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Nickel toxicity to benthic organisms: The role of dissolved organic carbon, suspended solids, and route of exposure

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ABSTRACT

Nickel bioavailability is reduced in the presence of dissolved organic carbon (DOC), suspended solids (TSS), and other complexing ligands; however, no studies have examined the relative importance of Ni exposure through different compartments (water, sediment, food). *Hyalella azteca* and *Lymnaea stagnalis* were exposed to Ni-amended water, sediment, and food, either separately or in combination. Both organisms experienced survival and growth effects in several Ni compartment tests. The DOC amendments attenuated *L. stagnalis* Ni effects (survival, growth, and ^{62}Ni bioaccumulation), and presence of TSS exposures demonstrated both protective and synergistic effects on *H. azteca* and *L. stagnalis*. ^{62}Ni trophic transfer from food to *H. azteca* and *L. stagnalis* was negligible; however, bioaccumulating ^{62}Ni was attributed to ^{62}Ni-water (^{62}Ni flux from food), ^{62}Ni-TSS, and ^{62}Ni-food. Overall, *H. azteca* and *L. stagnalis* Ni compartment toxicity increased in the following order: Ni-water >> Ni-sediment >> Ni-all (water, sediment, food) >> Ni-food.

1. Introduction

Metals enter aquatic environments through both natural sources (rock weathering, volcanoes, biogenic processes), and anthropogenic sources (mining, smelting, fossil fuel combustion) (Callender, 2003; Sen Gupta and Bhattacharyya, 2008). Once these metals enter aquatic ecosystems, they can become bioavailable to organisms, and can cause toxicity (Callender, 2003). Nickel is one such metal, and its ecotoxicology is experiencing recent attention in the literature (Costello et al., 2011, 2012; Nguyen et al., 2011; Vangheluwe et al., 2013; Croteau and Luoma, 2008). Relating the relative aquatic toxicity of Ni to the major exposure compartments (water, sediment, food) studies has received limited attention in the scientific literature. However, authors have characterized Ni toxicity in single compartments (water, sediment, food), and these have ranged from survival to bioaccumulation effects (Watras et al., 1985; Nowierski et al., 2005; Doig and Liber, 2006; Meyer et al., 2007; Croteau and Luoma, 2008, 2009; Cloran et al., 2010).

There are several factors affecting the bioavailability Ni in water and sediments, and these include: dissolved organic carbon (DOC), total suspended solids (TSS), acid volatile sulfides (AVS), Fe/Mn (oxy)hydroxides, and organic carbon (OC) present in water and sediments (Pyle et al., 2002; USEPA, 2005; Cloran et al., 2010). Metals, including Ni, have an affinity to these variables (DOC, TSS, OC, and AVS), and it is imperative to examine these variables for our understanding of Ni toxicity in water and sediments (USEPA, 2005; Sen Gupta and Bhattacharyya, 2008; Cloran et al., 2010). DOC is an important ligand for metals, and increased DOC concentrations have been shown to reduce metal bioavailability in waterborne exposures. However, metal toxicity is not always attenuated with increased DOC, and appears to be organism specific (De Schamphelaere et al., 2004; Doig and Liber, 2007, 2006; Cloran et al., 2010).

Once metals are associated with food they can become a dietary route of exposure (Doig and Liber, 2006). When determining metal uptake by organisms, others (Watras et al., 1985; Courtney and Clements, 2002; Wilding and Maltby, 2006; Nguyen et al., 2012) have spiked metals with food to follow organism toxicity and bioaccumulation. Croteau and Luoma (2008, 2009) have shown that metals either adsorb or absorb onto food, and were readily accumulated by *Lymnaea stagnalis* during feeding tests. Croteau and Luoma (2008) showed that *L. stagnalis* tissue content was higher in Cd, Cu, and Ni during feeding rather than water-only acute metal exposures. Organismal metal bioaccumulation can have food chain

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and food web effects that potentially can alter ecological function of a system (Hare, 1992). The objectives of this study were to (1) discern *Hyalella azteca* and *L. stagnalis* toxicity in multiple compartments (water, sediment, food); (2) determine the role that DOC and TSS amendments have on Ni bioavailability in these multiple compartments; (3) and whether the stable isotope ^{62}Ni bioaccumulates during feeding in these multiple compartment exposures.

2. Methods

2.1. Laboratory experimental design

L. stagnalis and *H. azteca* were exposed to a series of Ni-amended compartments (Ni-water, Ni-sediments, Ni-food, Ni-all (water, sediment, food)), and each compartment received overlying water amendments of TSS (425 μm sieved WD sediment), DOC (Aldrich Humic acid), or none (SI, Fig. S1). Each organism was exposed simultaneously on two sediment types collected from: Greenville Creek (GC), Ohio, USA (low AVS and OC), and Warden Ditch (WD), Ohio, USA (high AVS and OC). The four Ni exposures were: (Ni-water) Ni-amended water + clean sediment + clean food, (Ni-sediment) clean water + Ni-amended sediments + clean food, (Ni-food) clean water + clean sediment + ^{62}Ni-amended food, (Ni-all) Ni-amended water + Ni-amended sediments + ^{62}Ni-amended food, and a (Control) clean water + clean sediment + clean food (SI, Fig. S1). Control treatments represent baseline *H. azteca* and *L. stagnalis* responses to TSS and DOC without Ni amendments (Table 1 and SI, Tables S1 and S2).

Each 7 d Ni exposure had four treatments (reference, TSS, DOC, and Ni-only/None), four replicates of 10 organisms, with one of these replicates/treatment used strictly for sediment and water chemistry analysis. *H. azteca* were 7–14 d old (USEPA, 2000) and *L. stagnalis* were <7 d post–hatch at the start of each test (Brix et al., 2011). Organisms were exposed in 300 ml high lip beakers with 100 ml of sediment, and ~175 ml of overlying water. Water changes (culture water, Ni-water, TSS-water, and DOC-water) were delivered using a Zumwalt design that delivered ~1 L of water dispersed over all beakers twice a day.

2.2. Ni concentrations for the Ni-exposures

Nickel concentrations for all the water, sediment, and food (compartments) exposures were held constant to allow for comparisons of *L. stagnalis* and *H. azteca* Ni effects. However, due to varying sensitivities of *L. stagnalis* and *H. azteca*, the Ni-water concentrations required adjusting. Nickel LC_{75} concentrations for water and sediments were chosen to discern Ni effects with different sediment types, Food, DOC, and TSS additions. *L. stagnalis* Ni-water concentrations were spiked at ~400 μg/L of Ni, and *H. azteca* Ni-water concentrations at ~2000 μg/L of Ni. In the Ni-sediment exposures, both WD sediments (WDsed) and GC sediments (GCsed) were spiked with similar Ni concentrations (~200 mg/kg of Ni) (SI, Tables S1 and S2). The leaf and lettuce disks used in the Ni-food exposures were soaked in 1200 μg/L of ^{62}Ni. Changes were made to the Ni concentrations used during the Ni-all exposures since all compartments (water, sediment, and food) received Ni amendments. The Ni concentrations were reduced by half in the Ni-all exposures (water ~1200 μg/L of Ni, sediment ~100 mg/kg of Ni, and food soaked in 600 μg/L of ^{62}Ni) (SI, Tables S1 and S2).

2.3. DOC, and TSS amendments to water and sediments

The TSS-water pulse exposures were for 24 h, and after this time, the appropriate water source (culture-water, Ni-amended water) was given minus the TSS. The DOC-water and Ni-water was added for the duration of the exposure. The use of air pumps and glass pipette tips were used to suspend the TSS, and all treatments (Reference, TSS, DOC, and Ni-only) received airlines during the 24 h exposures.

Water samples for Ni and DOC were taken on Days 1 and 7. The Ni and DOC samples were filtered through acid cleaned 0.45 μm filters to determine dissolved Ni and OC fractions. All DOC concentrations were analyzed on Tekmar/Teledyne TOC combustion analyzer. The TSS samples were filtered through Whatman® filters for TSS determination, and separate water samples were analyzed for turbidity (NTU).

2.4. Food source and ^{62}Ni food labeling

L. stagnalis replicates were fed one romaine lettuce disk (17 mm in diameter), and *H. azteca* replicates were fed three microbial conditioned *Acer rubrum* leaf disks (10 mm diameter) (Croteau and Luoma, 2008; Naylor et al., 1989). The ^{62}Ni was added to both lettuce and leaf disks only in two Ni exposures: Ni-food and Ni-all. Food was soaked for 48 h in separate ^{62}Ni concentrations (1200 μg/L (Ni-food) and 600 μg/L (Ni-all)), gently stirred daily, and

Table 1
Lymnaea stagnalis growth (dry wt.) for all treatments during the 7 d Ni exposures.

Treatments										
Date	Exposure compartment		WD Ref	WD TSS	WD DOC	WD Ni-only	GC Ref	GC TSS	GC DOC	GC Ni-only
08-Apr-10	Ni-water	Mean (mg dry wt)	2.13a	1.37ab	1.67c	0.93ad, cd	1.70 a	0.97ab	1.37c	1.00ad
		St. Dev (mg dry wt)	0.12	0.15	0.15	0.45	0.26	0.29	0.15	0.10
07-May-10	Ni-sediment	Mean (mg dry wt)	2.00a	2.57b	1.73c	1.87d	1.93a	0.00ab	0.00ac	0.00ad
		St. Dev (mg dry wt)	0.17	0.93	0.06	0.32	0.32	0.00	0.00	0.00
27-May-10	Ni-food	Mean (mg dry wt)	2.67a	1.93b	1.73ac	1.70ad	2.57a	1.97b	2.20c	1.87d
		St. Dev (mg dry wt)	0.31	0.40	0.15	0.30	0.32	0.35	0.57	0.81
16-Jun-10	Ni-all compartments	Mean (mg dry wt)	1.10a	0.73b	0.90c	0.77d	1.17a	0.83b	1.10c	0.43ad, cd
		St. Dev (mg dry wt)	0.10	0.32	0.17	0.23	0.32	0.15	0.17	0.06
30-Oct-10	Controls	Mean (mg dry wt)	2.27a	2.60b	2.37c		2.27a	2.07b	2.33c	
		St. Dev (mg dry wt)	0.50	0.44	0.67		0.21	0.32	0.42	

WD = Warden Ditch sediment.
GC = Greenville Creek sediment.
TSS = Total suspended solids.
DOC = Dissolved organic carbon.
Ni = Nickel.
Single letters represent no statistical differences between treatments = a,b,c,d.
Different letters represent statistical differences between treatments = a,b,c,d.