

Adsorption and Desorption of Atrazine, Deethylatrazine, Deisopropylatrazine, and Hydroxyatrazine on Levy Wetland Soil

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The adsorption and desorption of atrazine, and its three metabolites, deethylatrazine (DEA), deisopropylatrazine (DIA), and hydroxyatrazine (HA), on Levy wetland soil were measured using the batch equilibration technique. Solution concentrations for each chemical were 1.3, 3.8, 6.6, and 13.2 $\mu\text{mol L}^{-1}$. Four 24-h period desorptions for each concentration were conducted immediately after adsorption. The adsorption of all the chemicals conformed to linear isotherms. Adsorption coefficients decreased in the order HA (109.7) > atrazine (38.6) > DIA (26.3) > DEA (22.1). The organic carbon partition coefficients (K_{oc}) for HA, atrazine, DIA, and DEA were 1500, 440, 290, and 240 L kg^{-1} , respectively. Percent of chemical desorbed was about 29% for DEA, 24% for atrazine, 23% for DIA, and 16% for HA. The slopes of all desorption isotherms were less than their respective adsorption slopes, indicating hysteresis. Atrazine and its metabolites were relatively more strongly adsorbed and the binding was less reversible in the Levy wetland soil than what has been reported for agricultural soils.

Keywords: *Atrazine; deethylatrazine; hydroxyatrazine*

INTRODUCTION

Atrazine [2-chloro-4-(ethylamino)-6-(isopropylamino)-*s*-triazine] is a herbicide widely used to control annual grasses and broadleaf weeds primarily in corn and sorghum (Weed Science Society of America, 1989). A fraction of the atrazine applied to agricultural fields and of the metabolites formed is transported to rivers and other surface waters by runoff (Goolsby et al., 1994; Spalding et al., 1994). Field-monitoring studies of surface waters have shown the detection of atrazine and its dealkylated metabolites deethylatrazine [2-amino-4-chloro-6-(isopropylamino)-*s*-triazine; DEA] and deisopropylatrazine [2-amino-4-chloro-6-(ethylamino)-*s*-triazine; DIA] (Thurman et al., 1991, 1992, 1994; Goolsby et al., 1994). Atrazine is detected in rivers and streams primarily because of its widespread use and moderate persistence. Its presence in surface water is a concern because of the potential to exceed the drinking water standard, which has been set at 3.0 $\mu\text{g L}^{-1}$ (maximum contaminant level; MCL). MCLs have not been established for DEA or DIA, but both metabolites are phytoxic (Kaufman and Kearney, 1970).

Atrazine metabolites form via biotic and abiotic processes (Erickson and Lee, 1989). Microbial degradation of atrazine results in N-dealkylation to form primarily DEA and DIA (Behki and Khan, 1986, 1994). The metabolite hydroxyatrazine [2-hydroxy-4-(ethylamino)-6-(isopropylamino)-*s*-triazine; HA] is formed by chemical hydrolysis, and the rate increases as soil pH decreases and soil organic carbon increases (Armstrong et al., 1967; Skipper et al., 1978). The major dealkylated metabolite of atrazine is DEA (Adams and Thurman, 1991). The predominant metabolite at depths greater than 10 cm, DEA, accounted for 26% of the ^{14}C in the 10–20-cm depth, whereas DIA accounted for less than 10% of the ^{14}C recovered at any depth (Sorenson et al., 1994). The major degradation product of atrazine in the top 10 cm of soil, HA, accounted for 9% of the ^{14}C present. The predominance of DEA compared to DIA may be due to the faster (2–3 times) removal of the

ethyl side chain from atrazine than of the isopropyl side chain by microorganisms (Mills and Thurman, 1994).

Sorption is the major process that controls the degradation (both biotic and abiotic) and mobility of a herbicide in soil (Koskinen and Harper, 1990). If a herbicide is irreversibly bound to a soil or if its desorption is very slow, its mobility or its release back to solution is negligible. Although adsorption and desorption of atrazine by agricultural soils have been studied extensively (Talbert and Fletchall, 1965; Clay et al., 1988; Clay and Koskinen, 1990a; Pignatello and Huang, 1991), little information exists on its sorption by wetland soils. There are even fewer studies on the adsorption/desorption of its metabolites in both agricultural and wetland soils. On agricultural soils, DEA is less adsorbed than atrazine and HA is more strongly bound to the soil matrix than atrazine DEA or DIA (Brouwer et al., 1990; Clay and Koskinen, 1990b; Roy and Krapac, 1994).

Because wetlands occupy depressions or low-lying areas in the landscape, surface runoff from agricultural fields is deposited or passes through wetland soils before it reaches other surface water bodies or groundwater under some conditions. Thus, wetlands, recipients of agricultural chemicals transported by runoff from non-point agricultural sources, are perceived as filters in the hydrologic cycle (Gilliam, 1994). Understanding of sorption is essential to predict the persistence and transport of herbicides through a wetland soil.

Previous studies on adsorption and desorption of atrazine or its metabolites from agricultural soils may not describe the fate of these chemicals in wetland soils. Wetland soils are different from agricultural soils in many ways. Wetland soils tend to have high organic matter contents, and a narrow pH range (6.5–7.5), while agricultural soils tend to have low organic matter content (0.1–3%), a greater pH range (5–7.5), and a wide range of textures (from sandy to heavy clay material). Wetland soils are also saturated with water

during some or all parts of the year. These differences affect the relative sorption characteristics of a herbicide in soil.

The objective of this study was to characterize adsorption and desorption of the herbicide atrazine and its metabolites, DEA, DIA, and HA on a tidal wetland soil.

MATERIALS AND METHODS

Soil. The soil used for this study was Levy silt loam (coarse-loamy, mixed, nonacid, thermic Typic Hydraquents), from the Kenon marsh along the James River in Prince George county, Virginia. The Levy soil is a tidal wetland soil, which remains saturated throughout the year and is inundated twice daily by tides. The soil was sampled from the top 0–10-cm depth and contained 57% silt, 37% clay, and 6% sand. The organic carbon content was 9% and soil pH was 6.3 (1:1 soil:water). Soil sample was wet-sieved through a 2-mm sieve to remove roots and was used within 24 h of collection.

Chemicals. Solutions (0.01 M CaCl₂) were prepared by mixing ¹⁴C-ring-labeled and technical grade chemicals to obtain 1.3, 3.8, 6.6, or 13.6 μmol L⁻¹ of atrazine, DEA, DIA, or HA. The atrazine solution contained 0.45 kBq mL⁻¹ of labeled atrazine with a specific activity of 155 MBq mmol⁻¹ and a radiochemical purity of 98.5%. The specific activity of DEA was 166 MBq mmol⁻¹ (99.1% radiochemical purity), and the solution contained 0.33 kBq mmol⁻¹. The DIA solution contained 0.26 kBq mL⁻¹ of labeled DIA with a specific activity of 128 MBq mmol⁻¹ and a radiochemical purity of 96.1%. The specific activity of HA was 322 MBq mmol⁻¹ (98.6% radiochemical purity), and the solution contained 0.39 kBq mL⁻¹. The purities of the technical grade atrazine, DEA, DIA, and HA were 98, 99, 98, and 97%, respectively.

Adsorption. Adsorption isotherms of atrazine, DEA, DIA, and HA on Levy soil were determined by using the batch equilibration technique. Wetland soil conditions were maintained by using fresh samples without drying and a relatively large water/soil ratio (10:1). A 5-mL aliquot of each chemical solution was added to 1 g of soil (oven-dry equivalent) in a 50-mL Teflon centrifuge tube to obtain a final 1:10 soil/solution ratio. Each concentration was replicated three times for each chemical. The initial weight of each tube with slurries but without the cap was recorded. The slurries were shaken on a reciprocal shaker at 250 rpm for 24 h and then centrifuged at 2000g (3000 rpm) for 30 min at 24 °C. The volume of the supernatant solution was then measured. A 0.5 mL of solution was taken from each tube, and the ¹⁴C content was analyzed by liquid scintillation spectrometry (LSS). The supernatant was then decanted (6.3 mL). The amount of chemical adsorbed after equilibration was calculated as the difference between the supernatant concentration and the amount of chemical initially added.

Desorption. Desorption studies were conducted immediately after the 24-h adsorption at each concentration of the four chemicals. The decanted 6.3 mL of the adsorption supernatant was replaced with an equal volume of fresh 0.01 M CaCl₂. The exact volume was evaluated by weighing. The tubes were vibrated to disperse soil pellet and placed on a reciprocal shaker at 250 rpm for 24 h. Tubes were then centrifuged for 30 min at 2000g (2000 rpm) and 24 °C. A 0.5-mL aliquot of the supernatant solution was analyzed for ¹⁴C using LSS. The amount of chemical that remained adsorbed to the soil was calculated by difference between the supernatant solution concentration and that which remained initially on soil. The process was repeated for a total of four desorptions with 24-h equilibration.

Adsorption and desorption isotherms were calculated using the linearized form of the Freundlich equation:

$$\log (x/m) = \log K_f + (1/n) \log C$$

where x/m is micromoles of atrazine or metabolite adsorbed per kilogram of soil, C is micromoles of atrazine or metabolite

per liter of supernatant after equilibration, and K_f and $1/n$ are empirical constants.

Statistical Analyses. Statistical evaluation included regression analysis, comparison of slopes, and calculation of the 95% confidence intervals for the intercept ($\log K_f$) and standard error of the slope ($1/n$).

RESULTS AND DISCUSSION

Adsorption. The linearized form of the Freundlich equation has reasonably described the adsorption of atrazine ($r^2 = 0.9$), DEA ($r^2 = 0.92$), DIA ($r^2 = 0.85$), and HA ($r^2 = 0.85$) by Levy soil (Figure 1). Adsorption coefficients (K_f) decreased in the order HA > atrazine > DIA > DEA (Table 1). This order was also reported by Brouwer et al. (1990) for four different soil types. The K_f values indicated that HA was more strongly adsorbed than atrazine or DEA or DIA on the Levy soil. Greater adsorption of HA ($K_f = 25 \mu\text{mol L}^{-1} \text{kg}^{-1}$) compared to atrazine ($K_f = 3.7 \mu\text{mol L}^{-1} \text{kg}^{-1}$) averaged for Plano and Waukegan soils was also reported by Clay and Koskinen (1990b). The large K_f values for HA (109.7 μmol L⁻¹ kg⁻¹) and atrazine (38.6 μmol L⁻¹ kg⁻¹) in this study compared to those for Plano and Waukegan soils indicate the extensive sorption capacity of this wetland soil.

The average slopes ($1/n_{ad}$) for adsorption of atrazine, DEA, DIA, and HA were 0.90, 0.92, 0.85, and 0.85, respectively (Table 1). These slopes, which are <1, indicate that the percentage of these chemicals adsorbed to the Levy soil decreased as the initial concentration increased.

The adsorption distribution coefficient (K_d), defined as $(x/m)/C$, where x is the amount of chemical adsorbed, m is the amount of adsorbent, and C is the chemical concentration at equilibrium, and organic carbon partition coefficient (K_{oc}), defined as K_d/f_{oc} , where f_{oc} is the organic carbon fraction of the soil, are given in Table 1. The K_{oc} values followed the order HA (1500 L kg⁻¹), > atrazine (440 L kg⁻¹), > DIA (290 L kg⁻¹), > DEA (240 L kg⁻¹). The K_{oc} values for atrazine in agricultural soils have been reported as 109 L kg⁻¹ (Dousset et al., 1994) and as 100 L kg⁻¹ (Wauchope et al., 1992). On a soil with 9.23% of organic carbon, Donati et al. (1994) obtained K_{oc} values of 401, 141, and 155 L kg⁻¹ for atrazine, DEA, and DIA, respectively. These figures are similar to K_{oc} values obtained mainly because of similar organic carbon content. With a low organic soil (0.18%), Roy and Krapac (1994) obtained K_{oc} values of 112 and 72 L kg⁻¹ for atrazine and DEA, respectively.

The K_{oc} values are widely used to predict herbicide adsorption because, for many nonionic compounds including herbicides, organic carbon is the main soil component responsible for adsorption (Shea, 1989). The Levy soil has relatively high organic carbon content (9%), and the chemicals would mostly be adsorbed to this fraction.

The K_{oc} is negatively correlated with aqueous solubilities of chemicals (Shea, 1989). The order of sorption of the chemicals on the Levy soil follows the order of decreasing aqueous solubility, which is DEA, DIA, atrazine, and HA (2.0, 1.2, 0.50, and 0.24 mM L⁻¹, respectively) (Erickson and Lee, 1989). HA, the least water soluble, was the most strongly adsorbed, atrazine was intermediate, and DIA and DEA were the most weakly adsorbed.

Desorption. Desorption isotherms related the amount of the chemical retained by the soil to chemical

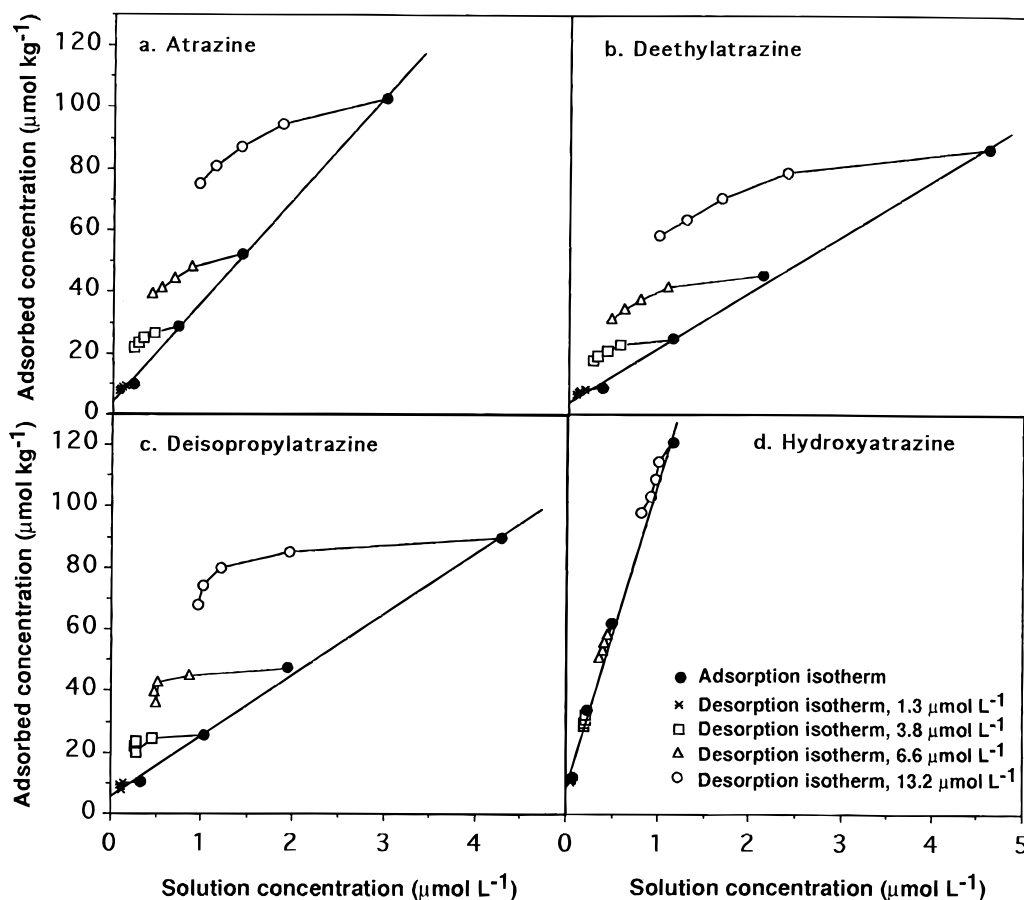


Figure 1. Adsorption and desorption isotherms for (a) atrazine, (b) DEA, (c) DIA, and (d) HA on Levy wetland soil.

Table 1. Adsorption Coefficients (K_f), Slope ($1/n$), and Organic Carbon Partition Coefficients (K_{oc}) of Atrazine, DEA, DIA, and HA on Levy Wetland Soil

chemical	K_f^a ($\mu\text{mol}^{1-1/n} \text{L}^{1/n} \text{kg}^{-1}$)	$1/n^b$	r^2	K_{oc} (L kg^{-1})
atrazine	38.6 (36.7–40.5)	0.90 (0.007)	0.85	440
DEA	22.1 (20.6–23.6)	0.92 (0.01)	0.92	240
DIA	26.3 (23.5–29.1)	0.85 (0.01)	0.85	290
HA	109.7 (103.3–115.9)	0.85 (0.01)	0.85	1500

^a Numbers in parentheses are the 95% confidence intervals (CI). For K_f , the CIs are the antilogs of $\log K_f - \text{CI} \log K_f$ and $\log K_f + \text{CI} \log K_f$. ^b Number in parentheses is standard error of the mean.

concentration in the solution at equilibrium (Figure 1). As the initial concentration increased, the K_{fdes} values increased (Table 2). Larger K_{fdes} values indicate a greater proportion of the chemical is retained by the soil. The K_{fdes} values for HA were greater than for the other chemicals. This indicates a larger proportion of non-desorbable HA on Levy soil than of atrazine or its metabolites.

The desorption isotherms for all the chemicals are nonlinear (Figure 1). The $1/n_{des}$ values for the more concentrated solutions are higher than the dilute ones, indicating a greater desorption rate. This was evident in all of the chemicals except HA. The Freundlich $1/n_{des}$ value takes into account the nonlinearity of the isotherms and merely estimates the desorption intensity or rate (Pignatello and Huang, 1991). The amount or the proportion of the nondesorbed portion of the chemical is better depicted by the K_{fdes} values.

Amount desorbed, expressed as percent of initially adsorbed, was lowest for HA followed by DIA, atrazine, and DEA (Figure 2). About 16% of the adsorbed HA was released back to the solution, whereas about 23%

Table 2. Desorption Coefficients (K_f) and Slope ($1/n$) for Atrazine, DEA, DIA, and HA on Levy Wetland Soil

chemical	initial concn ($\mu\text{mol L}^{-1}$)	K_f^a ($\mu\text{mol}^{1-1/n} \text{L}^{1/n} \text{kg}^{-1}$)	$1/n^b$	r^2
atrazine	1.3	13.9 (13.5–14.3)	0.20 (0.008)	0.97
	3.8	31.8 (31.1–32.6)	0.23 (0.005)	0.97
	6.6	48.7 (47.6–49.9)	0.25 (0.006)	0.98
	13.2	78.4 (76.5–80.3)	0.26 (0.009)	0.97
DEA	1.3	10.9 (10.8–11.1)	0.19 (0.003)	0.89
	3.8	24.8 (24.0–25.7)	0.22 (0.003)	0.92
	6.6	39.0 (36.0–42.0)	0.25 (0.006)	0.95
	13.2	60.2 (55.0–65.4)	0.26 (0.015)	0.95
DIA	1.3	11.7 (11.2–12.2)	0.13 (0.003)	0.31
	3.8	26.3 (25.2–27.3)	0.14 (0.006)	0.59
	6.6	43.8 (40.3–47.3)	0.15 (0.009)	0.63
	13.2	73.5 (66.7–80.4)	0.15 (0.007)	0.78
HA	1.3	39.2 (13.1–65.3)	0.45 (0.06)	0.84
	3.8	82.0 (62.2–101.9)	0.61 (0.04)	0.90
	6.6	94.9 (83.7–106.2)	0.62 (0.04)	0.97
	13.2	111.8 (106.9–116.6)	0.55 (0.08)	0.77

^a Numbers in parentheses are the 95% confidence intervals (CI) for K_f and the antilogs $\log K_f - \text{CI} \log K_f$ and $K_f + \text{CI} \log K_f$. ^b Number in parentheses is standard error of the mean.

of DIA, 24% of atrazine, and 29% of DEA were desorbed. About twice the amount of DEA was desorbed compared to that of HA. This relatively lower reversibility of HA means it is the least likely to move within the Levy soil compared to atrazine or the other metabolites.

The slopes of all desorption isotherms were less than their respective $1/n_{ads}$ values, indicating hysteresis. Hysteresis of atrazine (Clay and Koskinen, 1990a; Ma et al., 1993) and DEA (Roy and Krapac, 1994) adsorption has been reported before. Such differences in isotherms

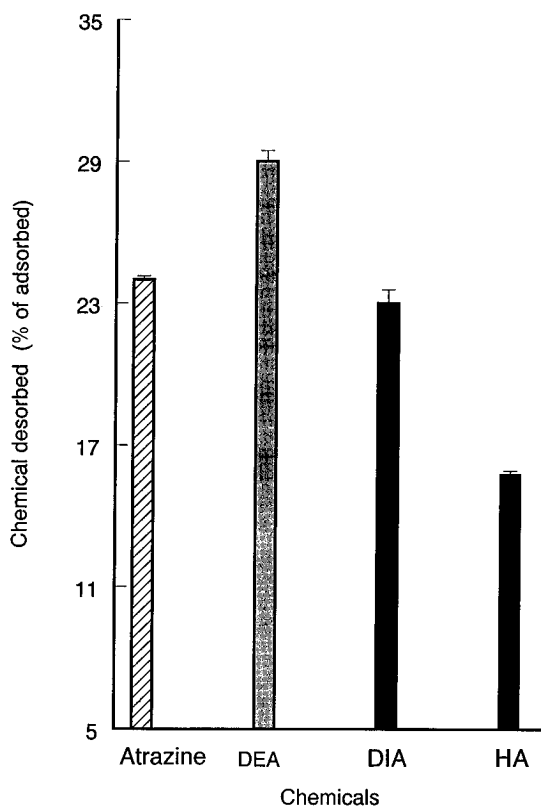


Figure 2. Desorption as percent of initially adsorbed atrazine, DEA, DIA, and HA from Levy wetland soil. Means of three replications \pm standard error.

may be caused by several factors including changes in solution composition, loss of herbicide due to degradation, and irreversible binding of herbicide to soil. The influence of 0.01 M CaCl_2 and other solutions on desorption of atrazine was investigated previously, and their effects were negligible (Clay and Koskinen, 1990a; Clay et al., 1988). Degradation of atrazine in adsorption/desorption solutions after 3 days was also negligible (Clay and Koskinen, 1990a). Roy and Krapac (1994) did not detect DEA or DIA after 3 days in desorption supernatant solution of atrazine. Nonattainment of equilibration could contribute to hysteresis as the rate of desorption is slow. However, the most likely cause for hysteresis in the relatively high organic content Levy soil is irreversible binding. Nonionic compounds including herbicides are irreversibly bound or trapped in the soil matrix (Koskinen and Harper, 1990). Such nondesorbable fraction is reported to increase as the herbicide ages in the soil (Wauchope and Myers, 1985; Pignatello and Huang, 1991). The bound atrazine which is nondesorbable has been characterized as water extractable, water nonextractable but methanol extractable, and methanol nonextractable (Clay and Koskinen, 1990a). The methanol-nonextractable is commonly referred to as soil-bound residue and is the most strongly sorbed.

In summary, HA was more strongly and extensively adsorbed than atrazine, DEA, or DIA. The amount desorbed was greater for DEA (29%), than for atrazine (24%), DIA (23%), and HA (16%). The relatively weaker and greater reversibility of DEA adsorption suggest the potential mobility of this chemical through the wetland soil. Results show that the Levy wetland soil has higher adsorptive and retention capacity for atrazine than what has been reported for agricultural soils.

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