

Sediment Toxicity in Central and South Florida Ecosystems

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There is growing concern in the U.S. over the extent of sediment contamination in freshwater, estuarine, and coastal areas (Lyman et al. 1987). Based on a limited database, it appears that both sediment and water are contaminated by a variety of pesticides in the St. Lucie River Watershed (SLR) in Central Florida and in the Everglades Agricultural Area (EAA) canal system in South Florida (Figure 1). This is not surprising since agriculture represents the major land use in South Florida and one-quarter of all pesticide active ingredients (a.i.) are used in this region to control pests on citrus, vegetables, sugar cane, rice, and ornamentals (Pait 1992). The SLR is in the northern part of St. Lucie County, and the EAA extends from Lake Okeechobee to Water Conservation Areas 1 and 2A. Sod farms, golf courses, maintenance of right-of-ways, mosquito control and household usage further contribute to pesticide contamination in South Florida.

Thus far, studies in South Florida have focused on monitoring exposure concentrations of contaminants in environmental media with little correlation to biological effects in ecological receptors. The South Florida Water Management District (SFWMD) began monitoring water and sediment in the mid-1980s in canals (Pfeuffer 1991). Sediment and water analyses indicates that atrazine, ametryn, bromacil, simazine, diuron, alpha-endosulfan, beta-endosulfan, endosulfan sulfate, ethion, hexazinone, and norflurazon were the most widely detected pesticides in surface water and DDE, DDD, ametryn, atrazine, dicofol, diquat, and endosulfan sulfate were the most frequently detected pesticides in sediments between 1991 and 1995 (Miles and Pfeuffer 1997). Several of the sampling sites were located in the EAA and others in the Homestead Agricultural Area (HAA) adjacent to Everglades National Park (in Dade County in Figure 1). Recent analyses of fish (i.e., largemouth bass, Florida gar) collected in 1995 by the U.S. Geological Survey through its National Water Quality Assessment (NAWQA) Program showed that concentrations of organochlorine residues (e.g., dieldrin) including DDT and its degradation products (p,p'-DDE) have remained prevalent in freshwater fish and are similar to measurements in the early 1970's in canals of the northern Everglades near agriculture (Haag and McPherson 1997). PCBs were not as frequent in 1995 as in the 1970's but concentration ranges were similar. The Florida Department of Environmental Protection (FDEP) conducted the only field study in South Florida that correlates contaminant exposure with biological effects in aquatic receptors (Graves and Strom 1995). The presence

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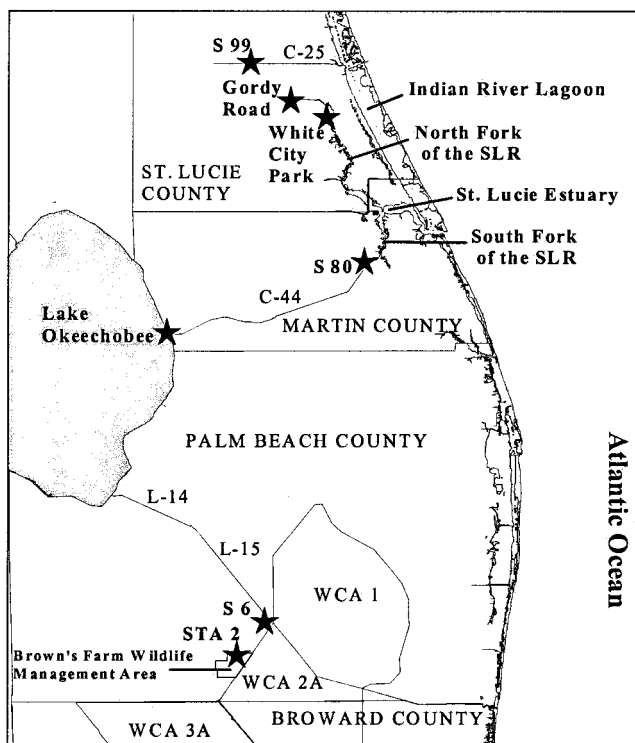


Figure 1. Site locations in central and south Florida.

of the organophosphate insecticides ethion (i.e., for citrus) and/or malathion (i.e., for mosquito control) in water were implicated as the causative agents in producing decreases in the abundance and densities of benthic macroinvertebrates in Ten Mile Creek, the major tributary to the North Fork of the St. Lucie River, an Outstanding Florida Water (OFW).

The Science Subgroup (1996) for the South Florida ecosystem restoration effort clearly states that chronic low-level agricultural and urban contaminant exposure is likely to represent one of the greatest risks to aquatic systems and that more consideration should be given to the role that pesticides and other contaminants play in the structure and function of South Florida ecosystems. This study presents the results of year one of a multi-year investigation to evaluate the toxicity of sediments in the EAA and SLR to aquatic species and conduct an ecological risk assessment.

MATERIALS AND METHODS

Bacterial species were used to assess the toxicity of sediments in the Microtox[®] basic solid phase test (BSPT), since it provides direct contact of a naturally occurring non-pathogenic luminescent bacterium (*Vibrio fischeri*) with whole

sediment samples (i.e., pore water and particles) increasing the probability for measuring responses to particle-bound and marginally soluble toxicants (Strategic Diagnostics Inc., Newark, DE). Microtox[®] measures the change in luminescence of a cell suspension (EC50; effective concentration that decreases light transmission by 50%) and is a function of the toxic effects of a substance on cell metabolism. Sediment was also physically and chemically characterized: total organic carbon (TOC), pH, ammonia, acid volatile sulfide (AVS), cation exchange capacity (CEC), and particle size distribution. The concentrations of volatile and semi-volatile organics, metals, PAHs, PCBs, and pesticides were also analyzed in each sediment sample.

Seven sediment sampling sites were chosen in South Florida based upon past water quality violations and preliminary sampling data (Graves and Strom 1995; Miles and Pfeuffer 1997; Pfeuffer 1991). Sites were located in or adjacent to ecosystems of concern: the SLR (S-80, Gordy Road, and White City Park), Indian River Lagoon (S-99), the Northern Everglades (S-6, and STA-2), and Lake Okeechobee (Figure 1). The Gordy Road site is located on Ten Mile Creek, a channelized stream that drains surrounding agricultural, primarily citrus, areas and is one of the sub-basins for the northern portion of the SLR. The C-25 canal contains sampling site S-99, and discharges into Indian River Lagoon (IRL) an estuary with more species than any other estuary in North America. The sampling site near S-80 is located at a pumping structure within the C-44 canal. C-44 is primarily a navigation route but is also used for drainage and irrigation by surrounding citrus communities. Since C-44 is a part of the western basin of the SLR and a majority of the surrounding land is used for agriculture, discharge from the canal is a suspected source of pesticides to the North Fork basin of the SLR. The chosen sampling location was downstream from a pumping station that discharges overflows from Lake Okeechobee, through agricultural regions, and into the South Fork of the SLR. White City Park is a site within the North Fork of the SLR. A six-mile stretch along the SLR is being considered for protection under the state of Florida's Save Our Rivers Plan. The Florida Department of Natural Resources has called the North Fork one of the last freshwater/ estuarine wilderness ecosystems in the region. Threatened and endangered species are known to reside in the North Fork basin (FDEP 1997).

S-6 is a primary pumping system in the EAA. S-6 is used to gauge inflows to a storm-water treatment area (STA-2) where nutrient-rich waters are managed before discharging into the Everglades Water Conservation Areas (Chimney et al. 2000). STA-2 is located between the EAA and Water Conservation Area-2A. Prior to its use as a storm-water treatment area, STA-2 was preserved as a habitat for wildlife. Lake Okeechobee is the second largest freshwater body completely contained within the contiguous United States. Land use around the Lake Okeechobee watershed is largely agricultural, with dairy production to the north and sugarcane and vegetable farms to the south. Lake Okeechobee serves as a source of irrigation for agricultural areas, drinking water for urban areas, and a major source of water for the Everglades.

A petite Ponar grab sampler was used to collect sediments except at STA-2, where samples were collected on foot due to its shallow water/ marsh location. Lake Okeechobee and STA-2 contained one transect. Three transects were taken at S-6, S-99, White City Park, and Gordy Road (A, B, C). Six transects were taken along S-80 (A1, A2, B1, B2, C1, C2). Each transect consisted of an aggregate of three sediment samples. Each sample was centrifuged at 9,000 rpm for 30 min to remove pore water. Microtox[®] BSPT was then performed on each sample within 48 h of collection and an EC50 was determined for each transect. All sediment samples were prepared by weighing 7.00 g with 35 mL of diluent and stirring with a Teflon magnetic stir bar for approximately 30 minutes. Nine serial dilutions of each sediment sample were made for testing with the highest concentration set at 99,000 mg/L. Light readings were taken after the bacteria were exposed for thirty minutes to the sediment.

Three artificial sediments were also formulated and used as reference controls following the methodology of Kemble et al. (1999) with α -cellulose as a source of carbon. Preparation of formulated sediments followed methods described by Walsh et al. (1991) and Harrahy and Clements (1997). Sand (Ottawa testing sand, U.S. Silica, Ottawa, IL) was rinsed with RO water until water was clear, then rinsed with de-ionized water for five min, and air-dried. α -Cellulose was from Sigma Chemical (St. Louis, MO). A silt and clay mixture (ASP 400; Englehart, Edison, NJ) was from Mozel (St. Louis, MO) and Dolomite (Spectrum Co., Gardena, CA) was used as a source of magnesium, calcium and bicarbonate buffers that naturally occur in soil and sediments. A Pearson correlation analysis was used to determine the presence of a relationship between particle size and EC50s.

RESULTS AND DISCUSSION

The sediment characteristics were: CEC from non-detectable in Gordy Road and White City Park to 160 meq/100g in Lake Okeechobee, AVS from non-detectable in Lake Okeechobee to 665 mg/kg in S-80, pH from 6.95 in S-80 to 7.3 in STA-2, S99 and White City Park, TOC from non-detectable in Gordy Road and White City Park to 20,600 mg/kg in STA-2, total ammonia from 2.6 mg/L in STA-2 to 14.0 mg/L in Lake Okeechobee, and calcium carbonate from non-detectable in Gordy Road and White City Park to 188,000 mg/kg in S-6. Gordy Road, White City Park and S-99 are predominantly sandy loams (> 68% sand) and S-6, STA-2, S-80 and Lake Okeechobee are silty/clay sediments (>64% silt/clay). Tables 1 and 2 show the analytical data for organic contaminants and trace metals.

Figure 2 shows the EC50s of formulated reference sediments and all sediment samples from the SLR and EAA sites. The formulated sediment (FSA) with high sand (~80%) content and low TOC (~1.5%) had an EC50 of 142,200 mg/L. The formulated sediment (FSB) with the highest TOC (~7.4 %), and high sand (~63 %) content had the highest EC50 value of 209, 800 mg/L. The formulated sediment (FSC) with low sand (~14 %) content, high silt and clay (~80%), and

Table 1. Concentrations of organic contaminants in sediment.**S6**

| Compound | Conc. (µg/kg) |
|-----------|---------------|
| Chlordane | 19 |
| DDD-p,p' | 7.8 |
| DDE-p,p' | 24 |
| DDT-p,p' | 2.4 |
| Ametryn | 5.2 |
| Bromacil | 43 |

S80 1

| Compound | Conc. (µg/kg) |
|----------------------|---------------|
| Benzo(a)anthracene | 340 |
| Benzo(a)pyrene | 180 |
| Benzo(b)fluoranthene | 290 |
| Benzo(k)fluoranthene | 120 |
| Chrysene | 310 |
| Fluoranthene | 1300 |
| Pyrene | 670 |
| Norflurazon | 13 |

S80 2

| Compound | Conc. (µg/kg) |
|----------------------|---------------|
| Benzo(a)anthracene | 64 |
| Benzo(a)pyrene | 50 |
| Benzo(b)fluoranthene | 160 |
| Benzo(k)fluoranthene | 160 |
| Chrysene | 110 |
| Fluoranthene | 160 |
| Pyrene | 120 |
| Norflurazon | 25 |
| DDE-p,p' | 5 |

S99

| Compound | Conc. (µg/kg) |
|-------------------|---------------|
| Diethyl phthalate | 7800 |

Lake Okeechobee

| Compound | Conc. (µg/kg) |
|----------|---------------|
| DDE-p,p' | 6.5 |

low TOC (~1.5%) had the lowest EC50 value of 20,100 mg/L. This may support earlier findings by Benton et al. (1995) and Ringwood et al. (1997) in that with clean sediments as mean particle size decreases (i.e., percent silt or clay increases) sediment toxicity increases in the Microtox[®] assay.

Sample sites S-99, S-6 and S-80 have sediments with the lowest EC50 values. The EC50 values do not correlate with particle size distribution. EC50s at S-99 show no correlation with contaminant concentrations. However, S-6 and S-80 sediment contained multiple organic contaminants and trace metals. Lake Okeechobee sediment was not toxic to bacteria, compared to the aforementioned sites, but the mercury concentration (0.39 mg/kg) exceeded threshold effect levels in sediment used as a screening benchmark by the National Oceanic and Atmospheric Administration (NOAA). The SFWMD pesticide monitoring database was compiled for S-99, S-80, and S-6 for 1992-1999 (Pfeuffer 2002). It indicates that all three sampling sites have a history of detectable pesticide concentrations. However, the important points to note are that more than one pesticide is typically detected in water and sediment at each site and that organochlorine compounds are ubiquitous. To characterize acute hazards in the ecological risk assessment process a set or subset of a species distribution of LC50 and EC50 toxicity data are usually compared to distributions of single chemical exposures either obtained from actual measured exposure concentrations or from models (SETAC 1994). In the SFWMD pesticide database, chlordane, DDT and ethion acute toxicity data for aquatic species exceeded background levels of these chemicals in sediment (at S-6 and S-99). Several other chemicals

Table 2. Concentrations of trace metals in sediment.**S6**

| Metal | Conc. (mg/kg) |
|-----------|---------------|
| Aluminum | 3820 |
| Arsenic | 1.7 |
| Beryllium | 0.11 |
| Chromium | 6.9 |
| Copper | 12.5 |
| Iron | 4140 |
| Lead | 4.6 |
| Mercury | 0.025 |

S80 1

| Metal | Conc. (mg/kg) |
|-----------|---------------|
| Aluminum | 6700 |
| Arsenic | 3.8 |
| Beryllium | 0.44 |
| Chromium | 13.8 |
| Copper | 16.4 |
| Iron | 9990 |
| Lead | 21.7 |
| Nickel | 4.7 |
| Zinc | 34 |
| Mercury | 0.076 |

S80 2

| Metal | Conc. (mg/kg) |
|-----------|---------------|
| Aluminum | 8600 |
| Arsenic | 4.8 |
| Beryllium | 0.57 |
| Cadmium | 0.46 |
| Chromium | 17.7 |
| Copper | 26.5 |
| Iron | 13200 |
| Lead | 15.6 |
| Nickel | 5.4 |
| Zinc | 61 |
| Mercury | 0.11 |

White City Park

| Metal | Conc. (mg/kg) |
|----------|---------------|
| Aluminum | 170 |
| Copper | 2.4 |
| Iron | 224 |

S99

| Metal | Conc. (mg/kg) |
|-----------|---------------|
| Aluminum | 1890 |
| Beryllium | 0.084 |
| Chromium | 3.1 |
| Copper | 17.7 |
| Iron | 1940 |
| Lead | 2.2 |

Gordy Road

| Metal | Conc. (mg/kg) |
|----------|---------------|
| Aluminum | 149 |
| Copper | 5.6 |
| Iron | 193 |

STA-2

| Metal | Conc. (mg/kg) |
|-----------|---------------|
| Aluminum | 6060 |
| Arsenic | 2.4 |
| Beryllium | 0.17 |
| Chromium | 7.4 |
| Copper | 3.3 |
| Iron | 3750 |
| Lead | 5.0 |
| Mercury | 0.034 |

Lake Okeechobee

| Metal | Conc. (mg/kg) |
|-----------|---------------|
| Aluminum | 9370 |
| Arsenic | 6.6 |
| Beryllium | 0.68 |
| Chromium | 23.3 |
| Copper | 14.6 |
| Iron | 17400 |
| Lead | 19 |
| Nickel | 12 |
| Selenium | 2.9 |
| Silver | 0.15 |
| Mercury | 0.39 |

Table 3. Microtox[®] EC50s for sediment. WCP = White City Park; GR = Gordy Road; LO = Lake Okeechobee; STA = Stormwater Treatment Area; FS = formulated reference sediment.

| Site | EC50 | 95% Confidence Interval |
|---------|--------|-------------------------|
| S-99 A | 322.1 | 118.5 to 875.4 |
| S-99 B | 131.7 | 41.09 to 422.3 |
| S-99 C | 252.6 | 84.31 to 757.1 |
| WCP A | 32240 | 23040 to 45100 |
| WCP B | 44020 | 39400 to 49180 |
| WCP C | 35690 | 22710 to 56110 |
| GR A | 18270 | 2225 to 150100 |
| GR B | 16810 | 14720 to 19190 |
| GR C | 23600 | 15180 to 36690 |
| S-6 A | 3420 | 2092 to 5589 |
| S-6 B | 3727 | 2642 to 5257 |
| S-6 C | 1012 | 485.0 to 2111 |
| S-80 A1 | 1175 | 649.8 to 2126 |
| S-80 A2 | 242.0 | 125.8 to 465.6 |
| S-80 B1 | 444.4 | 309.9 to 637.3 |
| S-80 B2 | 1005 | 966.9 to 1045 |
| S-80 C1 | 280.8 | 154.2 to 511.3 |
| S-80 C2 | 42.20 | 25.27 to 70.48 |
| STA2 | 9565 | 7024 to 13020 |
| LO | 13900 | 8990 to 21480 |
| FSA | 142200 | 84980 to 237800 |
| FSB | 209800 | No confidence range |
| FSC | 20100 | 17880 to 22590 |

(e.g., dieldrin, dicofol) also showed a small margin of safety between actual measured sediment exposures and acute toxicity values. The use of multiple chemical exposure scenarios to quantify and derive toxicity endpoints would be a more accurate approach at these sites because it is very probable that adverse biological effects may occur as a result of the interaction of low levels of multiple chemicals together which may not elicit such effects alone. Furthermore, background sediment concentrations of organochlorine chemicals may be compromising challenges with environmental exposures of organisms to chemicals presently being used, rendering them more toxic. Year two of the study will include single-species freshwater sediment toxicity testing.

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REFERENCES

Benton MJ, Malott ML, Knight SS, Cooper CM, Benson WH (1995) Influence of

- sediment composition on apparent toxicity in a solid-phase test using bioluminescent bacteria. *Environ Toxicol Chem* 14: 411-414
- Chimney MJ, Nungesser M, Newman J, Pietro K, Germain G, Lynch T, Goforth G, Moustafa MZ (2000) Stormwater Treatment Areas: Status of research and monitoring to optimize effectiveness of nutrient removal and annual report on operational compliance. Everglades Consolidated Report. South Florida Water Management District, West Palm Beach, FL
- Florida DEP (1997) North Fork St. Lucie River and St. Lucie Estuary. Southeast District Ambient Monitoring Program, Ecosummary, Port St. Lucie, FL
- Graves GA, Strom DG (1995) Pesticide Contamination in Ten Mile Creek. Ecosystem Management Report. Florida DEP Southeast District Ambient Water Quality Section, Port St. Lucie, FL
- Haag KH, McPherson BF (1997) Organochlorine pesticides and PCBs in southern Florida fishes: then and now. USGS NAWQA Fact Sheet-110-97
- Harrahy EA, Clements WH (1997) Toxicity and bioaccumulation of a mixture of heavy metals in *Chironomus tentans* (Diptera: Chironomidae) in synthetic sediments. *Environ Toxicol Chem* 16: 317-327
- Kemble NE, Dwyer FJ, Ingersoll CG, Dawson TD, Norberg-King TJ (1999) Tolerance of freshwater test organisms to formulated sediments for use as control materials in whole-sediment toxicity tests. *Environ Toxicol Chem* 18: 222-230
- Lyman WJ, Glazer AE, Ong JH, Coons SF (1987) An overview of sediment quality in the United States. EPA-905/9-88-002. Washington, DC
- Miles CJ, Pfeuffer RJ (1997) Pesticides in canals of south Florida. *Arch Environ Toxicol* 32: 337-345
- Pait AS, De Souza AE, Farrow DRG. (1992) Agricultural pesticide use in coastal areas: A national summary. National Oceanic and Atmospheric Administration, Rockville, MD
- Pfeuffer RJ (1991) Pesticide residue monitoring in sediment and surface water within the South Florida Water Management District: Volume 2. Technical Publication 91-01, South Florida Water Management District. West Palm Beach, FL
- Pfeuffer RJ (2002) Pesticide monitoring reports (1992-1999). South Florida Water Management District. West Palm Beach, FL
- Ringwood AH, DeLorenzo ME, Ross PE, Holland AF (1997) Interpretation of Microtox[®] solid-phase toxicity tests: the effects of sediment composition. *Environ Toxicol Chem* 16: 1135-1140
- Science Subgroup (1996) South Florida Ecosystem Restoration: Scientific Information Needs. Report to the Working Group of the South Florida Ecosystem Restoration Task Force. Miami, FL
- Society Environmental Toxicology and Chemistry (SETAC) (1994) Aquatic Dialogue Group: Pesticide Risk Assessment and Mitigation. SETAC Foundation for Environmental Education, Pensacola, FL
- Walsh GE, Weber DE, Simon TL, Brashers LK (1991) Toxicity tests of effluents with marsh plants in water and sediment. *Environ Toxicol Chem* 10: 517-525