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# Sorption–desorption of cyanazine in three Mississippi delta soils

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In recent years, public concern about the presence of pesticides in surface water has increased. Cyanazine has become of particular interest in Mississippi because of its widespread use and detection in the Mississippi River and its tributaries in the Yazoo River basin of Mississippi (Coupe et al. 1998; Pereira and Hostettler 1993; Pereira and Rostad 1990). Additionally, cyanazine has been detected in lakes, rivers, and streams of other Southern and Midwestern states (Senseman et al. 1997; Thurman et al. 1992). Cyanazine levels in surface water runoff typically range from 1.3 to 5.5 (g L<sup>-1</sup>) (Hansen et al. 2000; Verstraeten et al. 1999). Field losses of cyanazine occur typically through surface water runoff within 3 wk after application (Clausen et al. 1996).

Surface runoff is the primary source of pesticide in surface waters (Leonard 1990). Pesticides are transported in runoff in one of two ways, either adsorbed to suspended sediments or in solution. Pesticide sorption contributes to runoff losses in two ways: (1) strongly adsorbed pesticides are lost in the solid phase on entrained sediments, and (2) desorption contributes pesticides to the dissolved phase (Leonard 1990).

Cyanazine is used to control annual grass and broadleaf weeds in cotton (*Gossypium hirsutum* L.) and corn (*Zea mays* L.). Cyanazine has a water solubility of 170 mg L<sup>-1</sup> and pK<sub>a</sub> of 0.63 (Grayson 1986). Cyanazine, a member of the *s*-triazine herbicide family, is a weak base and readily adsorbs to soil (Cancela et al. 1990; Clay et al. 1988; Majka and Lavy 1977; Weber 1970). The triazines are easily protonated at pH levels below a herbicide's pK<sub>a</sub>. However, soil pH levels rarely fall below the pK<sub>a</sub> for cyanazine. Soil pH levels are often two or more units higher than the pK<sub>a</sub> of cyanazine; thus, cyanazine is not protonated, and other mechanisms such as hydrogen bonding and hydrophobic interactions become more important (Bouchard and Lavy 1985; Hance

Sorption and desorption of cyanazine with three Mississippi Delta soils (two silt loams and one silty clay) were studied under laboratory conditions. Cyanazine sorption calculated using the Freundlich equation was greatest for the Sharkey silty clay soil. Partition coefficients ( $K_d$  values) for cyanazine sorption ranged from 1.67 to 1.82, 1.92 to 2.15, and 3.65 to 3.96 ml g<sup>-1</sup> for the Bosket silt loam, Dubbs silt loam, and Sharkey silty clay soils, respectively. Differences in sorption and  $K_d$  values were attributed to clay content. At a given initial cyanazine concentration, cyanazine was desorbed more readily from the silt loam soils than from the Sharkey clay after the first 4-h desorption cycle. Desorption from the Sharkey clay continued for a longer period than that from the silt loam soils, with up to 6% cyanazine desorption from the Sharkey clay after a 16-h desorption cycle compared with 0% for the silt loam soils. Cyanazine losses increased with decreasing clay content, Dubbs = Bosket > Sharkey. This implies a potential relationship between cyanazine desorption and surface runoff losses of cyanazine.

**Nomenclature:** Cyanazine.

**Key words:** Pesticides, cation exchange capacity, high-performance liquid chromatography, sorption isotherm.

1969; Hayes 1970). Cyanazine sorption to soil is typically facilitated by ionic binding to negatively charged colloids (Koskinen and Harper 1990). Cation exchange also has been demonstrated as a method of triazine sorption to soil (Carringer et al. 1975; Senesi and Testini 1980). Cyanazine sorption has been shown to increase with decreasing pH (Weber 1970). Enhanced protonation at the sorption surface has been demonstrated for *s*-triazines at pH values above the pK<sub>a</sub> (Bailey et al. 1968). Additionally, soil type and properties such as texture and organic carbon dramatically influence cyanazine sorption to soil (Reddy et al. 1997).

In the United States, manufacture of cyanazine ceased in 1999, and its use was not allowed after September 2002. However, cyanazine continues to be manufactured and labeled for use in various cereal crops, corn, cotton, soybean [*Glycine max* (L.) Merr], potatoes (*Solanum tuberosum* L.), and forests outside the United States (Sonanda 2002). Thus, a better understanding of cyanazine sorption–desorption mechanisms and of how these processes are influenced by soil type and characteristic properties would be beneficial when evaluating dissipation mechanisms of cyanazine used outside the United States. Additionally, modeling sorption–desorption processes in different soils for an extensively used herbicide such as cyanazine may provide useful information on sorption–desorption processes of other triazine herbicides with similar physicochemical properties. Therefore, the objective of this study was to determine the effects of three Mississippi Delta soils, prone to surface runoff (Southwick et al. 1997), on cyanazine sorption–desorption processes.

## Materials and Methods

Soil samples taken from 0- to 25-cm depth were collected from fields at the Delta Research and Extension Center,

TABLE 1. Physical and chemical properties of three Mississippi Delta soils.<sup>a</sup>

Soil <sup>b</sup>	Texture <sup>c</sup>	Clay	Silt	Sand	Organic matter	pH	CEC <sup>d</sup>	Soil water content <sup>e</sup>
		%						cmol kg <sup>-1</sup>
Bosket	SL	13.8	50.0	36.2	0.7	6.8	15.5	4.1
Dubbs	SL	22.5	52.8	24.7	1.8	6.6	22.5	4.4
Sharkey	SC	52.5	43.8	3.7	1.6	7.4	39.4	11.3

<sup>a</sup> Soil properties determined by Mississippi Cooperative Extension Service, Soil Testing Lab, Mississippi State University, Mississippi State, MS.

<sup>b</sup> As mapped by the USDA SCS (1961).

<sup>c</sup> Abbreviations: SL, silt loam; SC, silty clay.

<sup>d</sup> Abbreviation: CEC, cation exchange capacity.

<sup>e</sup> Weight basis for oven-dried soil.

Stoneville, MS. Samples were collected from noncrop areas that had not been treated with cyanazine in recent years. Soils were air-dried, passed through a 2-mm sieve, and stored at room temperature until use. Moisture content was determined for each soil (Table 1). The soils included a Bosket silt loam (fine-loamy, mixed, thermic Mollic Hapludalf), a Dubbs silt loam (fine-silty, mixed, thermic Typic Hapludalf), and a Sharkey silty clay (very fine, smectitic, thermic Epiaquert). Table 1 lists the physical and chemical properties of each soil.

Cyanazine solutions were formulated by dissolving technical grade cyanazine (99% purity)<sup>1</sup> in methanol (ACS grade, 99.9%).<sup>2</sup> The cyanazine-methanol solutions were diluted with 0.01 M CaCl<sub>2</sub> to create 5% methanol, 0.01 M CaCl<sub>2</sub> solutions containing 15.3, 20.4, 25.5, and 30.5 μM cyanazine. These solution concentrations encompass the cyanazine<sup>3</sup> use rate range for the Bosket, Dubbs, and Sharkey soils after adjustment for soil texture and organic matter content when uniformly incorporated to 1.3 cm. Blank controls also were equilibrated with each soil type so that background levels of cyanazine could be confirmed. Deionized water was used for all solutions. Soil, 5 g (dry-weight basis), was added to 50-ml centrifuge tubes.<sup>4</sup> Cyanazine solutions were combined with each soil type (3:1) and equilibrated on a horizontal shaker (140 cycles min<sup>-1</sup>) for 24 h at room temperature (27 C). Each cyanazine concentration and soil combination was replicated four times. Preliminary studies indicated that cyanazine sorption was completed within 24 h. After equilibration, samples were centrifuged (4,700 × g for 20 min). The supernatant was removed and stored at 4 C until analysis. Desorption was determined immediately after sorption using the same samples. Herbicide-free 0.01 M CaCl<sub>2</sub> solution was added again to the soil pellet based on weight (3:1). The pellet was resuspended by vortexing. The samples were equilibrated for 4 h and centrifuged as described above. The supernatant was removed and stored until analysis. Four sequential 4-h desorption cycles were conducted. After four desorption cycles, desorption had ceased in all but three Sharkey clay treatments. After four desorption cycles, cyanazine residues in the samples were exposed to laboratory conditions for 2.4 d. Smith and Walker (1989) reported that under similar conditions the half-life of cyanazine was 2.6 d. Results from further desorption cycles may have been adversely affected by degradation.

Sorption and desorption were measured by determining the cyanazine content of the supernatant before and after equilibration. Before analysis, sample aliquots were filtered using 0.2-μm syringe filters.<sup>5</sup> Cyanazine was determined using high-performance liquid chromatography (HPLC). A

10-μL injection volume was used for all samples. For HPLC separation a silica C-18 reverse-phase column<sup>6</sup> (125 × 4 mm inside diameter) and an isocratic mobile phase of water-acetonitrile (70:30 v/v) at a flow rate of 1 ml min<sup>-1</sup> were used. Sorption was measured at 225 nm. Peak retention time was 4.9 min. The lower detection limit was 0.34 g ml<sup>-1</sup>. Background levels of cyanazine for each soil type were below detection limits (data not shown).

Cyanazine sorption was determined by subtracting the amount of cyanazine in the supernatant from that originally applied. Cyanazine sorption was regressed against equilibrium solution concentration for each soil. Comparisons of regression parameters were made at the 5% level of significance to determine the effects of soil type. Pearson's correlation was used to evaluate the relationships of  $K_d$  values and cyanazine sorption as the percentage of cyanazine applied with clay and organic matter contents, pH, and cation exchange capacity. Mean cyanazine sorption,  $K_d$  values, and percent-desorbed cyanazine were separated using Fisher's protected LSD ( $P < 0.05$ ).

## Results and Discussion

### Cyanazine Sorption

Cyanazine sorption increased as cyanazine concentration increased for all three soils within the concentration range evaluated (Figure 1). Adjusted  $r^2$  values were 0.98 for all soils, indicating a strong linear relationship between cyanazine sorption and cyanazine equilibrium concentration. Slope and intercept comparisons (Table 2) indicate that the order of sorption by soil type was Sharkey > Dubbs > Bosket. The order of increasing sorption follows the order of increasing clay content and CEC for the soils evaluated (Table 1). Cyanazine sorption as a percentage of cyanazine applied was directly correlated with clay content, pH, and CEC (Table 3). These results are similar to those reported for atrazine, where clay content, pH, and organic carbon content played a role in sorption (Novak et al. 1997). Partition coefficient ( $K_d$ ) values for cyanazine sorption ranged from 1.7 to 1.8, 1.9 to 2.2, and 3.7 to 4.0 ml g<sup>-1</sup> for the Bosket, Dubbs, and Sharkey soils, respectively (Table 4).  $K_d$  values also were positively correlated with clay content, pH, and CEC (Table 3).  $K_d$  values for the two silt loam soils were similar to those reported in the literature (Reddy et al. 1997). The  $K_d$  values reported for Sharkey clay were similar to those for atrazine sorption on Sharkey clay (Ma et al. 1993). Overall, the higher the clay content, the greater was the soil's adsorptive capacity.

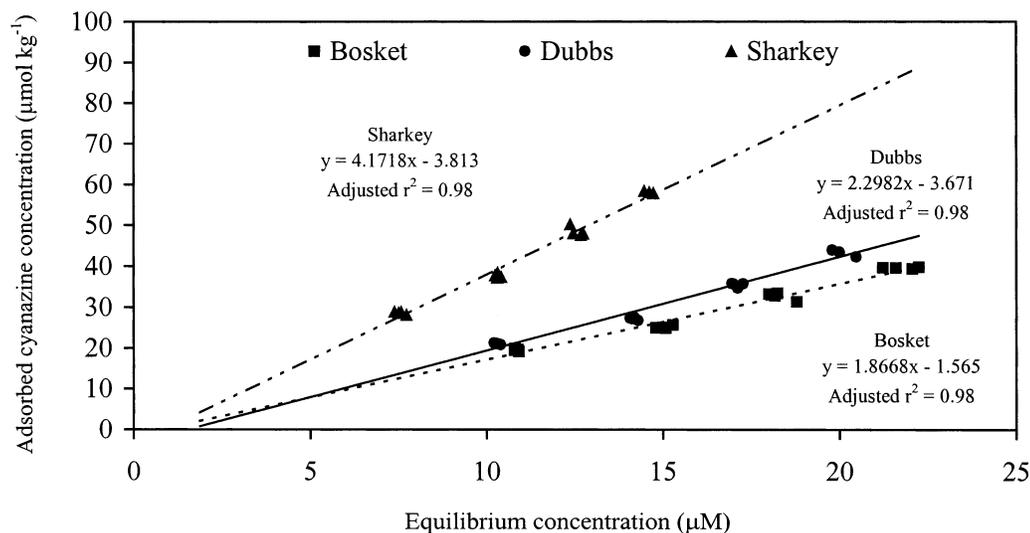


FIGURE 1. Cyanazine sorption isotherms for three Mississippi Delta soils.

### Desorption of Cyanazine

At the conclusion of the first desorption cycle, the percentage of desorbed cyanazine was greater in the two silt loam soils than in the Sharkey clay (Table 4). Desorption at this time increased with decreasing clay content. At the completion of the second desorption cycle (Table 4), there were no differences due to treatment. At the completion of the third desorption cycle, cyanazine desorption was greatest from the Sharkey clay (Table 4). Desorption was greater for the 25.5 and 30.5 M concentrations of the Dubbs soil than for other concentrations and for all concentrations of the Bosket soil (Table 4). Desorption from the Bosket silt loam and from the two lowest initial concentrations of the Dubbs silt loam was complete after two desorption cycles. After four desorption cycles, desorption from the Sharkey clay with initial concentrations of 20.4, 25.5, and 30.5 M was greater than that for the 15.3 M concentration and for all concentrations from the silt loam soils (Table 4). Cyanazine desorption from the silt loam soils did not occur during the fourth desorption cycle. Overall, cyanazine was desorbed more readily from the silt loam soils than from the Sharkey clay, with 36.5 to 45% cyanazine desorbed from the silt loam soil after the first desorption cycle compared with 21.8 to 27.5% from the Sharkey clay (Table 4). Desorption from the Sharkey clay continued longer than that from the silt loam soils, with 0% cyanazine desorbed from the silt loam soils after the fourth desorption cycle compared with 0 to 6% from the Sharkey clay. In general, total desorption did not differ between soil types (Table 4). There were some

TABLE 2. Summary of the effect of soil type on regression parameters for cyanazine sorption.

Soil <sup>a</sup>	Regression parameters		
	Adjusted $r^2$	Intercept <sup>b</sup>	Slope <sup>b</sup>
Bosket	0.98	- 1.57 a	1.87 a
Dubbs	0.98	- 3.67 a	2.30 b
Sharkey	0.98	- 3.81 a	4.17 c

<sup>a</sup> As mapped by the USDA SCS (1961).

<sup>b</sup> Means within the same column followed by the same letter are not significantly different at the 0.05 significance level.

concentration-specific differences. Total cyanazine desorption ranged from 47 to 62%. This was slightly less than that reported by Reddy et al. (1997). However, for a similar soil, they reported a slightly lower  $K_d$ . In this study, the percent desorption from the first desorption cycle was negatively correlated with  $K_d$  values (data not shown). Thus, for larger amounts of adsorbed cyanazine, a smaller percentage will desorb.

The total percentage of applied cyanazine that was recovered ranged from 67% with the 15.3  $\mu\text{M}$  cyanazine concentration applied to Sharkey soil to 84.2% with the 20.4  $\mu\text{M}$  concentration applied to the Bosket silt loam (Table 4). There are two possible reasons for cyanazine not having been recovered: (1) cyanazine sorption to centrifuge tubes and (2) degradation of cyanazine during sorption and desorption. Approximately 7% of cyanazine in equilibration solutions was adsorbed to the walls of centrifuge tubes (data not shown). Nonenzymatic degradation of cyanazine also may have attributed to the inability to account for all the applied cyanazine. Reddy et al. (1997) found that cyanazine can be degraded to desmethylpropanenitrile cyanazine, hydroxyacid cyanazine, desethyl cyanazine, cyanazine amide, and chloroacid cyanazine by hydrolytic and dealkylation reactions. In addition, they also found that these degraded products of cyanazine are less likely to adsorb to soil and more likely to remain in the aqueous phase than cyanazine. Thus, degraded products of cyanazine may have been re-

TABLE 3. Pearson correlation coefficients for the relationships of cyanazine  $K_d$  values and adsorbed cyanazine with clay and organic matter contents (%), pH, and CEC.

	Pearson correlation coefficients <sup>a</sup>			
	Clay	Organic matter	pH	CEC <sup>b</sup>
$K_d^c$	0.99	0.46	0.93	0.98
Adsorbed cyanazine <sup>d</sup>	0.99	0.49	0.91	0.99

<sup>a</sup> All coefficients are significant at the 5% level.

<sup>b</sup> Abbreviation: CEC, cation exchange capacity.

<sup>c</sup>  $K_d$ , amount adsorbed divided by final equilibrium concentration.

<sup>d</sup> Represented as percentage of applied cyanazine.

TABLE 4. Effect of three Mississippi Delta soils on cyanazine sorption and desorption.

Soil <sup>b</sup>	Initial cyanazine concentration μM	Adsorbed cyanazine		$K_d^c$ ml g <sup>-1</sup>	Desorbed cyanazine by equilibration cycle <sup>a</sup>					Total cyanazine recovered <sup>e</sup>
		μmol kg <sup>-1</sup>	% <sup>f</sup>		I (4 h)	II (8 h)	III (12 h)	IV (16 h)	Total <sup>d</sup>	
Bosket	15.3	19.5	42.5	1.80	41.8	17.8	0.00	0.00	59.6	82.8
	20.4	25.1	41.0	1.67	45.0	16.5	0.00	0.00	61.5	84.2
	25.5	32.7	42.7	1.77	42.8	17.3	0.00	0.00	60.1	83.0
	30.5	39.6	43.3	1.82	40.4	16.5	0.00	0.00	56.9	81.3
Dubbs	15.3	21.0	45.8	2.05	37.5	15.5	0.00	0.00	53.0	78.5
	20.4	27.2	44.4	1.92	39.8	17.3	0.00	0.00	57.1	80.9
	25.5	35.4	46.3	2.07	36.5	17.8	8.00	0.00	62.3	82.5
	30.5	43.2	47.2	2.15	36.5	17.0	6.75	0.00	60.3	81.3
Sharkey	15.3	28.6	62.3	3.80	21.8	15.3	9.50	0.00	46.6	66.7
	20.4	37.7	61.6	3.65	27.5	18.8	10.25	0.00	58.1	74.2
	25.5	48.4	62.4	3.85	26.8	17.5	9.75	6.00	60.1	74.5
	30.5	58.0	62.4	3.96	26.5	17.3	10.50	6.00	60.3	74.8
LSD <sup>g</sup>	0.8			0.10	7.2	NS <sup>h</sup>	0.90	1.29	7.9	

<sup>a</sup> Represented as percentage of the amount adsorbed in Column 3 for each 4-h desorption cycle.

<sup>b</sup> As mapped by the USDA SCS (1961).

<sup>c</sup>  $K_d$ , amount sorbed divided by final equilibrium concentration.

<sup>d</sup> Summation of the four desorption cycles.

<sup>e</sup> Percentage of total applied cyanazine that was recovered after sorption and desorption cycles.

<sup>f</sup> Percentage of total applied cyanazine adsorbed after the 24-h equilibration cycle.

<sup>g</sup> Means were separated using Fisher's protected LSD at the 0.05 significance level.

<sup>h</sup> Abbreviation: NS, not significant.

moved in the supernatant solution extracted during sorption and desorption.

In this study, clay content as it relates to CEC was the major factor influencing cyanazine sorption. Cyanazine desorption occurred more readily from the silt loam soils than from the Sharkey clay. These data support those of Reddy et al. (1997), who found that desorption of cyanazine was greater from silt loam soils than from clay soils. These results indicate that increasing the cyanazine rates to account for sorption on organic matter may not be justified. Further research on soils with greater variation in chemical and physical characteristics is needed before such recommendations can be applied to all situations.

### Sources of Materials

<sup>1</sup> Technical grade cyanazine, CHEM SERVICE, Box 3108, West Chester, PA 19381.

<sup>2</sup> Methanol, Burdick & Jackson, 1953 S, Harvey Street, Muskegon, MI 49442.

<sup>3</sup> Cyanazine, Cy-Pro® 4L label, Griffin Corporation, Valdosta, GA.

<sup>4</sup> Corning Brand 50-ml graduated plastic tubes with plug seal (05-538-55), Fisher Scientific Company, P.O. Box 869022, Plano, TX 75086-9022.

<sup>5</sup> Nylon syringe filters (2387), Alltech Associates Inc., 2051 Waukegan Road, Deerfield, IL 60015-1899.

<sup>6</sup> Hewlett Packard Hypersil ODS 5 m column (7982618-564), Hewlett-Packard Co., Little Falls Site 4300, 2850 Centerville Road, Wilmington, DE 19808.

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## Erratum

*Weed Science* regrets the inclusion of the erroneous species name in the title of an article recently published. The citation should be as follows:

- Dean S. Volenberg and David E. Stoltenberg. 2002. Inheritance of resistance in eastern black nightshade (*Solanum ptycanthum*) to acetolactate synthase inhibitors. *Weed Science* 50:731–736.