



Biogeochemical controls on the speciation and aquatic toxicity of vanadium and other metals in sediments from a river reservoir



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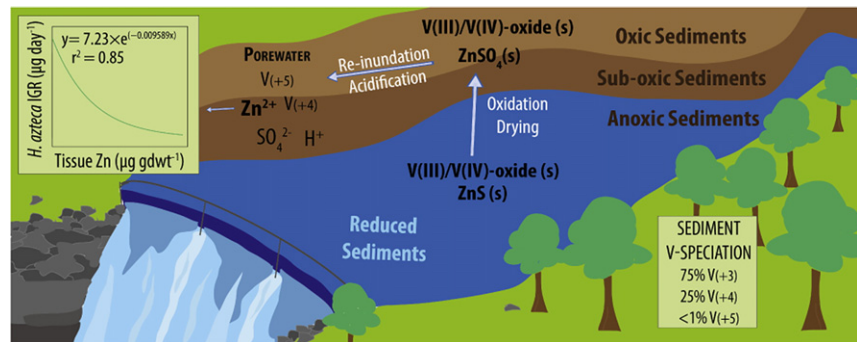
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HIGHLIGHTS

- Fate and effects of vanadium in hydrodynamic aquatic ecosystems are assessed.
- Sediment speciation led to low bioavailability of sediment V and thus no effects to organisms.
- Water level fluctuation and sediment oxidation led to Zn-release, affecting *H. azteca* growth.
- It is important to consider V sediment speciation when assessing environmental risk.

GRAPHICAL ABSTRACT



ARTICLE INFO

Article history:

Received 2 June 2017

Received in revised form 9 August 2017

Accepted 14 August 2017

Available online xxx

Editor: F.M. Tack

Keywords:

Vanadium toxicity
Vanadium speciation
Sediment metal toxicity
Bioavailability
Zinc release

ABSTRACT

Effects of hydrologic variability on reservoir biogeochemistry are relatively unknown, particularly for less studied metals like vanadium (V). Further, few studies have investigated the fate and effects of sediment-associated V to aquatic organisms in hydrologically variable systems. Our primary objective was to assess effects of hydrologic manipulation on speciation and toxicity of V (range: 635 to 1620 mg kg⁻¹) and other metals to *Hyalella azteca* and *Daphnia magna*. Sediments were collected from a reservoir located in a former mining area and microcosm experiments were conducted to emulate 7-day drying and inundation periods. Despite high sediment concentrations, V bioavailability remained low with no significant effects to organism survival, growth, or reproduction. The lack of V toxicity was attributed to reduced speciation (III, IV), non-labile complexation, and sorption to Al/Fe/Mn-oxyhydroxides. Zinc (Zn) increased in surface and porewater with inundation, for some sediments exceeding the U.S. EPA threshold for chronic toxicity. While no effects of Zn to organism survival or growth were observed, Zn body concentrations were negatively correlated with *H. azteca* growth. Results from this study indicate that V bioavailability and environmental risk is dependent on V-speciation, and V is less influenced by hydrologic variability than more labile metals such as Zn.

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1. Introduction

Human alteration of natural aquatic ecosystems using water control structures often has collateral effects to surrounding habitats. Beneficial ecosystem services can be impacted, such as fisheries health, water

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quality, carbon sequestration, and stream bank stabilization (Jager and Smith, 2008; Richter et al., 2003). Hydrologic variability caused by reservoir management may lead to a redistribution of sediment and altered biogeochemical cycling at the sediment-water interface (SWI) (Carvalho et al., 1998; Ney, 1996; Skalak et al., 2016). This is particularly true for the geochemistry of redox sensitive transition metals with multiple oxidation such as vanadium (V) (De Jonge et al., 2012).

Published studies on V ecotoxicology are limited, and consist mostly in presenting sources and surface water toxicity of V (Environment Canada, 2010; Irwin et al., 1997; Nriagu, 1998). Vanadium is toxic to organisms at elevated concentrations due to phosphate mimicry in the V(V)-oxidation phase, leading to inhibition of various phosphohydrolases and enzymes (Chasteen, 1983). *Hyaella azteca* and *Daphnia magna* have a lethal concentration (LC₅₀) of 400 µg L⁻¹ and effects concentration (EC₁₀) of 1000 µg L⁻¹, respectively, for surface water (Environment Canada, 2010). Recently, a new species sensitivity distribution (SSD) derived LC₅₀ of 0.64 mg L⁻¹ and HC5 of 0.05 mg L⁻¹ has been reported for midges in surface water (Schiffer and Liber, 2015). No similar values have been published for porewater or sediment. Release into the environment can occur from mine drainage, as combustion byproducts (fossil fuels, paper mills, smelting), and as a common impurity in fertilizers, with V production increasing at approximately 9% annually (Irwin et al., 1997; Perles, 2012).

Vanadium is chemically complex, influenced by a triad of pH, redox, and coordination chemistry. Upon introduction into the aquatic environment, V can be present in three oxidation states. In surface and oxygenated porewater oxo- and oxyhydroxide-V compounds are the most abundant and biologically available. Some examples include (1) vanadates, H₃V(V)O₄, H₂V(V)O₄⁻, HV(V)O₄²⁻, and V(V)O₄³⁻; (2) oxyhydroxides, V(V)O₂(OH)₂⁻, V(IV)OOH⁺; and vanadyl, V(IV)O²⁺. Of these, vanadate is thought to be the most toxic and most labile species, particularly in alkaline and oxic waters due to its anionic nature. Vanadyl can also be toxic to organisms when dissolved, although lower oxidation states (V(IV), V(III)) are mostly found in sediment phases, they may be rapidly transformed to aqueous vanadate via hydrolysis or oxidation (Irwin et al., 1997). Previous studies focus on V toxicity in surface water, with relatively little focus on sediment toxicity, sediment-water interactions, or V speciation at the SWI.

Porewater V bioavailability to aquatic organisms is partially controlled by the proportion of non-labile to labile forms (i.e. Fe/Mn oxyhydroxides, sulfide, and organic matter) present in sediments (Environment Canada, 2010). Selective extractions show the largest fraction of total V in sediments is typically non-labile, being associated with one of several mineral forms, accounting for 50–95% of total V (Abollino et al., 2006; Terzano et al., 2007). The largest labile fraction of total V is often adsorbed on the surface of Al/Fe/Mn oxyhydroxides (2–40%) (Harita et al., 2005; Larsson et al., 2013; Mikkonen and Tummavuori, 1994; Terzano et al., 2007). Although vanadyl sulfides and sulfates represent a minor V-fraction in most sediments (<1%), they can form in reducing conditions or through biological uptake (Wanty and Goldhaber, 1992). The fraction of V bound to organic matter is thought to be low (<10%) in most sediments as predominately reduced V(III) or V(IV) (Abollino et al., 2006). Bacteria and meso-/thermophilic methanogens can also mediate reduction of vanadate (Carpentier et al., 2003; Ortiz-Bernad et al., 2004; Zhang et al., 2014). Investigating the biogeochemistry of these V-phases is important to this study, as they are likely to influence V bioavailability.

While V-speciation and toxicity was the main focus of this study, it is important to consider the effects of co-occurring metals. At many sites metals are co-occurring and can vary in their relative bioavailability. Sediment chromium (Cr), nickel (Ni), and zinc (Zn) concentrations often exceeded probable effects concentrations (PEC) in the studied reservoir (Buchman, 2008). Dissolved Ni, and Zn species are primarily toxic to organisms as divalent cations and Cr(VI) toxicity (and carcinogenic properties) have been demonstrated for aquatic life (Gambrell et al., 1991). Within sediments, these metals are sensitive to redox changes,

pH alteration, and microbial activity. When exposed to sediments, metals can be adsorbed on Fe/Mn oxyhydroxides, precipitate with sulfide or carbonate discrete phases, or react with organic matter (Gambrell, 1994). There is currently no data on V as a multiple stressor with divalent metals, so it is unclear whether toxicological effects would be additive or antagonistic.

Our primary objective was to determine the effect(s) of sediment drying and re-inundation on V speciation and potential toxicity of elevated-metal sediments. We expected that porewater metal concentrations (including V) would increase with inundation of oxidized sediments and subsequent reduction to lower oxidation states would decrease bioavailability. Toxicity testing using *D. magna* and *H. azteca* provided an indication of bioavailable metal release associated with sediment oxidation. This study provides predictive toxicology tools for hydrologically variable systems and can inform reservoir management for improved biological health and water quality.

2. Methods

2.1. Site selection and sediment collection

A Ponar dredge was used to collect surface sediments (approximately top 8 cm) from embayments in Lake Catherine (Hot Springs, Arkansas, USA) near the outlets of two creeks [Wilson Creek (WIL) and Indian Springs Creek (SPN)] which drain a former V mine site (Supporting information (SI), Fig. S1). The mine and on-site refinery produced approximately 4.75 million tons of vanadium ore averaging 1.2% V₂O₅ predominately from the mineral Novaculite. Mining began in 1967 and ended in 1989. The site is currently undergoing reclamation, which started in 1996. A reference sediment (REF) was collected from an upstream embayment within Lake Catherine with similar bathymetric and hydrologic characteristics. The sites are affected by annual reservoir drawdown exposing mudflats in October, with re-inundation in spring. A laboratory control sediment (CNTL) was collected from Raisin River in Manchester, Michigan, USA. Within 24-hours of collection, the headspace of sediment containers was nitrogen purged and stored at 4 °C until experiments commenced. Additional information on site locations and sediment chemical properties are provided in SI, *Supplemental Results: Sediment type differences*, and Table S1.

2.2. Experimental design

Sediment-filled microcosms were used to conduct wetting/dry experiments to test redox effects on chemical and biological endpoints. The polycarbonate microcosms were fitted with Rhizon samplers at 1 and 3 cm sediment depths to facilitate porewater sampling (Fig. S2). Each microcosm was equipped with a small bottom drainage port with 250-µm mesh to reduce sediment loss. Three replicate microcosms for each sediment type were flooded for several days prior to starting the experiment. The experiment began with 7-days of sediment drying, followed by an 11-day inundation. To facilitate sediment drying, surface water was removed with a syringe and the bottom drainage hole was opened. During inundation, the drainage hole was closed to prevent leaching and water was introduced for 4 min every 12 h at a rate of 1.55 ± 0.5 cm³ s⁻¹ to emulate rainfall. Input water chemical composition had an average hardness of 152.9 ± 1.4 mg L⁻¹ CaCO₃, dissolved oxygen (DO) of 6.4 ± 0.2 mg L⁻¹, pH of 7.5 ± 0.1, conductivity ranging from 300 to 360 µS cm⁻¹, and temperature of 20 ± 0.5 °C.

2.3. Aqueous analyses

Laboratory analyses included total metal content, V-speciation, and general chemical parameters of surface water, porewater, and sediment. Surface water general chemical parameters included DO, pH, Eh, conductivity, and temperature measured on days 1, 7, and 11 for all microcosms, with the exception that Eh was not measured on day-1 due to

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