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Cadmium stable isotope variation in a mountain area impacted by acid mine drainage



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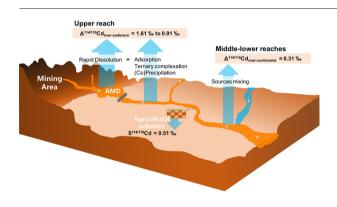
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HIGHLIGHTS

GRAPHICAL ABSTRACT

- Mineral, tailings, sediments and AMDcontaminated river water were analyzed for Cd isotope ratios.
- Distinctive Cd isotope signatures associated with transport and fate of riverine Cd.
- Agricultural activities contributed a heavy isotope signature input of Cd.
- Cadmium stable isotope is a promising tracer of attenuation processes in acid mine drainage.



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ABSTRACT

The pollution of natural waters and sediments with metals derived from acid mine drainage (AMD) is a global environmental problem. However, the processes governing the transportation and transformation of AMD metals such as Cd in mountainous areas are poorly understood. In this study, the Cd isotopic composition and Cd concentration of river water and sediments (16 sampling sites) from an AMD-affected river in southern China were determined. Cd concentration in river water declined from its source at a tailings dam (304 µg L⁻¹) to a point 14 km downstream (0.32 µg L⁻¹). Sediment Cd concentration ranged from 0.18 to 39.9 µg g⁻¹, suggesting that anthropogenic Cd is derived primarily from the tailing dam and easily enters the solid phase of the river. Isotopic data showed that the dissolved Cd in rivers was characterized by $\delta^{114/110}$ Cd values ranging from 0.21‰ to 1.03‰, with a mean of 0.48‰. The greatest Cd isotope difference was observed between the water and sediments in the LW dam ($\Delta^{114/110}$ Cd_{river-sediment} = 1.61‰, site 1), likely due to a rapid weathering dissolution of the ore tailings. In the river's upper reach (sites 2–3), isotope difference between river and sediment ($\Delta^{114/110}$ Cd_{river-sediment}) ranged from 1.0‰ to 0.91‰. This suggests that a host of secondary processes might have impacted Cd isotope fractionation, including adsorption, ternary complexation and/or (co)precipitation of Cd on secondary oxides and hydroxides. In the middle and lower reaches, an abruptly elevated $\delta^{114/110}$ Cd value near farmland (site 10) suggests the existence of a second Cd source. Based on the chemical properties of

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** Correspondence to: Y.-T. Tang, School of Environmental Science and Engineering, Sun Yat-sen University, Guangzhou 510275, PR China. *E-mail addresses:* haoqiu@sjtu.edu.cn (H. Qiu), eestyt@mail.sysu.edu.cn (Y.-T. Tang). water samples we can attribute this heavy isotope signature to agricultural fertilizer and drainage from agricultural fields. Our results suggest that Cd isotope is a tracer for identifying and tracking Cd sources and attenuation mechanisms (adsorption/(co)precipitation) in a complex mountain watershed.

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1. Introduction

Cadmium (Cd) is a trace element that naturally occurs in the environment at levels of parts per billion. Cadmium and its compounds are highly toxic, carcinogenic, and can eventually enter the food chain and pose a serious threat to human health (Nordberg, 2009). Cadmium mineral deposits are rare, and the element often occurs as an isomorphic substituent in minerals found in primary ores of Fe, Pb, and especially Zn (e.g., sphalerite) (Cullen and Maldonado, 2013). Metal sulfide deposits are common in many mountain regions and are often associated with extensive zones of pyritic alteration (Borrok et al., 2009). Weathering of metal sulfide deposits and historical mining activities generate acid mine drainage (AMD), a sulfuric acid-rich solution with low pH and a high content of toxic metals (Aranda et al., 2012). This acid flow enables heavy metals like Cd to be mobilized from mine wastelands (e.g. tailings) into their surrounding rivers (Dold, 2014). Anthropogenic activity is the primary source of the release of Cd, which can contribute over 0.64 t Cd to the surface water environment each year (Mayes et al., 2010). Because of difficulty of access and scarcity of monitoring equipment in areas with complex topography, the (bio)geochemical processes that control the mobility of metals such as Cd in mountain regions are hardly investigated.

Several investigations have demonstrated that isotopic signatures of Cu, Fe, and Zn vary substantially between natural and contaminated water pools (Balistrieri et al., 2008; Chen et al., 2009; Chen et al., 2014). Those isotopes have emerged as a useful tool for identifying both the sources of heavy metals in terrestrial environments and the main processes controlling the fate of those metals (Szynkiewicz and Borrok, 2016; Wiederhold, 2015). The adsorption or complexation of metals (e.g., Zn) on biotic and abiotic surfaces, (co)precipitation with oxides/oxyhydroxides, and biological uptake can influence the isotope composition of dissolved metals (Aranda et al., 2012; Borrok et al., 2008; Borrok et al., 2009; Kimball et al., 2009). Previous studies have reported Cd isotope fractionation during Cd adsorption and precipitation on minerals, and heavier Cd isotopes are preferential remained in aqueous solutions (Horner et al., 2011; Wasylenki et al., 2014). In a microbial Cd uptake experiment, Cd bound in the cell membrane exhibited light Cd isotopic compositions as a result of sequestration of Cd in cell membrane (Horner et al., 2013). Considerable variation in the isotopic signature of Cd has been observed in seawater from several ocean regions, and Cd isotope fractionation is strongly controlled by biological processes (Abouchami et al., 2011; Abouchami et al., 2014; Gault-Ringold et al., 2012; Horner et al., 2013; Lacan et al., 2006; Ripperger et al., 2007; Xue et al., 2013). A study of the isotopic composition of Cd in the mixing zone of Siberian river estuaries found that the riverine input of several branch rivers had significantly different Cd isotope signatures (Lambelet et al., 2013). So far most researches on Cd isotopes in terrestrial systems have focused on industrial (Chrastný et al., 2015; Cloquet et al., 2006; Martinková et al., 2016; Shiel et al., 2010) and agricultural pollution (Imseng et al., 2018; Salmanzadeh et al., 2017; Wiggenhauser et al., 2016). However, we know very little about the direction and magnitude of isotopic changes of Cd that might occur as a result of specific geochemical processes in AMD polluted areas. In this study, we used stable isotope ratios of Cd combined with other geochemical tools to investigate Cd sources, transport, and attenuation in contaminated waters and sediments of a typical AMD-affected mountain area in south China.

2. Materials and methods

2.1. Study site and sampling procedure

Dabao mountain (24°33′39″N, 113°42′57″E) (Fig. 1) is located at northern Guangdong Province in southern China. The region has a humid subtropical climate with average annual temperature of 20 °C and annual rainfall of 1350-1750 mm. Since the 1970s, large scale mining for iron and copper ores has taken place in the area. In the current study, typical mineral samples (Fe sulfide-rich minerals) and mine tailings were collected from the mine area. A total of 16 river water samples and 16 sediment samples were collected from the Liwu (LW) tailings dam (the main source of AMD), the Hengshi (HS) River (contaminated by AMD), the Fandong (FD) River (a tributary of HS) and the Taiping (TP) River (a non-contaminated river). Sampling sites were established using a Garmin global positioning system (GPS). Samples were collected from upper (sites 1-4), middle (sites 8-10), and lower (sites 11-13) reaches of the HS river in June 2016 (Fig. 1). Prior to sampling, water sample bottles were acid washed and rinsed three times with pure water. In all sites, five sub-samples were collected across the river to form a composite water sample to represent each sampling site. The sites for sediment sampling were located as close as possible to the water sampling sites. At each site, the top layer (0-3 cm) sediments were collected by using a grab sampler, and a composite sample was formed by a mixture of 5 sub-samples within 10 m² to represent each sampling site.

After sampling, the water samples were immediately split into two types and stored into 1 L polypropylene bottles in the icebox. 1) dissolved water samples filtered through 0.45 µm cellulose acetate filter (Sartorius Stedim Biotech, Germany), and 2) unfiltered water samples that contained water and suspended particulate matter (SPM) (Kimball et al., 2009; Szynkiewicz and Borrok, 2016). Water samples for cation and isotopic ratio analysis were acidified to pH < 2 with sub-boiling distillation 14 M HNO₃, and for NO_3^- and SO_4^{2-} anion analysis were unacidified (filtered). Water quality parameters, including pH, EC, Eh and temperature were measured when sampling by using a calibrated multiparameter instrument (HYDROLabDS5, USA). All the samples were transported to the laboratory within 12 h. Then water and sediment samples were refrigerated at 4 $^{\circ}$ C and $-18 ^{\circ}$ C before analysis. In order to present selected chemistry results graphically as a function of their sampling locations, the distance of each sampling site from the outlet of the LW dam was measured (Fig. 1).

2.2. Geochemical analysis

Sample preparation was conducted in a clean room facility. All acids used in this study were purified by sub-boiling distillation and the deionized water was >18.2 M Ω -grade purified from a Milli-Q water purification system (Millipore, Bedford, USA). For sediment samples, 50–100 mg was weighed into a 10-mL polytetrafluoroethylene (PTFE) bomb, and approximately 1 mL concentrated HNO₃ and 1 mL concentrated HF were added. The digestion was carried out at 190 °C for 2 days to remove organic material in the solid phase. Subsequently, each sample was taken to dryness at 120 °C, and the residue was dissolved in 1 mL concentrated HNO₃ and 1 mL H₂O and placed in a sealed bomb and digested at 190 °C for 24 h. After evaporation at 120 °C, the sample was dissolved in 2 mL of 2.0 M HCl, centrifuged at 5500 rpm

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