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## Redox effects on release kinetics of arsenic, cadmium, cobalt, and vanadium in Wax Lake Deltaic freshwater marsh soils

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### HIGHLIGHTS

- Low  $E_H$  leads to a high release of As, V, Fe, DOC, specific UV absorbance (SUVA).
- Dissolved Cd, Mn,  $SO_4^{2-}$ , and  $Cl^-$  increased under oxic conditions.
- Reduction of Fe (hydr)oxides under low  $E_H$  leads to release of associated As and V.
- Anaerobe, aerobic microorganisms, gram negative & positive bacteria, and fungi are found.

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### ABSTRACT

The impact of redox potential ( $E_H$ ), pH, iron (Fe), manganese (Mn), chloride ( $Cl^-$ ), aliphatic and aromatic dissolved organic carbon (DOC), and sulfate ( $SO_4^{2-}$ ) on the release of dissolved arsenic (As), cadmium (Cd), cobalt (Co), and vanadium (V) were studied in Louisiana freshwater marsh Wax Lake Delta soil (Mississippi River) using an automated biogeochemical microcosm apparatus. The experiment was conducted from reducing ( $-60$  mV) to stepwise oxidizing ( $+491$  mV) conditions. The initial pH was 7.4 and decreased under reducing conditions to 4.9, and remained constant during the increase of  $E_H$ . Concentrations of As ( $1.3$ – $120.5$   $\mu\text{g L}^{-1}$ ), V ( $0.9$ – $48.6$   $\mu\text{g L}^{-1}$ ), Fe, DOC, and the specific UV absorbance increased under reducing conditions and decreased with rising  $E_H$ . Release of As and V appeared to be related to changes of  $E_H$ /pH, co-precipitation with Fe oxides, and the release of dissolved aromatic carbon compounds. Concentrations of soluble Cd ( $4.8$ – $11.2$   $\mu\text{g L}^{-1}$ ), Mn,  $SO_4^{2-}$ , and  $Cl^-$  increased under oxidizing conditions. Release of Co ( $166.6$ – $258.2$   $\mu\text{g L}^{-1}$ ) was related to the chemistry of Fe, Mn and DOC. Phospholipid fatty acids analysis indicated the potential for the microbial community to be involved in biogeochemical processes such as the formation of sulfides, oxidation and reduction of compounds, and the bio-methylation of elements such as As. Overall, we measured a release of As and V under anoxic conditions, while oxic conditions favored the release of Cd. These results outline concern on the potential risk of mobilization of toxic elements in temporary waterlogged soils for agricultural purposes in deltaic ecosystems.

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### 1. Introduction

Riverine sediments and associated wetlands can be net sinks for pollutants such as potentially toxic elements (PTEs). However, during different flood-dry-cycles, these sediments may also act as a source of pollutants (Shaheen et al., 2014a). Wetland ecosystems

have a unique oxic and anoxic soil environment both spatially and temporally (Reddy and DeLaune, 2008; Ok et al., 2011). The intensity of soil reduction can be easily measured by soil oxidation–reduction (redox) potential ( $E_H$ ), which allows the prediction of the stability and dynamics of various nutrients and PTEs in soils and sediments (Yu et al., 2007; Rinklebe et al., 2016a,b). The solubility of PTEs under flooded conditions is largely governed by soil/sediment  $E_H$  and pH, DOC, Fe–Mn oxides,  $Cl^-$ , and  $SO_4^{2-}$  (Houben et al., 2013; Frohne et al., 2011, 2014, 2015; Schulz-Zunkel et al.,

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2015; Shaheen et al., 2014a,b,c; Vithanage et al., 2014). Thus, we hypothesized that an oscillation from reducing to oxidizing conditions in a wetland soil composed of Mississippi River deltaic sediment would allow us to determine the kinetics of redox-sensitive pollutants, such as arsenic (As), cadmium (Cd), cobalt (Co), and vanadium (V) due to changes in  $E_H$ /pH- values, DOC, and the related chemistry of sulfur (S), Fe, and Mn.

The mobilization and bioavailability of PTEs such as As, Cd, Co, and V in soils is partly controlled by their speciation and distribution coefficient (Du Laing et al., 2009; Bolan et al., 2013; Shaheen et al., 2013; Tian et al., 2014, 2015). Microorganisms govern many biogeochemical cycles, particularly in flooded soils and sediments (Huang, 2014; Moche et al., 2015). The redox transformations of Fe, S, and As are predominately driven by microorganisms, but abiotic As transformations coupled with Fe and S redox transformations may also occur (Huang, 2014). The microbial community composition can be characterized a number of ways including the analysis of phospholipid fatty acids (PLFA) (e.g., Langer and Rinklebe, 2009; Rinklebe and Langer, 2006, 2008). This method allows for the identification of microbial groups such as sulfate-reducing bacteria (SRB) or fungi (Taylor and Parkes, 1985; Macalady et al., 2000). While SRB mediate the formation of sulfides as a result of respiration processes that require  $SO_4^{2-}$  as a terminal electron acceptor (King et al., 2002), several bacteria and fungi are able to mediate the bio-methylation of PTEs such as As (Bentley and Chasteen, 2002).

There is a paucity of studies that specifically address redox-induced mobilization and immobilization processes of PTEs in Louisiana freshwater marsh Wax Lake Delta soil. Therefore, knowledge regarding specific soil mechanisms and approaches for regulating the release dynamics of dissolved As, Cd, Co, and V in soils of Wax Lake Delta is limited. Frohne et al. (2011, 2014; 2015), Shaheen et al. (2014b,c), and Rinklebe et al. (2016a) have studied the impact of  $E_H$  on metal(loid) release kinetics in a slightly acidic floodplain soil from Germany. Rinklebe et al. (2016b) studied the release dynamics of dissolved As, Ba, Cd, Cu, Pb, and Sr in seven different paddy soils originating from the U.S.A. and Asia under controlled-redox conditions. Shaheen et al. (2014a) investigated the release of dissolved Co, Ni, Se, and V in paddy rice soil in the northern portion of the Egyptian Nile Delta. However, the studied soils in Louisiana freshwater marsh Wax Lake Delta soil differ in their characteristics and metal content as compared to the previous studied wetlands. Since, the impact of reducing-oxidizing conditions on the release kinetics of As, Cd, Co, and V and related governing factors in Mississippi River deltaic soils have not been systematically studied to date, our goal was to mechanistically study the impact of a range of redox conditions and associated pH changes on the release kinetics and solubility As, Cd, Co, and V using an automated biogeochemical microcosm system using sediments collected from the Wax Lake Delta, USA. Specifically, the objective was to examine how potential controlling factors i.e., soil pH, DOC, PLFA, and the chemistry of Fe, Mn,  $Cl^-$ , and  $SO_4^{2-}$  govern the solubility of the redox-sensitive pollutants As, Cd, Co, and V under a continuous range of reducing to oxidizing conditions.

## 2. Materials and methods

### 2.1. Study site and field sampling

The study area, a predominantly fresh water marsh system within the Mississippi River deltaic plain, is located at the southern terminus of the Atchafalaya Basin, which receives the collective flow of approximately 30% of the Mississippi River and Red River (DeLaune and White, 2012). The Atchafalaya Basin has two primary outlets where active delta formation is currently underway with associated establishment of coastal freshwater marshes, the

Atchafalaya and Wax Lake Deltas (Supplemental 1). The sampling site was located along Hogs Bayou. All of the aboveground emergent marsh vegetation was removed by cutting the stems at the soil surface before three cores were collected within a 1 m<sup>2</sup> area. All cores were collected using 7 cm diameter pushcores to collect the top 15 cm of sediment. Samples were then placed on ice and returned to the laboratory where they were stored at 4 °C.

### 2.2. Soil characterization

Total C and N were determined on dried, ground subsamples using an elemental combustion system (Costech Analytical Technologies, Valencia, CA). Total P was determined by ashing dried ground subsamples at 550 °C followed by digestion with 6 M HCl (Andersen, 1976) and measured colorimetrically using a SEAL AQ2 Automated Discrete Analyzer (West Sussex, England). Microbial biomass C was determined using the fumigation-extraction method after Brookes et al. (1985) with modifications by White and Reddy (2000). Total organic carbon (TOC) was determined on the extracts using a Shimadzu Scientific Instrument TOC-VCSN (Columbia, MD). Measured total element concentrations were extracted by digestion of the samples in a microwave system (Milestone MLS 1200 Mega, Germany) (US EPA 3051A, 2007). Quality control of the extraction efficiency was performed using certified soil reference materials (BRM No. 13 and BRM No. 10a) obtained from the Federal Institute for Materials Research and Testing (BAM). Recovery ranged from 95 to 104% depending on the element. Total concentrations of As, Cd, Co, Fe, Mn, S, and V in the digested soil sample were measured by inductively-coupled plasma optical emission spectrometry (ICP-OES) (Ultima 2, Horiba Jobin Yvon, Unterhaching, Germany). Basic characteristics and the total element concentrations in the soil are presented in Supplemental 2. The total concentration of As, Cd, Co, and V was 33.7, 0.26, 9.7, and 48.5 mg kg<sup>-1</sup>, respectively. According to the range of maximum allowable concentrations for trace elements in agricultural soils as indicated by Kabata-Pendias (2011), the concentration of As was significantly higher than the world soil average content of (15–20 mg kg<sup>-1</sup>) and could be of concern if mobilized in an agricultural setting with potential transfer to the plant.

Phospholipid extraction and PLFA analysis were conducted according to the standard procedure described by Frostegard et al. (1991). Phospholipid fatty acids were designated according to the nomenclature described by Feng et al. (2003). Further details regarding the PLFA method is provided in Rinklebe and Langer (2006) and experimental specifics are outlined in Supplemental 3.

### 2.3. Automated biogeochemical microcosm experiment

A unique automated biogeochemical microcosm system (MC) was employed to simulate flooding of the soil in the laboratory. Controlling soil redox conditions in constantly stirred microcosms has several advantages over static incubations. Redox conditions are reproducible and controlled, and can be easily manipulated. Furthermore, the effect of  $E_H$  can be studied independent from other soil parameters and diffusion limitations. This system allows one to measure the  $E_H$ , pH, and temperature in each MC every ten minutes, thereby allowing one to mechanistically conduct experiments aimed at exploring underlying geochemical processes under fully controlled conditions. This system has been utilized in previous studies to determine the dynamics of trace metal(loid)s (Antić-Mladenović et al., 2011; Rupp et al., 2010; Frohne et al., 2011, 2014, 2015; Shaheen et al., 2014a; Rinklebe et al., 2016a,b,c). More technical details of the system are provided in Yu and Rinklebe (2011) and experimental specifics are presented in Supplemental 3.

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