Marine Pollution Bulletin 99 (2015) 61–69

Contents lists available at ScienceDirect

Marine Pollution Bulletin

journal homepage: www.elsevier.com/locate/marpolbul

Trace elements and heavy metals in the Grand Bay National Estuarine Reserve in the northern Gulf of Mexico

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ARTICLE INFO

Article history:
Received 13 January 2015
Revised 22 July 2015
Accepted 25 July 2015
Available online 31 July 2015

Keywords:
Environmental impact
Estuarine chemistry
Trace elements
Biogeochemical process
Lead isotopes
Grand Bay Reserve

ABSTRACT

The objectives of this study are to investigate distribution of trace elements and heavy metals in the salt marsh and wetland soil and biogeochemical processes in the Grand Bay National Estuarine Research Reserve of the northern Gulf of Mexico. The results show that Hg, Cd and to some extent, As and Pb have been significantly accumulated in soils. The strongest correlations were found between concentrations of Ni and total organic matter contents. The correlations decreased in the order: Ni > Cr > Sr > Co > Zn, Cd > Cu > Cs. Strong correlations were also observed between total P and concentrations of Ni, Co, Cr, Sr, Zn, Cu, and Cd. This may be related to the P spilling accident in 2005 in the Bangs Lake site. Lead isotopic ratios in soils matched well those of North American coals, indicating the contribution of Pb through atmospheric fallout from coal power plants.

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1. Introduction

Anthropogenic activities lead to the release of a significant amount of heavy metals and trace elements into estuaries from agricultural fields, residential areas, septic tanks, industrial discharges, and other point sources by rivers and creeks. Estuaries are known areas of inflow plagued with extreme physico-chemical changes that are the direct result of tidal variations (de Vallejuelo et al., 2014). Sediment, soil, water and organic material in these areas may contain significantly higher levels of heavy metals and trace elements. Partly due to large quantities of dissolved organic complexes and particulate matter, heavy metals are translocated for great distances and end up in the sediments of estuaries, thus reflecting in the relatively high levels of these elements in numerous estuarine organisms and in sediments (Ip et al., 2007) This produces a detrimental effect on the biogeochemical cycling and biochemical balance in estuaries. The major inputs of heavy metals and trace elements to estuaries are from riverine, atmospheric, and anthropogenic sources. Therefore, the environmental fate of heavy metals and trace elements such as Pb, Cd, Hg, and As in estuaries is of great concern (Adriano, 2001), specifically those originating in or around industrial centers, dumps, and former mine sites near estuaries.

The physical behavior of an estuary is a major factor in the transport and transformation of heavy metals and trace elements. Since estuaries are governed by the tidal and river flow from the sea, various salt mixing zones, and physicochemical parameters, the biogeochemical processes of trace elements and heavy metals could be drastically affected. These processes include: precipitation and co-precipitation, flocculation and net sedimentation, desorption and adsorption, immobilization and solubilization, re-suspension of subsequent metals and transformation. The results may yield complex distribution patterns, which may differ from one estuary to another.

The Grand Bay National Estuarine Research Reserve (Grand Bay NERR) (approximately 18,000 acres) is located in the Gulf of Mexico, along the southeastern corner of Mississippi in Jackson County. This reserve contains a variety of wetland habitats, both tidal and non-tidal, such as pine savannas, salt marshes, saltpans, bays and bayous, as well as terrestrial habitats that are unique to the coastal zone, such as maritime forests (Peterson et al., 2007). The Grand Bay NERR is an important ecosystem because of the biodiversity of habitats. These habitats support many important species of fish and wildlife. Commercially and recreationally

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http://dx.doi.org/10.1016/j.marpolbul.2015.07.062
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important species of finfish and shellfish such as brown shrimp, speckled trout and oysters abound in the region. Sea turtles, bottlenose dolphin and, on occasion, manatees can be found in the deeper waters of the reserve. Many species of carnivorous plants and orchids can be found in the higher savanna habitats. The Grand Bay NERR is a representative of the Louisianan biogeographic region and is in the Mississippi Deltaic subregion (Peterson et al., 2007).

In 2005, a local fertilizer company accidentally released 17.5 million gallons of waste water containing phosphorus (P) from retaining ponds into Bangs lakes (BL) in the Grand Bay Reserve, resulting in approximately 8 hectares of tidal marsh and 77 hectares of upland devastation (Peterson et al., 2007). Bangs Lake is an estuary of the Grand Bay Reserve of approximately 0.87 sq. mi. and its wetland is dominated by grass vegetation rooted in salt marsh soils comprised of an environmental matrix of biological and chemical interactions. The pH of the lake was drastically decreased and the PO_4 concentration was 144 μM (4.7 ppm), then has declined (Peterson et al., 2007). However, after 7 years of the accident, it is essential to re-assess the environmental effects of this P spilling accident on the biogeochemistry of trace elements and heavy metals of the reserve.

To date, a systematic study on the biogeochemistry of trace elements and heavy metals in the Grand Bay Reserve has not been conducted. Such a study would not only provide a solid scientific understanding of the biogeochemical processes controlling major biogeochemical cycling of these elements in the region, but would also secure sea food safety in large areas of the Gulf of Mexico. The objectives of this study are to investigate distribution of trace elements and heavy metals and major biogeochemical processes controlling their fate and transport in the Grand Bay National Reserve and to re-assess the environmental impacts of the 2005 P spill by ascertaining its long-term effects on the biogeochemistry of trace elements and heavy metals in the estuaries.

2. Materials and methods

2.1. Site description and field sampling

One hundred and twenty (120) samples were collected from four sites within the estuary in the summer of 2012: Bayou Heron (BH), Bayou Cumbest (BC), Crooked Bayou (CB), and Bangs Lake (BL) (Fig. 1). The Grand Bay National Estuarine Research Reserve (NERR) in the Gulf of Mexico is within a humid-temperate, nearly subtropical region with average annual rainfall of 184.4 cm between 1947 and 2003 (Peterson et al., 2007). The Grand NERR encompasses about 7446 hectares and consists of open water estuarine areas, muddy intertidal areas, a wide variety of marsh types, extensive and non-vegetated salt flats or pannes, and non-tidal areas (Peterson et al., 2007). The non-tidal areas include wet pine savanna, coastal bayhead and cypress swamps, freshwater marshes and maritime forests. There is also a 1.5–2.0 km wide marshy swampy wetland zone that separates the Grand Bay NERR shoreline from uplands that are underlain by Pliocene-age alluvial sandy-muddy soil – which is composed of gravelly Citronelle formation representing most of land surface in southern Mississippi (Otvos, 1990, 1997, 2000).

In general, Crooked Bayou is dominated by *Juncus* marsh and large vegetated shell midden. Bayou Cumbest is the site with soft sediments and unconsolidated oyster shell reefs. Bayou Heron is composed of a bottom type of soft muddy sediment that serves as a transition to a habitat of maritime slash pine flatwoods/savanna. Bangs Lake (BL) is more surrounded by industry and has extensive algal blooms in its shallow enclosed waters due to high P levels.

The geographical location of the sampling points were marked using a Garmin GPS. The samples were collected using a stainless steel soil testing probe at a depth of about 20 cm. The collected samples were packed in polyethylene bags and labeled before performing laboratory geochemical analysis. The packed samples were air dried in the greenhouse and homogenized using an agate mortar. The air-dried samples were then sieved through a 125 μm mesh.

2.2. Soil characterization

The determination of the concentration of the total organic carbon and nitrogen in the samples was performed with a Shimadzu TOC analyzer. Soil texture and particle size analyses were conducted using the hydrometer method (Klute, 1986). Free Fe oxides were analyzed by the citrate–bicarbonate dithionite method (Sparks, 1996). Fe and Mn in solution were determined with inductively coupled plasma optical emission spectroscopy (ICP–OES).

2.3. Trace element and heavy metal extraction and measurement

A Hot Block digestion method was employed to perform the tests of the total metal/metalloid contents in the estuarine soil samples (USEPA method 3050B). About 1 g of the soil was digested with a mixture of H_2O_2 (30%) and HNO_3 (concentrated). The samples were heated to 95 ± 5 °C (hot block) to reflux for 10–15 min without boiling. This solution was analyzed for trace elements and heavy metals using both inductively-coupled plasma–optical emission spectrometry (ICP–OES) (Perkin Elmer OPTIMA 3300 DV model) and inductively-coupled plasma mass spectrometry (ICP-MS) (Varian 820-MS ICP Mass Spectrometer). The ICP-OES was used for the analyses of Cu, Pb, Zn, Cr, Cd Ni, Fe, and Mn. The ICP-MS was used for the analyses of As, P, Hg, and Pb isotopes in all extracts. Multi-elements standards were made to the following concentrations: 0.02, 0.05, 0.1, 0.5, 1, and 2 mg/L for all elements except for Fe and Mn which was up to 5 mg/L for ICP–OES, while for ICP-MS, 0.02, 0.05, 0.1, 0.5 mg/L of P, As, Hg, and Pb isotopes were used.

The separate five independent standard solutions were prepared with concentrations from 0.05 mg/L to 2.0 mg/L and run as the samples for four times in order to check the accuracy and precision of ICP–OES. Independent standard solutions resulted in average 100.7–113.0% recoveries of Cd, Ni, Zn, Cr, Pb, Cu, Fe, and Mn at the concentrations from 0.1 mg/L to 2.0 mg/L and 90.5–120% average recoveries for the concentration at 0.05 mg/L. The relative errors among four repeated running were 0.1–10% for standard solutions with concentrations of 0.1–2.0 mg/L and 2–20% for standard solutions at 0.02 mg/L. Similar QA/QC procedures were also used for ICP-MS.

3. Results and discussion

3.1. Characterization of estuarial soils

The estuary soils are characterized by a majority of silt (54%) and sandy fractions, with relatively high organic matter (3.82% on an average) (Table 1). The silt contents ranged from 28% to 96% with 19% coefficient of variation (CV) and the sand particles ranged from 3.6% to 72% with 25% CV. Total organic matter contents varied from 0.52% to 10.2% with an average of 3.82% and a standard deviation of 2.1%. Organic N contents were in the range of 0.02–0.46% with an average of 0.11 ± 0.10%. The estuarine samples contained low amounts of iron and manganese oxide. Iron oxides averaged 0.16 ± 0.11% and Mn oxides were in the range of 0–0.012% with an average of 0.0006 ± 0.001%.

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