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Pesticide Body Residues of *Hyalella azteca* Exposed to Mississippi Delta Sediments

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Mississippi Delta portions of the Lower Mississippi Alluvial Plain are among the most intensively cultivated regions in the United States. The Delta has a long growing season and an average annual rainfall of approximately 130 cm, allowing weed and insect pests to flourish and requiring significant use of pesticides to control infestations and subsequent crop loss. Because of the widespread but controlled use of pesticides, the potential exists for low-level runoff into receiving watersheds (Snipes et al., 2004). Although surface water pesticide loads are often below effects concentrations, watershed sediments, acting as sinks, can accumulate these materials, potentially exposing benthic, epibenthic, and even aquatic organisms should sediments become resuspended in the water column (Cooper et al., 2003). In determining sediment quality, it is important to assess direct effects of contaminated sediments on organisms. However, there is also a need to assess potential indirect effects such as bioaccumulation within the ecological food web (Moore et al., 2005). The freshwater crustacean *Hyalella azteca* (Order: Amphipoda) is an epibenthic detritivore closely associated with surficial sediments. *H. azteca* occurs in lakes throughout most of Central and North America and is an important food source for larger invertebrates, fish, waterfowl, and amphibians (de March, 1981). The current study examines accumulation of pesticides in the test organism *Hyalella azteca*, exposed for 28 d to sediments from nine watersheds in the Mississippi Delta: three reference watersheds in the White River National Wildlife Refuge (NWR), three with active best management practices (BMPs), and three listed for legacy pesticide total maximum daily loads (TMDLs) according to USEPA Clean Water Act section 303(d).

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Materials and Methods

Surface sediment samples (top 5 cm) were collected during the summer (June to July) of 2004 at three sites within each of nine watersheds located in the Mississippi Delta, divided into three categories: NWR (reference), BMP, and 303d (impaired). Three NWR watersheds were Columbus Lake and Lower White Lake in Arkansas County, AR, USA, and Upper Swan Lake in Monroe County, AR, USA. Three BMP watersheds were Beasley Lake and Thighman Lake in Sunflower County, MS, USA, and Deep Hollow Lake in Leflore County, MS, USA. Three 303d watersheds were Bee Lake in Holmes County, MS, USA, Cassidy Bayou in Tallahatchie County, MS, USA, and Roebuck Lake in Leflore County, MS, USA.

Twenty-eight day static, nonrenewal whole sediment exposures using *Hyalella azteca* were performed according to Nebeker et al. (1984) and USEPA (1994). At the conclusion of the 28 d exposure period, surviving animals were counted, weighed (as wet weight), and placed in 5 mL pesticide-grade ethyl acetate for extraction and pesticide analysis using a method similar to that reported by Bennett et al. (2000) with modifications. The mixture was sonicated and centrifuged at 2000–2500 rpm, and the extract was concentrated to 1 mL volume using a high purity nitrogen evaporator. The 1 mL extract was subjected to silica gel column clean up and was reconcentrated to 1 mL under dry nitrogen for GC analysis.

Analytical chemistry was performed with two Hewlett Packard (now Agilent) model 6890 gas chromatographs equipped with dual HP 7683 ALS autoinjectors, dual split-splitless inlets, dual capillary columns, and a HP Kayak XA Chemstation that were all used to conduct all pesticide analyses. One HP 6890 was equipped with two HP microelectron capture detectors and the other with one HP microelectron capture detector, one HP nitrogen phosphorus detector, and a HP 5973 mass selective detector (Smith et al., 2006). Based on past and present pesticide use in the Mississippi Delta, 14 pesticides and three metabolites were targeted for analysis (Table 1).

H. azteca pesticide body residue data were analyzed using descriptive statistics and Kruskal-Wallis one-way ANOVA on ranks with Student-Newman-Keul's (SNK) multiple range test (Steel et al., 1997). Linear regression was performed on log 10 transformed growth (as wet weight) versus log 10 transformed pesticide concentration. Data analysis was conducted using SigmaStat® v.2.03 statistical software (SPSS, 1997).

Results and Discussion

Chemical analysis revealed variation in animal pesticide body residue concentrations for 11 of 17 pesticides examined among the three watershed categories (Table 1). Two herbicides, trifluralin and pendimethalin, had no measurable body residue amounts in animals exposed to sediments from any watershed. Herbicides cyanazine, alachlor, and metolachlor were greatest in animals exposed to BMP category sediments. Atrazine occurred in the greatest concentrations of any pesticide examined, but atrazine was not significantly different among watershed categories (Table 1). Within individual watersheds, *H. azteca* exposed to BMP category sediments Beasley Lake, Thighman Lake, and Deep Hollow Lake had the greatest herbicide body residues. Intermediate herbicide body residues were observed in animals exposed to 303d watersheds, Roebuck Lake, Cassidy Bayou, and Bee Lake. Lowest herbicide body residues occurred in organisms exposed to NWR watersheds, Upper Swan Lake, Lower White Lake, and Columbus Lake (Fig. 1). Insecticides chlorpyrifos, methyl parathion, chlorfenapyr, and λ-cyhalothrin were greatest in animals exposed to BMP sediments (Table 1). Greatest total insecticide body residues occurred in animals from BMP lake sediments: Beasley, Thighman, and Deep Hollow Lake sediments, and lowest in Cassidy Bayou (303d) and Columbus Lake (NWR) (Fig. 1). The metabolite, fipronil sulfone, was greatest in animals exposed to BMP watershed sediments and was lowest in NWR (Table 1). Legacy compounds (*p,p′*-DDT and metabolites) were greatest in *H. azteca* exposed to BMP and 303d sediments (Table 1). Specific watershed sediments from Beasley Lake (BMP), Roebuck Lake (303d), and Cassidy Bayou (303d) produced the greatest total legacy compound animal body residues whereas Columbus, Upper Swan, and Lower White lakes (NWR) consistently had the lowest (Fig. 1).

Table 1 Median *Hyalella azteca* pesticide body residue concentrations (nonlipid-normalized; ng/g) exposed to White River National Wildlife Refuge (NWR), Best Management Practices (BMP), and USEPA section 303(d) Clean Water Act (303d) watershed sediments

Pesticide	Type	NWR	BMP	303d
Trifluralin	Herbicide	0[a]A	0[a]A	0[a]A
Pendimethalin	Herbicide	0[a]A	0[a]A	0[a]A
Atrazine	Herbicide	946 A	4835 A	2435 A
Cyanazine	Herbicide	1 A	192 B	0[a] A
Alachlor	Herbicide	0[a] A	2399 B	0[a] A
Metolachlor	Herbicide	0[a] A	3251 B	0[a] A
Chlorpyrifos	Insecticide	90 A	3117 B	0[a] A
Methyl Parathion	Insecticide	2 A	2189 B	0[a] A
Fipronil	Insecticide	5 A	0[a] A	0[a] A
Fipronil Sulfone	Metabolite	14 A	201 B	22 A
Chlorfenapyr	Insecticide	5 A	216 B	26 A
Bifenthrin	Insecticide	7 A	138 A	64 A
λ-Cyhalothrin	Insecticide	158 A	1001 B	709 AB
Dieldrin	Legacy Insecticide	1 A	40 A	8 A
p,p′-DDE	Legacy Metabolite	4 AB	2 A	35 B
p,p′-DDD	Legacy Metabolite	13 A	45 AB	76 B
p,p′-DDT	Legacy Insecticide	502 A	2642 B	2160 AB

Median values with different capital letters are statistically significantly different ($p < 0.05$)

[a] Below detection limit (1 ng/g)

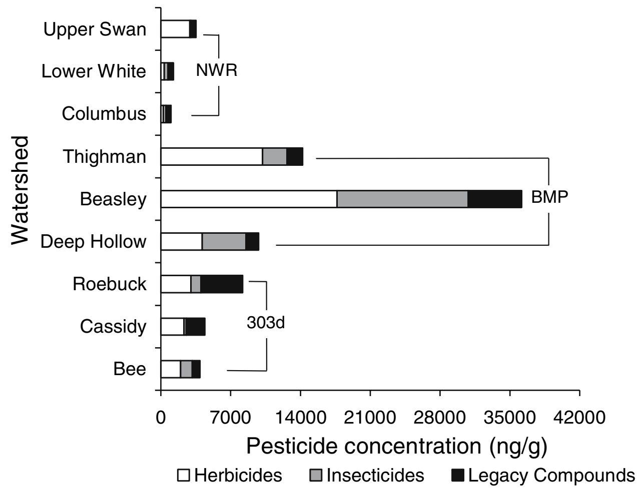

Fig. 1 *Hyalella azteca* pesticide body residues from sediments of nine watersheds in the Mississippi Delta

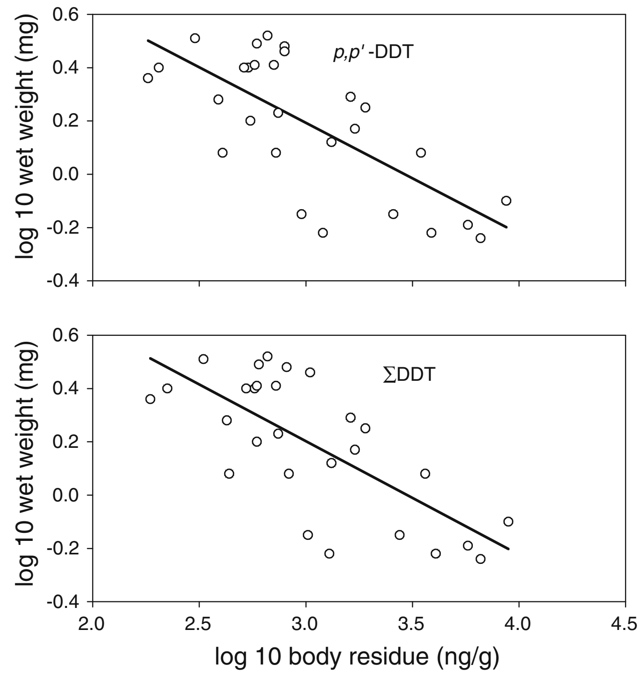

Fig. 2 Relationships between *p,p′*-DDT and ΣDDT body residues and growth (as wet weight) of *Hyalella azteca* exposed to Mississippi Delta sediments

Few studies have attempted to assess pesticide body residues in *Hyalella azteca* (Lotufo et al., 2000; Lotufo et al., 2001; Landrum et al., 2005). These published reports focused exclusively on body residues of legacy compounds, such as *p,p′*-DDT and metabolites, *p,p′*-DDD and *p,p′*-DDE. In the present study, legacy compounds occurred in tissues of animals exposed to sediments from all nine watersheds with current-use herbicides and *p,p′*-DDT predominant. Similar patterns of concentrations of *p,p′*-DDT occurred in fish tissues within the same region (Cooper, 1991; Knight and Cooper, 1996). Limited information exists on current-use insecticide body residues in crustaceans (Burgos-Hernández et al., 2005). Crustaceans in this study showed higher tissue concentrations than farmed shrimp from coastal lagoons in Mexico (Burgos-Hernández et al., 2005), due, in part, to more intensive row crop agriculture within the Mississippi Delta. In comparison with Mississippi fish tissues, *H. azteca* shows a greater propensity for accumulating current-use insecticides (Cooper, 1991; Knight and Cooper, 1996).

Comparisons of body residues among the three watershed categories provide three conclusions. First, some current-use pesticides are available to animals exposed to NWR reference sediments despite being in a national wildlife refuge. Second, BMPs have little if any impact on pesticide sequestration in *Hyalella azteca*. Third, 303d watershed sediments (listed for legacy pesticide TMDLs) had few available current-use insecticides but greater legacy compounds accumulated in *H. azteca* tissues.

Biologically relevant relationships were observed between *Hyalella azteca* 28 d growth and legacy compound body residues. Body residues of the legacy compounds, *p,p′* DDT (R^2 = 0.5125, F = 27.3, p < 0.001) and ΣDDT (sum of DDT and metabolites; R^2 = 0.5166, F = 27.8, p < 0.001), were significantly related to animal growth (Fig. 2). Tissue concentrations of these compounds elicited biologically significant growth impairment after 28 d sediment exposures. Despite banning *p,p′*-DDT from use within the United States in 1972 (USEPA OPP, 2001), this organochlorine insecticide and its metabolites persist with the potential to impact ecosystem components of Mississippi Delta watersheds more than 30 years later.

Based on *Hyalella azteca* pesticide body residues accumulated via contaminated Mississippi Delta sediments, significant concentrations of legacy compounds, insecticides, and herbicides can move from sediment to *H. azteca* tissues within 28 d eliciting biologically relevant growth impairment. Further studies are needed to elucidate the role of detritivore body residue pesticide contamination within aquatic ecosystems and associated effects on predatory aquatic organisms.

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References

Bennett ER, Moore MT, Cooper CM, Smith S Jr (2000) Method for the simultaneous extraction and analysis of two current use pesticides, atrazine and lambda-cyhalothrin, in sediment and aquatic plants. Bull Environ Contam Toxicol 64:825–833

Burgos-Hernández A, Garcia-Sifuentes CO, Aldana-Madrid ML, Meza-Montenegro MM (2005) Detection and quantification of insecticides in shrimp grown in a coastal farm in Sonora, Mexico. Bull Environ Contam Toxicol 74:335–341

Cooper CM (1991) Persistent organochlorine and current use insecticide concentrations in major watershed components of Moon Lake, Mississippi, USA. Arch Hydrobiol 121:103–113

Cooper C, Smith S Jr, Moore M (2003) Surface water, ground water and sediment quality in three oxbow lake watersheds in the Mississippi delta agricultural regions: pesticides. Int J Ecol Environ Sci 29:171–184

de March BGE (1981) Hyalella azteca (Saussure). In: Lawrence SG (ed) Manual for the Culture of Selected Freshwater Invertebrates. Canadian Spec Publ Fish Aquat Sci 54:61–77

Knight SS, Cooper CM (1996) Insecticide and metal contamination of a mixed cover agricultural watershed. Wat Sci Tech 33:227–234

Lotufo GR, Landrum PF, Gedeon ML, Tigue ML, Herche LR (2000) Comparative toxicity and toxicokinetics of DDT and its major metabolites in freshwater amphipods. Environ Toxicol Chem 19:368–379

Lotufo GR, Landrum PF, Gedeon ML (2001) Toxicity and bioaccumulation of DDT in freshwater amphipods in exposures to spiked sediments. Environ Toxicol Chem 20:810–825

Landrum PF, Steevens JA, McElroy M,Gossiaux DC, Lewis JS, Robinson SD (2005) Time-dependent toxicity of Dichlorodiphenyldichloroethylene to *Hyalella azteca*. Environ Toxicol Chem 24:211–218

Moore DW, Baudo R, Condor JM, Landrum PF, McFarland VA, Meador JP, Millward RN, Shine JP, Word JQ (2005) Bioaccumulation in the assessment of sediment quality: uncertainty and potential application. In: Wenning RJ, Batley GE, Ingersoll CG, Moore DW (eds) Use of Sediment Quality Guidelines and Related Tools for the Assessment of Contaminated Sediments. SETAC Press, Pensacola, FL, pp 429–495

Nebeker AV, Cairns MA, Gakstatter JH, Malueg KW, Schuytema GS, Krawczyk DF (1984) Biological methods for determining toxicity of contaminated freshwater sediments to invertebrates. Environ Toxicol Chem 3:617–630

Smith S Jr, Cooper CM, Lizotte RE Jr, Shields FD Jr (2006) Storm pesticide concentrations in Little Topashaw Creek, USA. Int J Ecol Environ Sci 32:173–182

Snipes CE, Evans LP, Poston DH, Nichols SP (2004) Agricultural practices of the Mississippi Delta. In: Nett MT, Locke MA, Pennington DA (eds) Water Quality Assessments in the Mississippi Delta: Regional Solutions, National Scope. ACS Symposium Series 877, American Chemical Society, Washington, DC, pp 43–60

Statistical Package for the Social Sceinces (SPSS), Inc. (1997) SigmaStat for Windows version 2.03

Steel RGD, Torrie JH, Dickey DA (1997) Principles and Procedures of Statistics: A Biometrical Approach. McGraw Hill, New York, NY

Environmental Protection Agency (USEPA) (1994) Methods for measuring the toxicity and bioaccumulation of sediment associated contaminants with freshwater invertebrates. EPA 600/R-94/024

Environmental Protection Agency (USEPA), Office of Prevention, Pesticides and Toxic Substances (OPP) (2001) Priority PBTs: DDT. http://www.epa.gov/pbt/ddt.htm