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Characterization of colloidal arsenic at two abandoned gold mine sites in Nova Scotia, Canada, using asymmetric flow-field flow fractionation-inductively coupled plasma mass spectrometry

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ABSTRACT

Asymmetric flow-field flow fractionation-inductively-coupled plasma-mass spectrometry was used to determine whether colloidal arsenic (As) exists in soil pore water and soil extract samples at two arsenic-contaminated abandoned gold mines (Montague and Goldenville, Nova Scotia). Colloidal arsenic was found in 12 out of the 80 collected samples (=15%), and was primarily associated with iron (Fe) in the encountered colloids. The molar Fe/As ratios indicate that the colloids in some samples appeared to be discrete iron-arsenic minerals, whereas in other samples, they were more consistent with As-rich iron (oxy)hydroxides. Up to three discrete size fractions of colloidal As were encountered in the samples, with mean colloid diameters between 6 and 14 nm. The pore water samples only contained one size fraction of As-bearing colloids (around 6 nm diameter), while larger As-bearing colloids were only encountered in soil extracts.

Introduction

Colloids are ubiquitous in the environment and typically composed of inorganic mineral phases, organic biopolymers or microorganisms <1 μm in diameter (Kretzschmar and Schäfer, 2005). Aquatic colloids can be small enough to remain suspended by Brownian motion for long periods of time in the absence of aggregation; e.g., colloids with 10 and 1 nm diameter can remain suspended for >20 and 200 years, respectively (Wilkinson and Lead, 2007). Colloidal material is present in all natural surface, soil pore and ground waters, and is significant for

the cycling and transport of contaminants, including metalloids (Kretzschmar and Schäfer, 2005). The presence, stability and mobility of colloids may impact the transport/mobility of and risk associated with contaminants, so failure to distinguish between colloidal and dissolved fractions can lead to inaccurate estimations of contaminant mobility and risk. When assessing the practical importance of colloidal contaminant transport, one must consider (1) the colloidal concentration, including mass concentration and the total number of colloids, (2) the size and shape of the colloids, including size distribution and particle morphology, (3) the colloidal composition, including mineralogy,

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elemental makeup and whether it includes organic components and (4) the surface characteristics, including surface charge, area and sorption capacity (Kretzschmar and Schäfer, 2005).

Arsenic, a toxic and carcinogenic metalloid, is ubiquitous in the environment (Dale and Freedman, 1982) due to geogenic occurrence and anthropogenic mobilization (Wang and Mulligan, 2006). Arsenic in natural waters exists predominantly as arsenate, As(V), and arsenite, As(III) (Henke, 2009). Water samples are typically filtered to <0.2 or <0.45 μm , and all of the arsenic in the filtrate is traditionally considered dissolved (Wilkinson and Lead, 2007), despite the fact that truly dissolved As species like As(III) and As(V) are <1 nm. Arsenic species between 1 and 200 (or 450) nm should therefore be considered colloidal rather than truly dissolved (Leshner et al., 2009; Dubascoux et al., 2008). To date, there is very limited evidence of the existence of colloidal arsenic in the environment. A laboratory study on arsenic leaching from mine tailings columns found iron-based colloids to be the primary means of arsenic transport; colloidal mineralogy was determined using X-ray adsorption techniques (Slowey et al., 2007). A second laboratory study investigated the influence of dissolved organic matter (DOM) on arsenate binding to iron (oxy)hydroxides, and found that arsenate binding to organic colloids was small or negligible in the absence of Fe(III), but increased significantly when Fe(III) was present (Bauer and Blodau, 2009). Finally, a field study demonstrated conclusively, using ultrafiltration and extended X-ray absorption fine structure (EXAFS) spectroscopy, that iron-based arsenic colloids exist in a pond receiving acid mine drainage. These arsenic colloids were present at ppm concentrations, had a diameter between 1.3 and 5 nm and accounted for $>50\%$ of the total "dissolved" arsenic (Zanker et al., 2002). These results generally confirm the known importance of iron (oxy)hydroxide minerals for arsenic cycling, due to their strong binding interaction (Henke, 2009).

Arsenic background levels in Nova Scotia surface soils are high in comparison to the Canadian soil guideline of 12 mg/kg (Wong et al., 1999), due to glacial transport during the last ice age, resulting in the crushing of mineralized bedrock. Consequently, geogenic arsenic in ground water is also elevated frequently, and many wells in Nova Scotia are naturally contaminated with arsenic (Goodwin et al., 2008) beyond the Canadian drinking water guideline of 10 $\mu\text{g/L}$ (Wong et al., 1999). Additional arsenic contamination problems can arise from historical gold mining activities, which occurred extensively throughout Nova Scotia from the 1860s to the 1940s (Wong et al., 1999). Gold is typically found in quartz veins, which host arsenopyrite (FeAsS), the most common arsenic-bearing mineral (Henke, 2009). After the mining activities ended, approximately three million tonnes of arsenic-rich tailings had been produced, and were deposited on the surface of Nova Scotia mine sites and in local swamps, streams and rivers (Dale and Freedman, 1982).

This study aims to test if colloidal arsenic exists in waters in contact with abandoned gold mine tailings at two sites in Nova Scotia, Canada. Samples were collected at Montague and Goldenville. Since high arsenic concentrations have been found in such waters, concern exists regarding its mobility and the resulting risk. The physical and chemical speciation of arsenic in these waters could have a major influence on its

mobility and (potential) transport from the sites. To determine whether colloidal As species occur at these sites, asymmetric flow-field flow fractionation-inductively-coupled plasma-mass spectrometry (AsFI-FFF-ICP-MS) was used for the first time in this study to characterize arsenic-bearing colloids with respect to their size and elemental composition.

1. Methods and analytical instrumentation

1.1. Sampling sites (Nova Scotia, Canada)

Montague is located on the outskirts of Dartmouth, and is used for recreation purposes. Mining and milling took place from 1863 to 1940 and generated approximately 132,000 tonnes of mine tailings (Dale and Freedman, 1982). Mitchell Brook runs adjacent to the tailings, and tailings materials have been moved over 2 km downstream by it. Goldenville is located in Guysborough County; mining and milling occurred here from 1862 to 1941 and generated approximately 586,000 tonnes of tailings. These tailings occupy an area of 0.25 km^2 that is adjacent to the former mill sites. Tailings extend more than 8 km along the flood plain of Gegogan Brook (Wong et al., 1999). Maps of the sampling areas are presented in the supplemental information (Appendix A Fig. S1).

1.2. Sample collection and pretreatment

The soils at the two mine sites are strongly disturbed and contain large and variable amounts of weathered tailings materials. Some representative end members, general types of mineralogies produced at the end of the various tailings weathering processes, include calcium-rich tailings at Goldenville, containing on average 0.145% arsenic and 0.47% calcium (Kavalench, 2010), hardpan tailings at Montague, characterized by a large amount of scorodite and low pH, overlain with a layer of hardpan, and containing up to 33% arsenic and 23% iron, with molar ratios ranging from 0.85–1 (DeSisto, 2008), and wetland tailings (reducing; rich in organic matter) at Montague. Samples of these end member types were collected at 10 cm intervals from the surface for the calcium-rich and hardpan tailings, and from 25 and 75 cm depth for the wetland samples. Additional samples, representing mixtures of native soils and weathered tailings materials, were collected at various locations from each mine site.

Eighty samples were collected during four sampling campaigns in May 2009, March 2011, May 2011 and June 2012. The exact sampling locations are shown in the maps in the supplemental information (Appendix A Fig. S1). Surface water samples were collected using 1 L high density polyethylene (HDPE) bottles (Fisher Scientific, Nepean, ON). Pore water samples from the saturated zone (*i.e.*, ground water) of soils were collected using suction PVC lysimeters (Soil Moisture Equipment Corporation, Santa Barbara, CA) and stored in 8 mL HDPE bottles (Fisher Scientific, Nepean, ON) with no headspace. Pore water from the unsaturated zone was extracted from soil samples by filtration. Soil samples were collected using 125 mL HDPE bottles (Fisher Scientific, Nepean, ON) and 50 mL plastic test tubes (Sarstedt, Montreal, QC). From these soil samples, aqueous extracts

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