



The complexity of biosorption treatments for oxyanions in a multi-element mine effluent



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ABSTRACT

Selenium (Se) is a contaminant in effluents from coal mines and coal-fired power stations, where it is encountered as the oxyanion selenate (SeO_4^{2-} , hereafter Se^{VI}). Se^{VI} can be removed from solution with Fe-treated biosorbents, but the efficacy of these treatments in effluents with multiple contaminants is unclear. This study investigates the interactions between Se^{VI} and the oxyanions SO_4^{2-} and NO_3^- . We produce a sustainable biosorbent, *Gracilaria* Modified Biochar (GMB), by treating a waste product generated after the commercial extraction of agar from cultivated seaweeds with ferric chloride (FeCl_3) and converting it to biochar through pyrolysis. We then test interactions between Se^{VI} and competing oxyanions in mock solutions and a real-world mine effluent with high concentrations of SO_4^{2-} and NO_3^- . GMB immediately removed 98% of the Se^{VI} from the mock solution, but only 3% from the mine effluent with the same initial Se^{VI} concentration. Notably, 83–89% of the Se^{VI} was removed by GMB when concentrations of Se^{VI} and SO_4^{2-} were less than or equimolar in mock solutions. Higher concentrations of SO_4^{2-} reduced the uptake of Se^{VI} . There was no interference from NO_3^- on the biosorption of Se^{VI} . GMB is a successful biosorbent for Se^{VI} , however, high concentrations of SO_4^{2-} will compromise the biosorption of Se^{VI} .

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1. Introduction

Biosorption of contaminants from industrial effluents using dried algal biomass is a promising approach to the sustainable treatment of industrial effluents (Gadd, 2009). However, one limitation to the use of algal biomass as a biosorbent is that dried algae are only effective at adsorbing a sub-set of the many elements that are common in industrial effluents (Kidgell et al. 2014). One critical metalloid often found in effluents from coal mining and processing facilities is the oxyanion selenate (Se^{VI}) (Chapman et al. 2009). Se^{VI} is toxic at concentrations slightly in excess of essentiality due to its tendency to impair the function of enzymes in vertebrates (Hamilton, 2004; Yang et al. 2010). Se^{VI} , like most metalloids, is difficult to remove from effluents as it is encountered as an oxyanion (Sappington, 2002; Mondal et al. 2004) for which passive biosorbents have a low affinity (Mondal et al. 2004; Ghazvini and Mashkani, 2009; Figueiredo and Quintelas, 2014). However, algal-based biosorbents can be modified by a treatment of ferric

chloride (FeCl_3) followed by pyrolysis to yield an Fe-treated biochar with a high biosorption capacity for Se^{VI} in solution (Roberts et al. 2014).

One potentially abundant source of biomass for biosorption applications is the use of the waste biomass produced in the commercial extraction of agar from cultivated red seaweeds. The main genus of algae grown for the extraction of agar is *Gracilaria*. Commercial cultivation of *Gracilaria* is increasing rapidly worldwide, particularly in Indonesia where more than 500,000 tonnes are produced annually (Sibeni and Calderini, 2012). The granular material remaining after the extraction of agar is currently stored as a waste with no economic value or further use (Seo et al. 2010). However, Roberts et al. (2014) demonstrated that the waste can be used to produce a biosorbent with a strong capacity to adsorb Se^{VI} by treating the *Gracilaria* extraction waste with FeCl_3 . The biosorbent had high removal rates of Se, both as selenite (Se^{IV}) and Se^{VI} , within the course of a few hours. Furthermore, the adsorption capacity was increased 3-fold once the FeCl_3 -loaded biomass was converted into biochar by pyrolysis. Biosorption of Se^{VI} by the final product, *Gracilaria* Modified Biochar (GMB), was pH and temperature independent, negating the need to adjust both the

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pH and the temperature prior to biosorption of any industrial effluent and significantly simplifying the method (Roberts et al. 2014).

However, one limitation of this biosorption process, and of analogous processes, is that it is typically not assessed using real-world effluents. Rather, research has focused on the biosorption of elements from simple mock solutions with single elements (Gadd, 2009). The behavior of a biosorbent in real-world effluents, such as mine effluents, is much more complex than the behavior in mock solutions (Kidgell et al. 2014). The advantage of examining biosorption capacities using real-world effluents is that it offers insight into potential interactions among elements (Vijayaraghavan and Joshi, 2013) and specifically the differences between metals and metalloids (Kidgell et al. 2014). The presence of non-target elements with similar properties may impede the uptake of the target element due to competition for common uptake sites (Brix et al. 2001; Hamilton, 2004).

Sulfate (SO_4^{2-}) is a common oxyanion in effluent waters from coal mines, particularly in areas where coal has a high S content (Rodriguez et al. 2012). SO_4^{2-} is known to interfere with the bioremediation of Se^{VI} by passive biosorbents (Lalvani, 2004; Yamani et al. 2014). For example, the bioremediation of Se^{VI} in effluent waters is reduced in the presence of SO_4^{2-} , irrespective of the type of biosorbent used (Lalvani, 2004; Yamani et al. 2014). Alumina, chitin, activated carbon and impregnated chitosan beads all have a reduced capacity for the biosorption of Se^{VI} in the presence of SO_4^{2-} (Lalvani, 2004; Dobrowolski and Otto, 2012; Yamani et al. 2014). Nitrate (NO_3^-) is another common constituent of mining effluents, especially in effluents where ammonium nitrate and fuel oil (ANFO) explosives are used on site, which can interfere with the biosorption of Se^{VI} . For example, the biosorption of Se^{VI} from agricultural drain water, using soil as a biosorbent, was reduced when the concentrations of NO_3^- was 5 mg L^{-1} or more (Bailey et al. 2012). While it is clear that the presence of other oxyanions interfere with the uptake of Se^{VI} by a biosorbent (Lalvani, 2004; Bailey et al. 2012; Dobrowolski and Otto, 2012; Yamani et al. 2014), it is uncertain whether the iron-based biosorbents have an affinity for other oxyanions that interferes with the uptake of Se^{VI} . Answering this question will require experiments that first examine the biosorption capacity of an iron-based biosorbent to oxyanions in mock solutions and then examine the biosorption capacity of the iron-based biosorbent in a complex real-world effluent with multiple oxyanions.

This study examines the comparative ability of GMB to remove Se^{VI} from a single-element mock solution and a mine effluent that is contaminated with the potentially inhibiting oxyanions SO_4^{2-} and NO_3^- . The specific aims of this paper are to 1) compare the biosorption capacity of GMB for Se^{VI} in both a single-element mock solution and in a real-world mine effluent, 2) compare the biosorption capacity of GMB for target (Se^{VI}) and non-target (SO_4^{2-} and NO_3^-) constituents of mock solutions, and 3) determine the biosorption capacity of the GMB for Se^{VI} as a function of differing relative concentrations of non-target (SO_4^{2-} and NO_3^-) compounds to quantify the interactions between elements. This information will establish the performance of the biosorbent under real-world mine conditions as a step towards understanding the industrial application of a promising Se^{VI} treatment.

2. Materials and methods

2.1. Real-world mine effluent

A sample of a mine effluent was obtained from a coal mine operated by Anglo American Coal (Canada). The sample was collected in June 2013 from an effluent sedimentation pond and

cold-shipped to James Cook University in Townsville, Australia. The effluent was stored in a fridge at 4°C until use. The concentration of total Se was $108 \mu\text{g L}^{-1}$ and speciation analyses showed this to be 99% Se^{VI} with a small amount of Se^{IV} . The elemental composition of the effluent is shown in Table S1.

2.2. Biomass preparation

Gracilaria extraction waste was obtained from AgarIndo Bogatama in Indonesia (for details see Roberts et al. 2014). Prior to use as a biosorbent, the *Gracilaria* extraction waste was rinsed with MilliQ water and dried in an oven at 60°C for 24 h. The dry biomass was then loaded with a 5% FeCl_3 solution (Sigma Aldrich) at a rate of $25 \text{ g biomass L}^{-1}$ for 24 h at 15°C before being rinsed twice with MilliQ water to remove excess FeCl_3 . After a second drying in the oven at 60°C for 24 h, the FeCl_3 -loaded biomass was converted into GMB by a pyrolysis process where the biomass was pyrolysed at a temperature of 450°C for 1 h while being continuously purged with N_2 gas at a rate of 4 L min^{-1} (Bird et al. 2011). The biochar was then cooled to room temperature under continuous N_2 flow and stored for use.

2.3. Performance of GMB in Se^{VI} mock solution and mine effluent

A biosorption experiment was performed to compare the performance of GMB in a Se^{VI} mock solution and a real-world mine effluent containing Se^{VI} and the oxyanions NO_3^- and SO_4^{2-} . The concentration of Se^{VI} in the mock solution was prepared to reflect that in the effluent ($108 \mu\text{g L}^{-1}$) at $100 \mu\text{g L}^{-1}$. Total Se was measured to allow a direct comparison of performance in the two solutions. Since both solutions contained 99% Se^{VI} , data for total Se are hereafter referred to as Se^{VI} .

A 1 g L^{-1} Se^{VI} solution was made by mixing Na_2SeO_4 (Sigma Aldrich) with MilliQ water after which the solution was diluted to $100 \mu\text{g L}^{-1}$. The mock solution and the mine effluent were treated multiple times with GMB to measure sequential reductions in dissolved Se^{VI} after multiple deployments of biosorbents. Each treatment involved exposing the Se^{VI} source (mock or effluent) to a number of deployments of new GMB (1, 2, 3, 4, 6, 8, 10, 12 or 14 sequential deployments). Each replicate consisted of a 250 ml container containing 1 g GMB and 100 ml of mock Se^{VI} solution or mine effluent. The GMB was exposed to the mock Se^{VI} solution or effluent for 1 h at 15°C in a shaker incubation cabinet (100 rpm). After 1 h the solution was filtered using a $75 \mu\text{m}$ filter mesh and transferred into a new container with 1 g of new GMB. The final solution for each treatment was collected for analysis of total Se after the final deployment. The solution was filtered out from the GMB as described above, after which it was filtered with a $0.45 \mu\text{m}$ syringe filter and collected in falcon tubes. Stock solutions were sampled at the start of the experiment to determine initial starting concentrations.

2.4. Comparative assessment of the biosorption capacity of GMB for Se^{VI} , SO_4^{2-} and NO_3^-

A biosorption experiment was conducted to evaluate the relative biosorption capacity of the GMB for the target Se^{VI} and non-target compounds SO_4^{2-} and NO_3^- in equimolar solutions. Biosorption capacity, in this study, is defined as q , with a higher q signifying a higher biosorption capacity of the GMB to an oxyanion and the highest biosorption capacity referred to as q_{max} . q is expressed as the amount of an element removed per gram biosorbent (e.g. $1.2 \mu\text{g Se}^{\text{VI}} \text{ g}^{-1}$ GMB).

To identify the biosorption capacity of GMB for each of the compounds, a q_{max} kinetic experiment was undertaken. Seven concentrations of Se^{VI} , SO_4^{2-} and NO_3^- were prepared (0.01, 0.05, 0.1,

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