Pesticide Presence and Concentrations In Surface Waters of Selected Lakes and Reservoirs (<500 acres) of Mississippi

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ABSTRACT

Surface water from 46 small (100 to < 500 acres) lakes and reservoirs located throughout Mississippi was sampled to test for presence and concentration levels of eighteen current-use or residual pesticides or their breakdown products. Each lake was sampled an average of five times from November 4, 2004 to October 6, 2005. In all, 3,988 analyses were performed, and the overall detection rate was 13%. The most frequently detected compound was \sum DDT which exhibited an 85% detection rate. Fortunately, all concentrations were below 0.25 µg/L. Dieldrin, which had an approximate 34% detection rate, had no detections above 0.05 µg/L. The next three most commonly detected compounds were breakdown products of DDT and fipronil. The herbicide 2,4-D was commonly detected, and 13% of tested samples were quantifiable above 1.0 µg/L, with the highest observed concentration being 2.75 µg/L. Atrazine, the seventh most often detected compound, had the highest observed single concentration (14.47 µg/L) of all compounds tested and had an average concentration of 0.125 µg/L. Metolachlor was the only other compound detected at a concentration greater than 1.0 µg/L. Pendimethalin was the only compound not detected in any samples at any of the lakes. Butler Lake and Long Creek Reservoir both had 13 compounds detected. Only two pesticides were detected at Davis Lake, Filter Lake, and Flatland Lake. Watershed land-use revealed few specifics because of the scale of resolution (all mixed cover watersheds). Detection of specific pesticides indicated that urban and agricultural land uses both made substantial contributions to surface water contamination.

Keywords: Water Quality, Surface Water, Nonpoint Source Pollution

Introduction

As humankind increasingly relies on pesticides for production of agricultural products and control of disease and pests, the potential for environmental contamination by these necessary chemicals is a constant concern. Use of pesticides in the United States can be traced to the 1860s. Concern about persistence and negative effects on vertebrates resulted in the banning of organochlorine insecticides in 1972. It also resulted in significant increases in our understanding of detrimental effects of all pesticides, particularly as quantified by toxicity testing (Blus 1995). The United States has roughly 80,000 substantial reservoirs, many of which are public access water bodies. Mississippi has a large number of natural lakes, created by river meander, in addition to thousands of small to large constructed reservoirs. These waterbodies are used for a variety of purposes, including commercial, sports and subsistence fishing, and also swimming and recreation. Previously (Cooper et al. 2004), we reported results of a water quality survey of pesticides in larger (>500 acres) public access lakes and reservoirs located in Mississippi. Herein, we present findings of a similar study of smaller (<500 acre surface area) lakes and reservoirs.

Methods Field Methods and Study Sites

Lake sampling was done from boat by Mississippi Department of Environmental Quality (MDEQ) personnel at 46 lakes throughout Mississippi. Surface water grab samples were collected in specially cleaned, solvent-rinsed glass jars according to United States Environmental Protection Agency (U.S. EPA) recommendations. Samples were immediately placed on ice and transported to the United States Department of Agiculture's National Sedimentation Laboratory in Oxford, MS where they were stabilized, and processed.

Sampling of the 46 lakes resulted in 225 collections. Samples were collected between November 04, 2004, and October 06, 2005, with a single collection period from each lake during winter (November-December 2004), a single collection period during spring (March-April 2005), two collection periods during summer (June-July 2005), and two collection periods during fall (August-October 2005). Some lakes were not sampled every period due to inaccessibility or safety issues.

Lake surface areas ranged from 102 to 459 acres, with a mean area of 215 acres. Lake types included 29 reservoirs, 15 oxbows,

and two natural lowland lakes (Flatland Lake and Lake Gillirad). A summary of watershed land use /

land cover characteristics for sampled lakes during this study is given in Table A (data for Horseshoe Lake were unavailable). Locations for sampled lakes within the state are shown in Figure 1.

Pesticide Analyses

Gas chromatography (GC) was used for all analyses except for 2,4-D. Lake samples for GC analyses were extracted within one hour of receipt by adding 1.0 g KCl and 100 mL pesticide grade ethyl acetate (EtOAc), shaking vigorously by hand for about one minute, and stored at 4°C



Figure 1. Location for sampled lakes within the state.

(usually <24 h) for pesticide analyses via GC using a modified method similar to that of Bennett et al. (2000) and Smith and Cooper (2004). Briefly, sample preparation involved partitioning in a separatory funnel, and discarding the water phase. The EtOAc phase was dried over anhydrous sodium sulfate and concentrated by rotary evaporation to near dryness. The extract was taken up in about five mL pesticidegrade hexane, cleaned up by silica gel column chromatography, and concentrated to 1.0 mL under dry nitrogen for GC analysis. Mean extraction efficiencies, based on fortified samples, were >90% for all pesticides.

Two Hewlett Packard (now Agilent) model 6890 gas

Table A.	Summary of watershed land use/land cover
character	istics for lakes that were sampled during this study
(except H	lorshoe Lake, unavailable).

	Mean	Median	Maximum
Urban Acres	50	0	708
Forest Acres	2513	822	24940
Agriculture	2052	83	43081
Acres			
Pasture Acres	1444	978	11806
Disturbed Acres	10	0	144
Water Acres	321	258	1888
Wetland Acres	984	369	11224
Total Acres	7275	3186	51262
Urban %	1%	0%	6%
Forest %	35%	46%	81%
Agriculture %	14%	2%	87%
Pasture %	23%	25%	57%
Disturbed %	0%	0%	2%
Water %	10%	6%	36%
Wetland %	17%	2%	94%

chromatographs each equipped with dual HP 7683 ALS autoinjectors, dual split-splitless inlets, dual capillary columns, and a HP Kayak XA Chemstation were used to conduct all pesticide analyses (Smith and Cooper 2004). One HP 6890 was equipped with two HP micro electron capture detectors (µECDs) and the other 6890 with one HP µECD, one HP nitrogen phosphorus detector (NPD), and one HP 5973 mass selective detector (MSD).

The main analytical column was a HP 5MS capillary column (30 m x 0.25 mm i. d. x 0.25 µm film thickness). Column oven temperatures were as follows: initial at 85°C for one minute, ramp at 25°C min⁻¹ to 190°C, hold at 190°C for 25 minutes, ramp at 25°C to 230°C, and hold for 30 minutes. The carrier gas was UHP helium at 28 cm sec⁻¹ average velocity with the inlet pressure at 8.64 psi and inlet temperature at 250°C. The µECD temperature was 325°C with a constant make up gas flow of 40 cc min⁻¹ UHP nitrogen. The autoinjector was set at 1.0-µL injection volume in fast mode. Under these GC conditions, all 17 pesticides were analyzed in a single run of 61.80 min. When deemed necessary, pesticide residues were confirmed with a HP 1MS capillary column (30 m x 0.25 mm i.d. x 0.25-µm film thickness) and/or with the MSD. The MSD was used only when there was a question as to the identity of a particular pesticide. Online HP Pesticide and NIST search libraries were used when needed.

Analyses for 2,4-D were made according to EPA Method 4015 screening by immunoassay using the 2,4-D RaPID Assay® enzyme linked immunosorbent assay (ELISA) products and instructions from Strategic Diagnostics Inc. A known quantity of the sample is added to an enzyme conjugate followed by paramagnetic particles with antibodies specific to chlorophenoxy herbicides attached. Both the 2,4-D which may be in the sample and the enzyme conjugate (labeled 2,4-D enzyme) compete for antibody binding sites on the magnetic particles. After allowing the reaction to occur, a magnetic field is applied to hold the paramagnetic particles with 2,4-D and labeled 2,4-D analog bound to the antibodies on the particles, in proportion to their original concentration in the tube. Unbound reagents are decanted. The 2,4-D is then detected by adding hydrogen peroxide and the chromogen, 3,3',5,5'-tetramethylbenzidine, that catalyzes the conversion of the substrate and chromogen mixture to a colored product. The color developed is inversely proportional to the concentration of 2,4-D in the sample and is measured using a photometer at a wavelength of 450 nm. Sample results are compared to results from known standards containing 2,4-D in the range from 1.0 to 50.0 μ g/L.

The concentration level of detection and level of quantification for 2,4-D using the ELISA technique are much higher than those methods for other pesticides using the GC techniques. Additionally, availability of analysis materials for 2,4-D prohibited testing on all samples. Thus, a subset of 163 samples was analyzed. Consequently, data reduction and interpretation for 2,4-D analysis results may differ from that of other pesticides discussed in this paper. A summary of analyte method limits is given in Table B.

Table B. Targeted pesticides, levels of detection (LOC) and							
limitis of quantitation (LOQ) (ng/L). Insecticides in bold.							
Pesticide	LOD	LOQ					
Alachlor	0.5	5					
Atrazine	1.0	10					
Bifenthrin	0.1	1					
Chlorfenapyr	0.5	5					
Chlorpyrifos	0.1	1					
Cyanazine	0.5	5					
p,p′-DDD	0.1	10					
p,p'-DDE	0.1	1					
p,p′-DDT	1.0	10					
Dieldrin	0.1	1					
Fipronil	0.1	1					
Fipronil sulfone	0.1	1					
λ -Cyhalothrin	0.1	1					
Methyl	1.0	10					
parathion							
Metolachlor	1.0	10					
Pendimethalin	0.5	5					
Trifluralin	0.1	1					
2,4-D	*0.70 µg/L	*1 µg/L					
*Note: Levels are given in μ g/L for 2,4-D, all other units							
are in ng/L.							

Results and Discussion Occurrence

During a one year period we collected 225 surface water samples in 46 natural lakes or constructed reservoirs. We tested each of those samples for 18 pesticide analytes (current use, residual organochlorine or their breakdown products) which resulted in 3,988 individual analyses. Pesticides were detected in 13% of the nearly 4,000 individual tests. At least one pesticide was detected in 88% of the 225 surface water samples. Of those 225 surface water "grab" samples, the 26 which had no detections were all collected during the summer (June 2005) period of low rainfall. Frequency of occurrence of individual compounds was dominated by DDT which was followed by its metabolites (Table C). DDT occurred in 85% of all samples analyzed, while DDE (27%) and DDD (15%) ranked third and fifth in pesticide occurrence. The second most commonly observed compound was Dieldrin, with a 34% frequency of occurrence. Fipronil Sulfone, the degradation product of fipronil, the fourth most frequently encountered compound, was detected in 20% of samples. The herbicides with the greatest number of occurrences were 2,4-D (13%) and Atrazine (11%). They ranked sixth and seventh, respectively, in frequency of occurrence.

Occurrence patterns were generally driven by isolated events associated with individual watersheds or more pervasive trends caused by seasonal rainfall patterns and pesticide applications. There was essentially no runoff or contamination during the early summer dry period as shown by only seven pesticide detections of a possible 612. Instances of isolated elevated pesticide concentrations were observed from lakes on single dates. Late in the summer, heightened concentrations of Atrazine, Chlorpyrifos, Dieldrin, Fipronil Sulfone, and DDT were recorded from Lake Henry near Greenwood, MS after thunderstorm-like rainfall. Another example of elevated pesticide concentrations occurred in Long Creek Reservoir near Meridian, MS in July, 2005, where Trifluralin, Methyl Parathion, Alachlor, Chlorpyrifos, Cyanazine, Dieldrin, Fipronil, Chlorfenapyr, Bifenthrin and DDT were associated with large amounts of precipitation over a several day period. Butler Lake near Natchez produced similar results after localized thunderstorm activity. Other observed instances of spiked pesticide concentrations on single dates from single lakes could not definitively be associated with increased rainfall using National Oceanic and Atmospheric Administration weather station information.

Although pesticides were detected in 88% of samples, the majority of detections were associated with ∑DDT or Dieldrin. Since these legacy insecticides have been banned for decades in the United States, time for the insecticides to dissipate beyond detection is the only course of action for general environmental improvement. When we excluded ∑DDT and Dieldrin from the occurrence data, 140 samples (62%) had no detections. Fifty-one additional samples had a single detection, and there were 19 that only contained two detecPesticide Presence and Concentrations In Surface Waters of Selected Lakes and Reservoirs (<500 acres) of Mississippi Cooper, et al

tions. These accounted for 82% of all surface water samples. Pesticides were detected during all seasons. When data were sorted by lake, ∑DDT was found in all lakes. When it was excluded, no lake had pesticide detections year-round. While greatest concentrations were detected in oxbow lakes, comparisons of detections in reservoirs and natural lakes revealed no major differences in seasonal occurrence.

Pesticide Concentrations

While pesticide occurrence per individual grab samples was high, overall concentrations were very low (Table C). However, of the 51 samples that contained only one pesticide detection, 16 had values greater than 0.1 µg/L, possibly indicating that their presence was associated with an individual event. The overall mean concentration of herbicides was 0.02 µg/L. Four herbicides occurred at concentrations above 0.1 µg/L. Of 25 detections, Atrazine, the predominant corn herbicide in the United States, had 11 concentrations above 0.1 µg/L and also had the greatest maximum herbicide concentration (14.47 µg/L). 2,4-D, the most widely used residential herbicide in the United States, occurred in 13% of samples and had a maximum concentration of 2.75 µg/L. While Metolachlor occurred in only two percent of the samples (mean concentration=0.01 µg/L), its maximum concentration was 1.95 µg/L.

The overall mean concentration for insecticides was

Table C. Summary of pesticide occurrence and mean concentration observed for all samples collected during this study. Occurrence is given as number of detections above quantitation limit (Table B) divided by total possible detections (n=225). Concentration is given as ug/L. Insecticides in bold.

Compound	Occurrence	Concentration	Standard Error	Maximum
pp′-DDT	85%	0.0635	0.0025	0.2441
Dieldrin	34% 27% 20%	0.0016 0.0013 0.0027	0.0003 0.0002 0.0005	0.0311 0.0223 0.0637
pp'-DDE				
Fipronil sulfone				
pp'-DDD	15%	0.0013	0.0004	0.0703
2,4-D*	13%	0.1883	0.0780	2.7500
Atrazine	11%	0.1253	0.0697	14.4655
Chlorpyrifos	8%	0.0205	0.0050	0.5726
λ -Cyhalothrin	6%	0.0028	0.0010	0.1967
Chlorfenapyr	5%	0.0003	0.0001	0.0247
Trifluralin	4%	0.0005	0.0002	0.0375
Bifenthrin	4%	0.0029	0.0018	0.3803
Methyl parathion	4%	0.0038	0.0014	0.1490
Fipronil	2%	0.0005	0.0002	0.0289
Metolachlor	2%	0.0098	0.0087	1.9510
Cyanazine	1%	0.0004	0.0003	0.0599
Alachlor	1%	0.0040	0.0036	0.8065
Pendimethalin	0%	0.0000	0.0000	0.0000

* Note: The mean concentration value shown for 2,4-D was calculated including non-quantifiable observations using a value of 0.00, while occurrence here is based only on quantifiable readings. See text for more information.

only 0.01 μ g/L. Maximum concentrations are listed in Table C. Thirty-seven \sum DDT concentrations were in excess of 0.1 μ g/L, and the maximum \sum DDT concentration was 0.33 μ g/L. Dieldrin, while commonly encountered, had no detections of 0.1 μ g/L or greater in the 76 samplings where it was present. However, chlorpyrifos, lambda-Cyhalothrin, Bifenthrin, and Methyl Parathion all had at least one observed concentration above 0.1 μ g/L.

Pesticide application and seasonal rainfall patterns created predictable concentration trends. For the six herbicides tested by gas chromatography, highest overall concentrations were found in the summer (0.04 μ g/L), lowest in fall (0.01 µg/L), and intermediate and similar concentrations observed in spring (0.02 μ g/L) and winter samples (0.02 μ g/L). Only five lakes had these herbicides with concentrations of over 1.0 µg/L. Dump Lake (3.65 µg/L, April 2005), an oxbow lake in Yazoo County; Lake Henry (14.47 µg/L, June 2005), an oxbow lake in Leflore County; Lake Mary Crawford (4.65 µg/L, November 2004), a reservoir in Lawrence County; and Little Eagle Lake (1.43 µg/L, July 2005), an oxbow lake in Humphreys County, had high concentrations of Atrazine during one visit. The only other herbicide found above 1.0 µg/L was Metolachlor, observed in Long Lake, an oxbow lake in Sunflower County, at a concentration of 1.95 µg/L in July 2005. Immunoassay tests for the herbicide 2,4-D showed that it had highest concentrations in spring (1.31 µg /L) and summer (1.18 μ g /L), and lower values for fall (0.40 μ g /L) and winter (0.35 μ g /L) seasons. Since the immunoassay test limit of quantification for 2,4-D was 1.0 µg /L, all 21 measurements of 2,4-D were above 1.0 µg /L. However, 24 analyses indicated non-detection, yielding an average concentration of 0.1883 μ g /L. Quantification of lower value concentrations of 2,4-D using a different method would provide a more precise actual average concentration of 2,4-D in Mississippi lakes and reservoirs.

Pesticide detections above 0.1 μ g/L were spread across seasons. Of the 225 lake samples, 15 samples in winter, seven samples in spring, 42 samples in summer, and 12 samples in fall reached 0.1 μ g/L. Summer detections were not directly comparable to the other seasons because lakes were sampled twice in summer, but Σ DDT was present in 32 of the 42 summer surface water samples. Chlorpyrifos exhibited an unexplained seasonal phenomenon; it contaminated 35% of winter samples, and all winter detections exceeded 0.1 µg/L. A few other pesticides showed tendencies toward seasonal trends. Conversely, the only period of the study in which \sum DDT was totally absent from lake samples was during the low rainfall part of the summer sampling.

Pesticides and Land Use Patterns

We delineated major categories of land use (Table A) for each of the 46 sampled watersheds. No watershed had only a single land use. When different principal watershed land uses were compared, the number of occurrences by pesticides followed the trend from agriculture to forest to pasture and wetlands. Unfortunately, urban land use and land cover percent for any watershed were so small as to be highly overshadowed by other uses. Only eleven watersheds had a quantifiable urban percentage.

We tabulated the number of occurrences of individual pesticides for each land use category. Insecticides $\sum DDT$ and Dieldrin dominated instances of occurrence in all land use categories. The weed killer 2,4-D was detected in 20 lakes, making it the most widely detected herbicide. In order of occurrence after **CDDT** and Dieldrin, Fipronil Sulfone was followed by 2,4-D and Atrazine in all land use categories. Chlorpyrifos and lambda-Cyhalothrin were the next most prevalent compounds observed in all categories except urban, where Fipronil and Trifluralin were the next most detected pesticides. Thus SDDT, 2,4-D, Atrazine, and Fipronil Sulfone were common regardless of dominant land use. \sum DDT, the most commonly detected residual insecticide, appears to be ubiquitous as other studies have indicated (Cooper et al. 2004). Low concentrations (mean = $0.05 \mu g/L$) of \sum DDT were present in most samples (85% occurrence). Use of DDT was banned in 1972; use actually peaked in 1968, but its application was so widespread from 1945 to 1972 that it is likely found in every watershed in the state of Mississippi. Fipronil occurred in 2.2% of the 225 samples, and its residual in 20% of samples. It is registered for insect control in corn, indoor pests and turf grass, and for termite control (Termidor®). It is also the active ingredient in tick and flea

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collars (Frontline Plus®).

In our Mississippi study, 2,4-D was detected in 43% (including trace detections) of all sites and was followed by atrazine contamination at 36% of all sites. Several pesticides used extensively in agriculture were infrequently detected. These included the herbicides Metolachlor, Cyanazine, Trifluralin, Alachlor and the insecticide Chlorpyrifos which is also used in non-agricultural settings. Nationwide, 2,4-D has historically been the most used active ingredient in non-agricultural herbicide markets with between seven and nine million pounds used in the home and garden sector and between 17 and 20 million pounds used in the industrycommercial-government sector each year (U.S. EPA 2001). Like 2,4-D, Atrazine, the most commonly used agricultural herbicide in Mississippi other than perhaps glyphosate, was found frequently in small water bodies, but it occurred less frequently than in larger lakes and reservoirs (Cooper et al. 2004). Like within larger lakes in Mississippi, common pesticides were associated with all land uses, not just agriculture (Cooper et al. 2004).

Cooper (1990) found agricultural soils to be a continuing source of DDT in Mississippi. Coupe et al. (2000) studied pesticide occurrence in air and rain from an urban site and an agricultural site in Mississippi. Every sample collected from either site had detections of multiple pesticides although total concentration was five to 10 times higher at the agricultural site. There were six pesticides in current use that were found in more than 20% of the samples taken. Of those six, all but one were insecticides. The lone herbicide was atrazine.

For nationwide comparison, U.S. Geological Survey (U.S.G.S.) analyzed patterns of pesticide use across the United States as part of the National Water Quality Assessment (NAWQA) program (Gilliom et al., 1999) and confirmed that concentrations of herbicides and insecticides in agricultural streams of the nation closely followed use patterns. Urban streams had the highest insecticide concentrations; seven of 11 urban streams had total insecticide concentrations in the upper 25% of all streams sampled, although some agricultural streams in irrigated agricultural areas of the western United States also had high levels. The most frequently detected compounds in agricultural areas were the herbicides atrazine, metolachlor, cyanazine, and alachlor which were ranked in the top five in national herbicide use for agriculture. The most heavily used herbicides also accounted for most of the detections in rivers and major aquifers and many of the detections in urban streams and shallow groundwater (Gilliom et al. 1999). In our study, when watersheds were separated by land use, bearing in mind that urban use was never a significant portion of catchment area, all uses showed a high degree of similarity in compounds detected.

In summary, we found pesticides were measurable in small lakes and reservoirs throughout the state of Mississippi. Banned organochlorine insecticides dominated frequency of occurrence regardless of land use or season. When these residual insecticides are excluded from compound detections, incidence of detection for all 225 surface water samples was reduced from 88% to 62%. The detection rate for all 3,988 individual analyses was 13%. Occurrence was dominated by DDT, Dieldrin, Fipronil Sulfone, 2,4-D, and Atrazine. While greater concentrations were detected in oxbow lakes, comparisons of detections in reservoirs and natural lakes revealed no major differences in occurrence, nor were there any specific compound differences when chemicals were sorted by land use. Occurrence patterns were driven by either isolated rainfall events associated with individual water bodies or larger trends caused by seasonal rainfall patterns and pesticide applications. Four herbicides had concentrations above 0.1 μ g/L; of the 46 lakes in the study, only five lakes had herbicide concentrations that exceeded $1.0 \mu g/L$. Five insecticides exceeded 0.1 µg/L in 31 waterbodies.

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