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Distribution of trace metals and Pb isotopes in bottom sediments of the Murucupi River, North Brazil

Diomar Cavalcante Oliveira^{a,b,*}, Jean Michel Lafon^a, Marcelo de Oliveira Lima^b^a Institute of Geosciences, Federal University of Pará, Brazil^b Environmental Section, Evandro Chagas Institute, Pará, Brazil

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ABSTRACT

The Murucupi River belongs to the hydrographic network of the Pará River estuary, at the southern portion of the Amazon River mouth, which consists of a fluvial-marine transitional zone under strong impact of both tidal and fluvial currents. The geochemical results obtained for bottom sediments from the Murucupi River, the Arrozal Channel, and the Pará River indicate a natural variation of Pb, Cr, Cu, Zn, and Ni content among these water ways with no significant anthropogenic influence. According to the threshold effects level (TEL), the contents of trace metals do not offer risk to the local biota. By contrast, the differences in the Pb isotopic composition of sediments in the Murucupi River, the Arrozal Channel, and the Pará River are significant. These isotopic signatures indicate an anthropogenic contribution of Pb in the Murucupi River originating from the domestic effluents of urban centers; industrial waste represented by red mud is not included. These results demonstrate that the Pb isotopic signature is a prospective indicator for future contamination of bottom sediments by trace metals and is useful for identifying contaminants among the possible anthropogenic sources.

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1. Introduction

Bottom sediments constitute a compartment of the aquatic ecosystem with high relevance for environmental studies. They are considered as reservoirs of bioavailable trace metals in chemical mechanisms such as adsorption, precipitation, and aggregation to clay minerals that facilitate capturing and accumulation of these contaminants that may be released in the water column when physicochemical alterations occur in the environment (Duan et al., 2010; Dou et al., 2013; Pardo et al., 1993).

Trace metals in bottom sediments are attributed to natural origins such as rocks and soils, and suspended sediments in addition to anthropogenic origins such as industrial emissions and urban effluents (Alleman et al., 2000; Bollhöfer & Rosman, 2000; Cundy et al., 1997; Molisani et al., 1999). The determination of metal concentrations in bottom sediments is used to distinguish metals from pollution sources from those naturally introduced into the environment (Cunha et al., 2009; Geraldés et al., 2006; Hansmann & Koppel, 2000).

Additional difficulties to assess anthropogenic contributions in bottom sediments may arise when several pollutant sources are involved, due to this each contribution must be determined (Bird, 2011; Véron et al., 1999). In addition, appropriate values for the regional background are needed to identify the pollutant contributions (Galuszka, 2007).

In the study of contamination by trace metals, Pb is prominent because it displays different isotopic signatures according to the source, whether pollution or natural sources. Pb of natural origin displays an isotopic signature that corresponds to the average of the source rocks, whereas that of anthropogenic origin records the specific isotopic signature of the mineral deposit from which it was extracted (Faure, 1986). These signatures are not affected by geochemical, biochemical, or industrial physicochemical processes and can be modified in the environment only by mixing processes. Therefore, isotopic ratios, particularly the ²⁰⁶Pb/²⁰⁷Pb ratio, may be used to distinguish between natural and anthropogenic contributions of this metal in the environment (Álvarez et al., 2012; Banner, 2004; Choi et al., 2007; Komarek et al., 2008; Kylander et al., 2010; Zhang et al., 2008).

In Brazil, several studies have been performed to identify the anthropogenic sources of Pb in the environment by using its isotope signature in lake, lacustrine, and estuarine sediments because this metal is strongly affected by industrial and urban wastes (Cunha et al., 2009; Geraldés et al., 2006; Moura et al., 2004; Marques et al.,

* Corresponding author at: Environmental Section, Evandro Chagas Institute, Pará, Brazil.

E-mail address: diomarcavalcante@yahoo.com.br (D.C. Oliveira).

2006; Oskierski et al., 2009; Oliveira et al., 2012), stream sediments from mining areas (Moraes et al., 2004), and atmospheric aerosols from the main Brazilian industrial and urban centers (Bollhöfer & Rosman, 2000; Babinski et al., 2003; Mirlean et al., 2005).

The Barcarena region is an industrial harbor area that encompasses industrial plants for alumina, aluminum, and fertilizer production; kaolin processing; and manganese sintering (CDP, 2013). These industrial processes generate waste that is deposited in the settling basins, which constitutes a potential risk for aquatic life. The Murucupi River, a tributary of the Pará River, also receives untreated sanitary and domestic effluents from urban areas. The Murucupi River is used for recreational purposes and subsistence fishing and provides a water supply for human consumption. It is characterized as a Class II freshwater river under Brazilian legislation; however, no studies have been conducted to evaluate the urban and industrial impacts on this river and to refine its classification (CONAMA, 2004).

The aim of this study is to investigate the possible involvement of effluents originating from urban centers and industrial wastes as pollution sources of the Murucupi River by determining the levels of trace metals including, Cr, Cu, Ni and Zn in addition to Pb isotopic signatures. Moreover, this study evaluates the potential of Pb isotopic signatures for detecting future anthropogenic impacts of contamination by trace metals. Finally, an estimate of the reference values is proposed for the concentration and isotopic composition of natural Pb for this sector of the Pará River estuary system.

2. Materials and methods

2.1. Study area

The hydrographic network of the Barcarena region – state of Pará, Brazil, where the Murucupi River is located, belongs to the Marajó Bay - Pará River estuary system, between the city of Belém and the south-southwestern coast of Marajó Island at the southern portion of the Amazon River mouth (Martins & Mendes, 2011). The entire hydrographic system, which includes the area of focus, encompasses Guajará Bay and the Carnapijó, Acará and Guamá rivers eastward (Fig. 1). Previous geochemical and Pb isotopic studies focused on Guajará Bay and Guamá River, which are under the direct influence of the city of Belém, the largest urban area in the region with around 2.7 million inhabitants (Moura et al., 2004; Santos et al., 2012).

The Murucupi River originates within a conservation area and is located in the vicinity of the red mud storage lagoon of the world's largest alumina refinery, Hydro Alunorte. Red mud is a waste product created by the processing of Al-rich bauxite for alumina production. Along its course, the river crosses two urban areas, Vila dos Cabanos and Laranjal, that release domestic effluents *in natura* into the river. The Murucupi River is approximately 8 km long and flows into the Arrozal Channel, which in turn flows into the Pará River and the Barcarena River to the west and east, respectively. The geological substratum of the Barcarena region consists of a Cenozoic sedimentary package of which the stratigraphic succession displays little variation along the studied area. In land areas, the Barreiras Formation sediments outcrop in the higher parts, whereas the river banks are constituted by Quaternary Post-Barreiras unconsolidated sediments (Rossetti, 2001).

2.2. Collection and preparation of samples

Bottom sediment samples were collected 1 km apart at seven points along the Murucupi River (M1, M1A, M2, M3, M4, M5, and M6 samples), six points along the Arrozal Channel (AC1, AC2, AC3,

AC4, AC5 and AC6 samples), and four points along the Pará River (PA1, PA2, PA3 and PA9 samples) in a sampling campaign conducted in November 2011. During the same period, samples of red mud (RM1) from the storage lagoon of the alumina refinery and a soil sample (P1) located in a non-navigable part of the Murucupi River were also collected in order to obtain geochemical and isotopic data for possible industrial and natural sources, respectively. The M1, M1A, and M2 samples were collected in the vicinity of the access bridge to Vila dos Cabanos, which is in close proximity to a wastewater dumpsite in the river. The other samples are representative of the course of the Murucupi River from this urban area until its mouth. Samples from the Arrozal Channel and Pará River, which are larger bodies of water and are less susceptible to anthropic contribution, were collected for comparison with those of the Murucupi River.

Sampling was performed by using a Van Veen-type dredge apparatus to collect sediment at a 5 cm depth from the surface of the bottom sediment. The samples were identified, stored in plastic bags, and packed in Styrofoam boxes until arrival at the laboratory.

For the chemical and isotopic analysis, the fine fraction (silt+clay) was separated by using the classical wet sieving method including a stainless steel sieve with an aperture of 0.063 mm (Förstner, 1989). The samples were dried at 50 °C before being pulverized and homogenized with an agate mortar and pestle.

3. Mineralogical and granulometric analyses

The relative proportion of sand, silt, and clay was obtained via particle-size analysis involving 1 g of the entire sample of sediment by using an Analysette[®] Microtec Plus laser diffraction device at the Laboratory of Applied Mineralogy and Geochemistry of the Institute of Geosciences of the Universidade Federal do Pará (UFPA).

Mineralogical results were obtained by X-ray diffraction in the Laboratory of Mineral Characterization of the Institute of Geosciences of UFPA by using an X-ray diffractometer (PAN analytical X'Pert Pro MDP model [PW3040/60]) equipped with a PW3050/60 goniometer (Theta/Theta) and a ceramic X-ray tube with Cu anode. The mineralogical composition of the bottom sediment sample was determined by using the powder diffraction method (Jenkins & Snyder, 1996). The clay minerals were identified through oriented lamina in the fraction smaller than 2 μm (Moore & Reynolds, 1992). Diffractogram processing and mineral identification were obtained by using the X'Pert Data Collector (version 2.1A) and X'Pert statistics software version 2.1B, respectively.

3.1. Chemical analyses

3.1.1. Organic matter

The proportion of organic matter (OM) was determined according to the procedures proposed by Gaudette et al. (1974), which are based on oxidation of organic C (OC) in an acid medium, H₂SO₄, by excess of a strong oxidizing agent, K₂Cr₂O₇. Approximately 300 mg of the fine fraction were transferred to a 500 mL Erlenmeyer flask, into which 10 ml of K₂Cr₂O₇ (1 mol L⁻¹) and 20 ml of concentrated H₂SO₄ were added. The sample was heated on a hot plate for 30 min at 100 °C and was then added to a volume of approximately 200 mL of distilled water. To remove Fe₂O₃, 10 mL of 85% H₃PO₄ was added to a solution containing excess K₂Cr₂O₇ that was titrated with Fe(NH₄)₂(SO₄)₂·6H₂O 0.5 mol L⁻¹ in the presence of a diphenylamine indicator. To obtain %C and %OM, the following equations were used:

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