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Relation of Usage to the Occurrence of Cotton and Rice Herbicides in Three Streams of the Mississippi Delta

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During the 1995 growing season water samples were collected from three streams in the Mississippi delta and were analyzed for selected cotton and rice herbicides and metabolites. The purpose of the study was to relate the use of these herbicides to their occurrence in streams of the delta, to describe how the geochemistry of these herbicides affects their occurrence, and to report the occurrence of selected metabolites. The total concentration of eight herbicides and their metabolites exceeded 5 μ g/L throughout most of the growing season with a median total of 15 μ g/L. The order of occurrence was molinate > fluometuron > cyanazine > metolachlor > norflurazon > atrazine > prometryn > propanil. The distribution and duration of the total herbicide concentration found in this study are much different from that found in regional studies of herbicides in the U.S. Midwest. In the Midwest, the total herbicide concentration in surface water showed a sharp peak during the spring immediately after application of herbicides to crops, followed by a gradual decrease. In the Mississippi delta, the total herbicide concentration in surface water was more sustained, with multiple peaks due to different application times and postemergent applications to cotton and rice.

Introduction

Contamination of water in the midcontinent United States from pesticide application to corn ($Zea\ mays$) has been a major water-quality issue during the past decade. Numerous studies of ground and surface water have been completed by the U.S. Geological Survey (1-3), the U.S. Environmental Protection Agency (4), the U.S. Department of Agriculture (5), state agencies, and many pesticide manufacturers (6). Perhaps equally important to water quality in the southeastern United States is the application of pesticides to cotton ($Gossypium\ hirsutum$) and rice ($Gossypium\ hirsutum$) and rice ($Gossypium\ hirsutum$)

Cotton receives as much as 7 kg/ha of herbicides and 5 kg/ha of insecticides. Rice may receive as much as 6 kg/ha of herbicides but <1 kg/ha of insecticides (7). The intensive application of pesticides is necessary, especially in the humid Southeast where weed and insect pressures are great. Applications of pesticides to cotton and rice are 3–5 times greater per hectare than applications of pesticides to corn and occur more frequently (4.7 annual applications versus 1.2), yet there have been few regional studies of water quality

and pesticide fate in the cotton- and rice-producing areas of the southeastern United States.

There have been many small-scale studies, either field or plot size, that examined the runoff and leaching potential of cotton and rice herbicides (8-14). Coupe (15) conducted a literature search and review as well as a search of the U.S. Environmental Protection Agency's STORET database for historic pesticide data collected within the Yazoo River Basin, Mississippi, and found only a few isolated studies with a small number of samples.

Senseman et al. (16) sampled lakes and streams eight times during a three-year period in four counties of eastern Arkansas for 17 current-use pesticides. The most frequently detected pesticides in this study (16), in order of occurrence, were metolachlor > atrazine > norflurazon > cyanazine in 13, 11.5, 8.2, and 7.4% of the samples, respectively. Pennington (17) collected samples for cotton pesticides in the delta part of the Yazoo River Basin in Mississippi in June and September 1994 and July 1995, and in nearly every sample collected there were detectable concentrations of the cotton pesticides cyanazine, fluometuron, and norflurazon. Pereira and Hosteller (1) collected surface-water samples along the length of the Mississippi River several times during 1991 and 1992. These data indicated that cotton and rice herbicides appear only in the lower Mississippi River downstream from the confluence of the Mississippi and Ohio Rivers and that substantial amounts of these herbicides are being discharged to the Gulf of Mexico (1). The concentrations of these herbicides were highest in the mouth of the Yazoo River, which drains the delta part of Mississippi.

These few water-quality studies indicate that cotton and rice herbicides may be a water-quality issue in the Mississippi delta. Therefore, before a detailed study of the effect of cotton and rice pesticides on water quality throughout the entire cotton- and rice-growing areas of the Southeast was initiated, a reconnaissance study was conducted from April through September 1995 on three streams in the Yazoo River Basin in northwestern Mississippi to determine which compounds and metabolites might occur in surface water. This study was conducted in a joint effort by the U.S. Geological Survey's National Water-Quality Assessment and the Toxic Substances Hydrology Programs.

The major herbicides that were examined in this study included the rice herbicides molinate and propanil and the cotton herbicides cyanazine, fluometuron, metolachlor, and prometryn. In addition, the herbicide atrazine, which is of major importance in the Midwest but of limited usage in the Mississippi delta, was examined. The specific goals of this report are (1) to relate the use of cotton and rice herbicides to their occurrence in surface water of three streams of the Mississippi delta, (2) to report the occurrence in surface water of some of the major metabolites of these cotton and rice herbicides, and (3) to describe how the geochemistry of some cotton and rice herbicides affects their occurrence in surface water.

Study Area and Sample Collection

Surface-water samples were collected from the Steele Bayou near Rolling Fork, Deer Creek near Hollandale, and the Big Sunflower River near Anguilla (Figure 1). The drainage areas for these sites are 1040, 250, and 670 km², respectively. These streams flow generally north to south and are parallel to each other. The study area is in the sparsely populated Yazoo River Basin (YRB). Mississippi's largest river basin, the YRB consists of $\sim\!\!33\,700~{\rm km^2}$ (Figure 1) and is divided almost equally between the Mississippi alluvial plain (referred to as

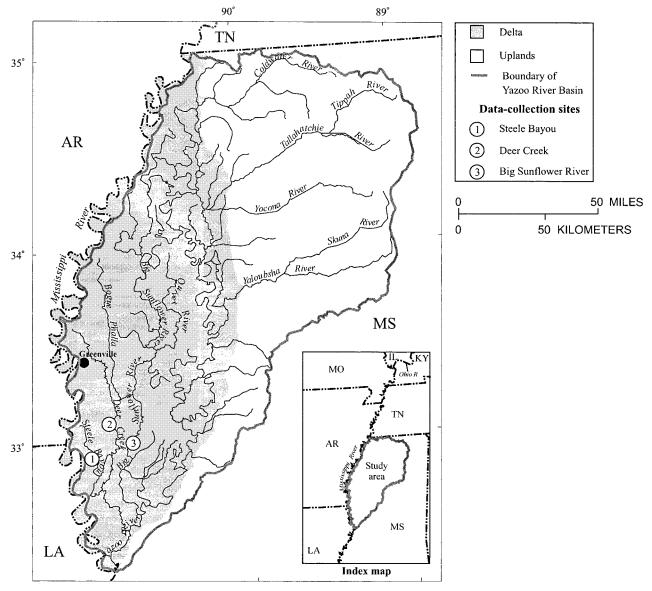


FIGURE 1. Location of data collection sites in the Yazoo River Basin, Mississippi.

the Mississippi delta), an intensive agricultural area of mostly soybean, cotton, and rice production, and the uplands, which generally consist of forests, pastures, and small farms.

The topography of the Mississippi delta is characterized by relatively flat, poorly drained land, with slopes of 5.7-7 cm/km. The climate is subtropical, with long hot summers and short moderate winters. The average annual temperature at Greenville, MS, is $18\,^{\circ}$ C, with average monthly temperatures ranging from $8\,^{\circ}$ C in January to $33\,^{\circ}$ C in July. Normal annual precipitation is $130\,$ cm, with the most intense rainfall occurring from December to April. Minimal rainfall occurs in September and October; however, excessive rainfall, producing locally intense runoff, can occur at any time of the year.

Average velocities in the three streams are quite slow, except at high flow, because of the relatively flat channel slope. In addition, the analytes of interest were in the dissolved phase. Therefore, surface-water samples were collected with an open bottle at the centroid of flow. The samples were filtered on-site using baked glass-fiber filters, with a nominal pore size of $0.7~\mu m$, and a ceramic pump head with Teflon tubing. Samples were placed in the dark, chilled to 4 °C, and shipped to the U.S. Geological Survey laboratory in Lawrence, KS, for analysis. The surface-water

samples were collected weekly to biweekly, beginning April 12, 1995, and ending September 26, 1995.

Experimental Methods

Materials. The C₁₈ cartridges (Sep-Pak from Waters, Milford, MA) contained 360 mg of 40- μ m C₁₈-bonded silica. Phenanthene-d₁₀ was obtained from Ultra Scientific (North Kingstown, RI) and prepared in ethyl acetate. Terbuthylazine was obtained from Supelco (Bellefonte, PA) and prepared in methanol. Atrazine, cyanazine, deethylatrazine, deisopropylatrazine, and prometryn were obtained from Supelco; fluometuron, metolachlor, molinate, and norflurazon were obtained from ChemService (West Chester, PA); demethylfluometuron (DMFM) and trifluoroylmethylphenylurea (TFMPU) were obtained from the U.S. Department of Agriculture, Agricultural Research Service (Stoneville, MS); demethylnorflurazon was obtained from Sandoz Agro. Inc. (Des Plaines, IL); cyanazine-amide was obtained from E. I. DuPont de Nemours & Co. Experimental Station (Wilmington, DE); deisopropylprometryn and trifluoroylmethylaniline (TFMA) was obtained from Ciba-Geigy Corp. (Greensboro, NC); and 3,4-dichloroaniline (3,4-DCA) was obtained from Aldrich Chemical Co., Inc. (Milwaukee, WI).

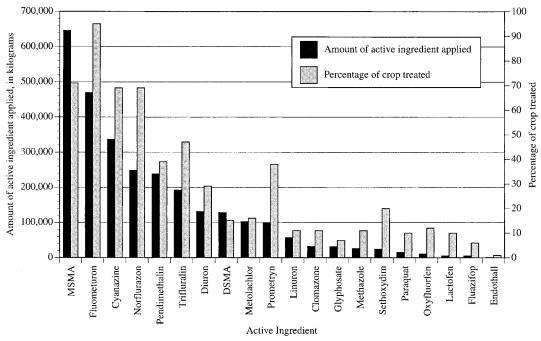


FIGURE 2. Herbicides applied to Mississippi cotton in order of total amount applied and percentage of crop treated [data from Gianessi and Anderson (18)].

Extraction Procedure. Solid-phase extraction was automated on a Waters Millilab workstation (Milford, MA). The C₁₈ Sep-Pak cartridges were conditioned sequentially with 2 mL of methanol, 6 mL of ethyl acetate, 2 mL of methanol, and 2 mL of distilled water. Each 123-mL water sample was spiked with a surrogate standard, terbuthylazine (1.23 ng/ μ L, 100 μ L), and pumped through the cartridge at a rate of 20 mL/min by the robotic probe. Analytes were eluted with ethyl acetate and spiked automatically with phenanthene- d_{10} (0.2 ng/ μ L, 500 μ L). The extract was evaporated by a TurboVap (Zymark, Palo Alto, CA) at 45 °C under a nitrogen stream to 100 μ L.

Gas Chromatography/Mass Spectrometry (GC/MS) Analysis. GC/MS analysis of the elutes was done using a Hewlett-Packard model 5890A GC interfaced to a 5970A mass selective detector (MSD) (Palo Alto, CA). One microliter of sample was injected automatically. Separation of the herbicides was accomplished with a fused-silica capillary column of 5% phenyl methyl silicone (Ultra 2) with a film thickness of 0.33 μ m, 12 m \times 0.2 mm i.d. (Hewlett-Packard, Palo Alto, CA). Helium was used as the carrier gas at a flow rate of 1 mL/min and a head pressure of 35 kPa. The column temperature was held at 60 °C for 1 min, ramped at 6 °C/min to 200 °C, and then ramped at 30 °C/min to 250 °C, where it was held for 4 min. The samples were injected in the splitless mode using an autoinjector at an injector temperature of 180 °C.

The source of the mass spectrometer was held at 280 °C. The emission current was 70 eV. The electron multiplier was set at 400 V above autotune. The filament and multiplier were not turned on until 4 min into the analysis. An autotune using perfluorotributylamine was performed daily prior to analysis of samples. The calibration curve was prepared on the basis of the area ratio of the base peaks relative to the response of the 188 (amu) ion of phenanthene- d_{10} , the internal standard. Confirmation of the compounds was based on the presence of the molecular ion and two confirming ions, a retention time match within 0.2% relative to phenanthene- d_{10} , and correct area ratios of the confirming ions.

The detection and quantitation limits were $0.05\,\mu\text{g}/\text{L}$ for all compounds. All laboratory blanks were free of herbicides or metabolites. The variation of the duplicates was $\pm 5\%$ at

1 standard deviation. The correlation coefficients of the standard curves were 0.998 \pm 0.002. Any samples $^>10\,\mu g/L$ were diluted and reanalyzed.

Results and Discussion

Usage of Cotton and Rice Herbicides. Figure 2 shows the amount of cotton herbicides used by active ingredient and also by total amount of acres treated in Mississippi in 1992. [Crop acreage is from 1992; pesticide use data compiled from 1990 to 1993 (18).] The most used herbicide by kilograms of active ingredient was monosodium methyl arsenate (MSMA); it is used on \sim 70% of the cotton in Mississippi (18). Fluometuron was the most extensively used cotton herbicide, with 95% of cotton acres being treated. The next eight herbicides in order of active ingredient used in Mississippi were cyanazine, norflurazon, pendimethalin, trifluralin, diuron, dimethylsodium arsenate (DMSA), metolachlor, and prometryn (Figure 2). These 10 herbicides account for 90% of all the herbicides that were applied to cotton by mass of active ingredient and also for the majority of the acres that were treated (18). In this paper cyanazine, fluometuron, metolachlor, norflurazon, prometryn, and the metabolites of cyanazine, fluometuron, norflurazon, and prometryn are examined.

Figure 3 shows the amount of rice herbicides used in Mississippi in 1992. The most important rice herbicide in both amount of active ingredient applied and in acreage treated was propanil, with >220 000 kg of active ingredient used and >90% of the rice acres treated. Next was molinate, followed by thiobencarb, glyphosate, and pendimethalin, which make up most of the herbicides applied to rice in Mississippi. The study described in this paper examines two of the five rice herbicides, molinate and propanil, and a degradation product of propanil.

Occurrence of Cotton Herbicides in Surface Water. Figure 4 shows concentrations through time of selected herbicides in the Steele Bayou, Deer Creek, and the Big Sunflower River. Three herbicides, cyanazine, fluometuron, and metolachlor, are representative of the weed-control applications to cotton in the Mississippi delta. Metolachlor concentrations were highest in early April in samples from

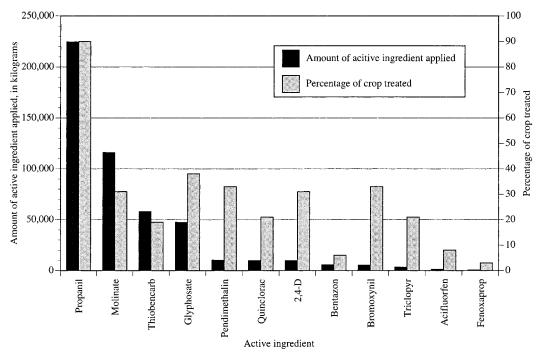


FIGURE 3. Herbicides applied to Mississippi rice in order of total amount applied and percentage of crop treated [data from Gianessi and Anderson (18)].

the Steele Bayou and the Big Sunflower River at concentrations as high as 6.2 and 12.8 μ g/L, respectively. This early detection of metolachlor was probably a result of application to corn, which is also grown in the Mississippi delta. Metolachlor concentrations decreased from this initial peak and then increased again in mid-June. Generally, by the middle of April most of the corn in the delta has been planted, but the cotton, rice, and soybean planting has only just begun. The second peak of metolachlor in mid-June results from metolachlor use on cotton and soybeans and is reflective of the later planting times for these crops. The second peak is of lower concentration and is probably a result of both smaller application rates of metolachlor to cotton and less rainfall and runoff that occurred during the early summer of 1995. Metolachlor appeared later in Deer Creek (during June) without the initial rise attributed to applications of metolachlor to corn in the other basins.

Peak fluometuron concentrations of 6-16 µg/L occurred in May and June from water samples collected from the Steele Bayou, Deer Creek, and the Big Sunflower River (Figure 4). Fluometuron is used primarily as a preemergent herbicide applied at planting but can also be used as a postemergent herbicide. It is used for weed control in cotton, and therefore the timing of the pulse of fluometuron in surface water is much different from metolachlor's, because application times are different for corn and cotton. The concentrations of fluometuron began increasing about the beginning of May and then peaked during the beginning of June. Fluometuron's concentration remained >2.5 μ g/L until early July in samples from all three sampling sites, reflecting the continued use of fluometuron on cotton as a postemergent herbicide. Fluometuron has a half-life of \sim 85 days (19). The solubility of fluometuron is 110 mg/L (19), and it has a low sorption coefficient (K_{oc} of 100). This combination of water solubility, intense use, low sorption coefficient, and half-life are factors in fluometuron occurring in the surface waters of the delta in concentrations >1 μ g/L for over a month during the growing season.

Cyanazine increased from background levels at the start of the study to $> 6 \,\mu g/L$ in samples from Deer Creek during July (Figure 4B). The maximum concentrations occurred

during the third week in July. Cyanazine's major use is as a postemergent herbicide on cotton. This is reflected in its occurrence in the three streams during late July after most of the other herbicides in this study had peaked and the concentrations were decreasing.

Figure 4 demonstrates a fundamental difference in how agricultural chemicals affect water quality in the Mississippi delta compared to the corn-growing areas of the Midwest. In surface water of the Midwest, the maximum concentrations of agricultural chemicals occur during the "spring flush" period (2). That is, herbicides are applied to corn and soybeans during planting, which occurs during a relatively short time period throughout the Midwest. Thus, the occurrence of herbicides in the surface water of the Midwest follows rainfall after planting (i.e., the spring flush). In the Mississippi delta, the long growing season allows farmers more flexibility in the types of crops planted and a longer planting time. In addition, cotton has applications of herbicide as much as 12 weeks after planting, resulting in the occurrence of cotton herbicides in surface water from early April until August as shown in Figure 4.

Figure 5 shows the pattern of herbicide and metabolite occurrence in samples from the Big Sunflower River for fluometuron, norflurazon, cyanazine, and prometryn. Fluometuron occurred at concentrations of 0.5 to almost $7 \mu g/L$ during the growing season. The degradation products contributed little to the total concentration early in the season but became proportionally greater in July (Figure 5A). Two of the three fluometuron metabolites that were determined were demethylated metabolites DMFM and TFMPU. These metabolites were reported by Mueller and Moorman (20) as the major soil metabolites of fluometuron. Of these two metabolites, the first degradation product, DMFM, was the most common and present in the highest concentration. TFMPU was not detected in any sample (data not shown). TFMA, the third metabolite of fluometuron, was detected infrequently and in low concentrations.

Norflurazon and demethylnorflurazon (Figure 5B) were also detected in samples from the Big Sunflower River. The highest concentration of norflurazon occurred early in the season (April and May) and then decreased later in the season.

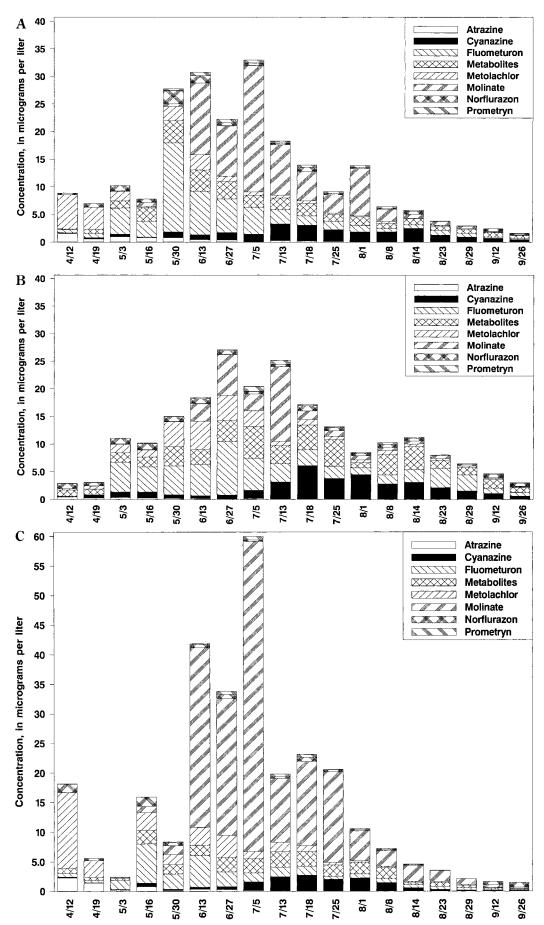


FIGURE 4. Total concentrations of herbicides and selected metabolites in (A) Steele Bayou, (B) Deer Creek, and (C) the Big Sunflower River, 1995.

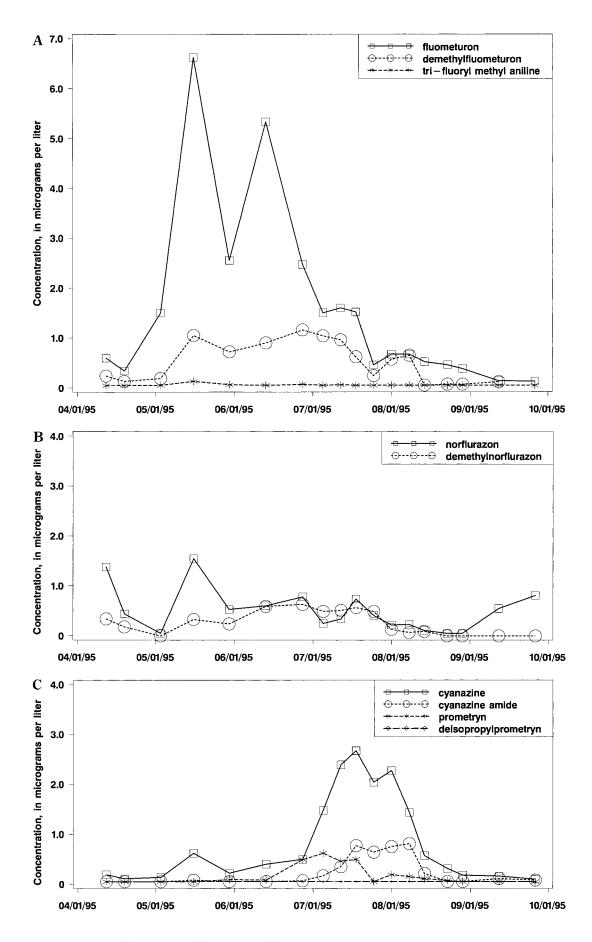


FIGURE 5. Concentrations of (A) fluometuron, (B) norflurazon, (C) cyanazine, and prometryn and selected metabolites in the Big Sunflower River, 1995.

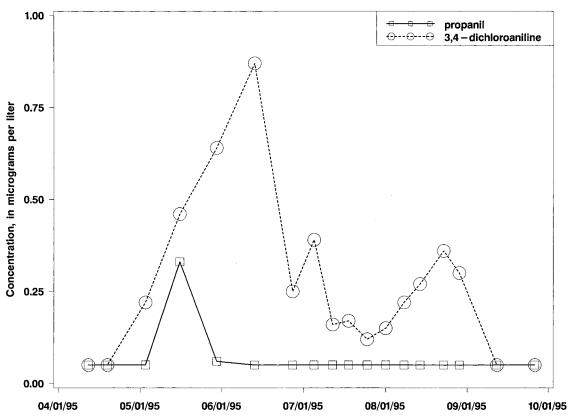


FIGURE 6. Concentrations of propanil and 3,4-dichloranline in the Steele Bayou, 1995.

During this time, the concentration of the degradation product, demethylnorflurazon, increased, which indicates that soil degradation and dissipation of the parent compound occurred. Norflurazon occurred at lower concentrations than fluometuron because of its less intensive use (half as much) and possibly shorter half-life [fluometuron has an 85-day half-life and norflurazon's half-life is from 45 to 180 days (19)].

Cyanazine, prometryn, and their metabolites were also detected in samples from the Big Sunflower River (Figure 5C). Cyanazine amide, a major degradation product of cyanazine (21), occurred with the parent compound at about half the concentration of the parent compound and increased relative to the parent compound later in the season (August). Prometryn was not detected above 1.0 μ g/L, and its degradation product, deisopropylprometryn, was not detected above the reporting level of $0.05\,\mu$ g/L. The less extensive use (5 times less than fluometuron) probably was responsible for the low concentrations in samples from the Big Sunflower River. Samples from the other two sites, the Steele Bayou and Deer Creek, had a similar distribution of prometryn and no detection of deisopropylprometryn (data not shown).

Occurrence of Rice Herbicides in Surface Water. The highest concentration of molinate, $50\,\mu g/L$, was in a sample from the Big Sunflower River (Figure 4C). Concentrations increased rapidly during mid-June to a peak concentration of $50\,\mu g/L$ during early July. Concentrations decreased to $\sim 1\,\mu g/L$ during late August. The occurrence of molinate in samples from the Steele Bayou and Deer Creek followed the same general pattern but was less (Figure 4A,B); the maximum concentrations were 23 and $13.5\,\mu g/L$, respectively. Molinate, with a water solubility of 970 mg/L (19), is applied directly to the rice flood when used as a postemergent herbicide. This flood can be released to the receiving streams when the field is flushed or when it is drained before harvest.

The concentration of propanil was generally equal to or less than the reporting level of the method (0.05 μ g/L)

throughout the study (Figure 6). A metabolite of propanil, 3,4-DCA, was detected after the first of May, at all sites, with a maximum concentration of $0.9\,\mu\mathrm{g/L}$ in a sample from Deer Creek. Although 3,4-DCA is a metabolite of propanil (22), it is possible that other pesticides may metabolize to 3,4-DCA (23). However, given the large amount of propanil used in the Mississippi delta compared to other herbicides that may degrade to 3,4-DCA such as diuron, it is assumed that the majority of the 3,4-DCA observed came from propanil. Molinate is detected much more often than propanil and in much higher concentrations, yet there is twice as much propanil used as molinate (Figure 2). This is probably a result of propanil's half-life of 1 day (molinate's is \sim 21 days) and the application of molinate directly into the rice flood.

Occurrence of Total Herbicides and Selected Metabolites in Surface Water. Total herbicide concentrations (parent compounds plus degradation products) ranged from 2.5 to >25 μ g/L in April and May (Figure 4). The concentrations in April were due to the use of corn herbicides, primarily atrazine and metolachlor. From late May through late July, total concentrations peaked at $27-60 \mu g/L$. The combination of cotton and rice herbicides was responsible for these totals, with major contributions from molinate (rice), fluometuron (cotton), metolachlor (cotton), cyanazine (cotton), and norflurazon (cotton). Quantified metabolites made up ~ 10 20% of the total concentration and maintained a steady relation to the total herbicide concentration throughout the study. Later in the growing season (late August or early September), concentrations of total herbicides decreased to $< 5 \mu g/L$. The total herbicide concentration was $\ge 1.5 \mu g/L$ at the end of the study at all three sampling sites. The total herbicide concentrations dissipated in a similar pattern in each of the three streams.

The total herbicide concentration in streams of the Mississippi delta was of the same order of magnitude as found in Midwest streams following application. However, there was a larger variety of herbicides in delta streams, and the

concentrations were more sustained with multiple peaks due to different application times and postemergent applications to cotton and rice. For instance, atrazine and metolachlor, two herbicides used preemergent on corn in the delta, had their maximum concentrations in April versus May to June in the corn belt. In contrast, fluometuron, a preemergent herbicide used on cotton, has peak concentrations at the end of May and early June, but the concentrations dissipated slowly, due to fluometuron's use as a postemergent herbicide. Molinate, a postemergent herbicide used on rice, has peak concentrations around the middle to the end of June, and cyanazine, used as a postemergent herbicide on cotton, has its peak concentrations in July.

The factors of high usage, more continuous application, long half-lives, and solubility all play important roles in increasing and sustaining concentrations of herbicides in streams in the cotton-growing areas of Mississippi. Most of the herbicides used on cotton and rice do not have enforceable drinking water standards, and in most cases the acute toxicity of these herbicides to aquatic fauna is low. However, there have been no studies that examined the long-term effects on an ecosystem of chronic exposure to low levels of multiple herbicides.

Acknowledgments

The use of brand names in this paper is for identification purposes only and does not imply endorsement by the U.S. Geological Survey.

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