-organisms contribute to the gas–liquid–solid phase reactions and influence the physico-chemical reaction environment. However, each low-grade ore system is unique and dependent on dynamic components such as varying mineral compositions from separate deposits, non-uniform gas distribution through the heap that flows counter-current to irrigation solution dispersing down the heap *via* gravity and capillary forces, as well as diverse and dynamic microbial communities that are inherent to the ore body or have been introduced to the system. The resulting reaction environment is difficult to predict, and therefore, control.

The primary aim of the designed experimental system was to create identical heap bioleaching conditions within multiple agglomerate-scale column reactors such that the reaction kinetics and microbial population dynamics within each column were comparable to each other. Irrespective of the scale of experiments, the influence of sub-processes such as gas–liquid mass transfer, solution-ore contacting and mineral surface availability are expected to impact on microbial attachment, growth and propagation studies. In the design of this novel experimental system, a bottom-up approach was proposed in order to eliminate or minimise these effects on the observed microbial trends.

The bulk low-grade ore sample was pre-treated and processed into various size fractions. Each ore sample was then re-constructed to represent the particle size distribution (PSD) of the bulk sample. To minimise the effects of gas–liquid mass transfer, solution contacting and the resulting non-uniform heat balance across the ore bed, the mass of each ore sample was chosen to occupy only a small volume (one-fifth) of the mini-column reactor. In this way, multiple agglomerate-scale ore samples of identical PSD and mass were created, essentially grab samples of a larger heap.

A single experiment consisted of multiple mini-column reactors. The ore samples were sterilised using γ-irradiation (50 kGy) to prevent the indigenous microbial populations from influencing the outcome of the experiments. As such, upon addition of a well-characterised inoculum, in this case a pure *At. ferrooxidans* culture, subsequent microbial growth and activity may be directly associated with that of the known culture. In addition, since micro-organisms favour initial attachment to mineral surface defects [18], and heat sterilisation may alter the mineral surface either through thermal stress or the deposition of secondary mineral phases [32,33], gamma (γ-) irradiation was chosen over autoclaving for ore sterilisation. Each ore sample was acid agglomerated, creating a narrower particle size distribution and uniformly wetted environment for microbial colonisation, and thereafter, loaded into the mini-column reactors between two sets of glass beads. The mini-column reactor configuration was also designed to enhance solution-ore contacting by uniformly dispersing irrigation solution over the top of the packed bed. This irrigation scheme served to minimise the effects of poor gas-solution-ore contacting on microbial population dynamics. The residence time distribution (RTD) study was used in conjunction with compartment models to diagnose the behaviour of solution within the packed bed reactor, providing insight into the extent of mixing and the proportion of stagnant to flowing solution present.