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Data Submitted by: Baohua Xiao Date: September 15, 2005

Data Collected by: Baohua Xiao

Data Analyzed by: Baohua Xiao

Project PI: Dr. Weilin Huang

Analysis is one of the following (please bold if submitting electronically, circle if submitting in paper form):

- **Soil (bulk soil, dry soil, or porewater)**
  - Surface Water (dissolved, particulate, or whole water)
  - Groundwater (dissolved, particulate, or whole water)
  - Air (gas, particle, precipitation)
  - Detritus
  - Biota (specify)
  - Other

Short Description of Data Collection:

Analyses of Total Organic Carbon (TOC) and Total Organic Nitrogen (TN):

The sediment samples were pretreated to remove inorganic carbon before analysis of TOC. In brief, 5 g of the grinded samples was placed in a centrifuge bottle and reacted overnight with 0.1M HCl solution at 60 °C. After decarbonation, the residual solids were rinsed with Milli Q water several times until the pH of the supernatants became neutral. The solids were then freeze-dried.

The decarbonated sediments and the HA and POM fraction of SOM were analyzed for TOC and TN using a CHNS analyzer (NA1500 Series, Carlo Ekba) hosted in the Institute of Marine and Coastal Sciences.

Particle Size Distribution:

The particle size distribution of the sediments was determined using a standard wet sieving method (Method of Soil Analysis, Dane and Topp, 2002). 15 g of dried sediment sample was weighed and placed in a 250 ml glass beaker. 200 ml of diluted sodium hexametaphosphate solution (~0.5 mM) was added into the beaker. The content of each beaker was stirred for one hour to disaggregate sediments. After disaggregation, the slurry was carefully poured into a stack of sieves with a pan at the bottom (the sieve system is water-tight.), and the beaker was rinsed with the diluted sodium hexametaphosphate solution several times to assure that all the solids have been transferred. After the sieve stack was filled up with the solution, it was placed on a horizontal shaker and shaken for 2 hours. After completion of the sieving, all sieves and the
pan with sediment fractions were placed in an oven set at 70 °C for 24 hrs. After drying, the weights of the sieves before and after removal of sediment fractions with using a sieve brush were recorded. The weight difference was used as the weights of the sediment fractions having different particle size ranges.

**Sediment Organic Matter (SOM) Fractionation:**

The natural organic matter associated with each sediment was fractionated into humic acid (HA) and particulate organic matter (POM) using a wet chemical procedure we developed in our laboratory and published (Song et al., 2002; *Environmental Science and Technology*). In brief, 20 g of each freeze-dried sediment sample was grinded using a mortar and pestle until no sandy particles could be felt. The sample was divided into two fractions, one (5 gram) to be used in TOC analysis described below, and the 15 g was placed in a 250 mL plastic centrifuge bottle for SOM fractionation. The sediment was first extracted with 150 mL of 0.1M NaOH solution (as many as 10 times) until the supernatant became colorless. The supernatants obtained from each extraction step for a given sediment were combined, mixed, and acidified with 6M HCl solution to pH 1. The HA was allowed to precipitate over 24 hours. The HA precipitates were rinsed with Milli-Q water three times, freeze-dried, and its weight was then recorded. The residual solid after base-extractions was further treated to remove inorganic matrix. It was allowed to react for 24 hours with HF/HCl mixture solutions (22M HF and 6M HCl at a volumetric ratio of 2:1) in a water bath preset at 60 °C. During reaction, the content of each plastic bottle was stirred manually. After demineralization, the residual solids were rinsed with dilute HCl solution (0.1M) twice followed by three times of Milli-Q water. It was then freeze-dried and weighted. This fraction of organic matter is defined as POM. The mass percentages of HA and POM in the bulk sediments were calculated based on their weights. HA and POM fractions were used directly for TOC and TN measurements.
Method Detection Limit Information:
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