Ammonia Fertilizer Influences Atrazine Adsorption–Desorption Characteristics[†]

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The application of ammonia-based fertilizer (anhydrous ammonia, aqua ammonia, and urea) initially increases soil pH. Changes in soil pH influence dissolved organic carbon (DOC) concentrations in soil solution and herbicide sorption characteristics to soil. A batch study was conducted to evaluate the influence of ammonia application on atrazine [2-chloro-4-(ethylamino)-6-(isopropylamino)-1,3,5-triazine] adsorption and desorption characteristics to a Brandt silty clay loam (fine silty, Pachic Udic Haploborolls) and Ves clay loam (fine silty, mixed mesic typic Hapludalf). Ammonia treatment rates were equivalent to N concentrations found in bands when 0, 100, and 200 kg of N ha^{-1} were injected to soil. Atrazine treatments were 0 (0.01 M CaCl$_2$), 2.08, 6.57, or 50.80 μmol of atrazine L^{-1}. The application of 200 kg of N ha^{-1} increased soil pH from 6 to 9 and increased the DOC concentrations from 60 to 700 ppm in the soil solution. The addition of ammonia decreased atrazine sorption by 50% and doubled atrazine desorption. Ammonia-induced changes in both pH and DOC concentration appeared to influence atrazine sorption characteristics. Ammonia-induced changes in atrazine sorption/desorption characteristics have the potential to increase atrazine movement through soil. These results suggest that atrazine movement may be reduced by physically separating the application of atrazine and ammonia-banded fertilizers.

Keywords: *Ammonia–atrazine interaction; atrazine; sorption characteristics; fertilizer–herbicide interaction*

INTRODUCTION

Atrazine adsorption and desorption characteristics may be influenced by both soil pH and dissolved organic carbon (DOC) content. Soil pH influences the protonation of atrazine and the charge of the adsorbent surface (Koskinen and Harper, 1990; Calvet, 1980). Atrazine, a weakly basic molecule ($pK_a = 1.7$), is adsorbed to a greater extent in low pH than in high pH soils (McGlamery and Slife, 1966; Clay et al., 1988a; Goetz et al., 1988) and has greater desorption from high pH than from low pH soils (Clay and Koskinen, 1990b). DOC has been reported to bind with herbicides in solution (Madhun et al., 1985; Wang et al., 1992) and may influence either adsorption or desorption characteristics or both (Clay et al., 1988b).

Anhydrous ammonia and aqua ammonia are used as N sources for corn and applied in the spring or early summer at approximately the same time as atrazine. These N fertilizers quickly increase soil pH to 9 or greater (Kissel et al., 1988; Norman et al., 1987; Clay et al., 1995). Ammonia-based fertilizers have been reported to increase DOC in solution from 5% to 9-fold under both laboratory and field conditions (Norman et al., 1987; Tomasiewicz and Henry, 1985; Clay et al., 1995).

Applying atrazine over fertilizer slots enhanced atrazine movement through the soil profile (Clay et al., 1994). A portion of the enhanced movement may be due to the macropore created by the injection slot (Clay et al., 1994). However, the chemical changes in soil

Table 1. Selected Physical and Chemical Characteristics for the A Horizon of Brandt Silty Clay Loam and Ves Clay Loam Soil

	soil type	
parameter	Brandt silty clay loam	Ves clay loam
sand, %	17	43
silt, %	56	27
clay, %	27	27
organic carbon,[a] g kg^{-1}	2.4	2.2
pH[b]	5.7	5.6

[a] Organic carbon determined by combustion. [b] pH determined in soil:0.01 M CaCl$_2$ (1:1 v/v).

induced by ammonia fertilizer application also may impact sorption characteristics and impact atrazine fate in soil. The objective of this study was to investigate the influence of ammonia fertilizer application on atrazine adsorption and desorption characteristics.

MATERIALS AND METHODS

Adsorption. Soils used in this study were a Brandt silty clay loam (fine silty, Pachic Udic Haploborolls) and a Ves clay loam (fine silty, mixed mesic typic Hapludalf). The Brandt silty clay loam was collected at Aurora, SD, and the Ves clay loam was collected at Lamberton, MN. Selected soil characteristics are shown in Table 1. The soil was air-dried and passed through a 2-mm sieve.

Ten grams of soil was placed in a centrifuge tube, and 5 mL of fertilizer treatment solution was added at time 0. Aqua ammonia was applied at 0 (0.01 M CaCl$_2$), 1400, and 2800 μg of N g^{-1} of soil. The N concentrations were equivalent to N concentration of injected fertilizer bands applied at 0, 100, and 200 kg of N ha^{-1} in soil. Five milliliters of atrazine solution was added 0 (immediately) or 8 days after the fertilizer was applied. The atrazine concentrations were 0 (control), 2.08, 6.57, or 50.80 μmol of atrazine L^{-1} in 0.01 M CaCl$_2$ solution and spiked with 1.26 kBq of uniformly ring labeled [^{14}C]atrazine.

After atrazine addition, a 1-day batch equilibration study was conducted by (i) mechanically shaking the treated soil slurry for 24 h at 25 °C, (ii) centrifuging the slurry at 8000 rpm (approximately 8000*g*) for 20 min to separate supernatant and soil, and (iii) analyzing the supernatant for pH, DOC, and ^{14}C. Supernatant pH was determined by placing a standardized pH electrode into the solution immediately after separation from the soil. DOC concentration was determined by diluting 1 mL of supernatant with 20 mL of Nanopure water (Barnstead/Thermolyne Corp., Dubuque, IA) (DI water filtered to remove organics) and using a Dohrmann DC-180 Carbon Analyzer (Rosemount Analytical Division, Santa Clara, CA).

The amount of ^{14}C remaining in solution was determined by placing a 1-mL aliquot of supernatant into 6 mL of scintillation cocktail and counting the solution using a Packard 1600 TR liquid scintillation analyzer (Packard Instrument Co., Downers Grove, IL). The amount of atrazine adsorbed was the difference between initial solution and final solution values. The linear form of the Freundlich equation

$$\log[C_s] = \log K_f + 1/n \, \log[C_e] \qquad (1)$$

was used to describe atrazine adsorption. In the Freundlich equation, C_s is micromoles of herbicide adsorbed per kilogram of soil, C_e is micromoles of herbicide per liter of supernatant solution after equilibration, and K_f and $1/n$ are empirical constants.

Desorption. Soil for desorption experiment was prepared by pretreating soil with ammonia fertilizer at the three rates described above for 0 and 8 days, adsorbing 1.26 kBq of uniformly ring labeled [^{14}C]atrazine to soil at 50.80 μmol L^{-1}, and conducting a 24-h batch equilibration with soil–solution separation as described above. After solution was separated from soil, 4 mL of supernatant was removed and analyzed for the amount of ^{14}C remaining in solution. Four milliliters of the initial solution for each treatment was added to soil; the slurry was dispersed and shaken for 24 h, followed by centrifugation to separate soil from solution. This 24-h desorption process was repeated four times (Clay and Koskinen, 1990b). The amount of ^{14}C present in the aqueous solution after each desorption step was determined as previously described. The Freundlich equation (eq 1) was used to describe atrazine desorption. The adsorption and desorption experiments were conducted twice in triplicate for both soils.

DOC–Atrazine Interaction. One hundred grams of each soil was placed in a flask, and 100 mL of NH$_4$OH treatment solution was added to evaluate the potential for DOC–atrazine interactions. The application rates for the NH$_4$OH solutions dissolved in 0.01 M CaCl$_2$ were 0 and 5200 μg of N g^{-1} of soil for Brandt silty clay loam and 0 and 13 000 μg of N g^{-1} of soil for Ves clay loam. The flasks were shaken for 24 h, and two 30-mL aliquots of DOC-containing supernatant were isolated from each treatment by soil type by centrifugation. Atrazine at a concentration of 1.4 μmol L^{-1} was added to each aliquot. One aliquot was spiked with uniformly ring labeled [^{14}C]atrazine (7.8 kBq for Brandt silty clay loam and 0.2 kBq for Ves clay loam), and the other aliquot was spiked with nonradioactive atrazine. The DOC–atrazine solution was allowed to incubate at 23 °C. Solution was removed at intervals ranging from 2 h to 8 days. The ^{14}C-free portion was used to determine the solution pH and DOC content changes during incubation. A 100-μL aliquot of the solution containing [^{14}C]atrazine was spotted on thin layer chromatography plates and allowed to dry, and the plates were developed with 1-butanol/acetic acid/water (11:5:4 v/v/v) solution (Clay and Koskinen, 1990b). Autoradiographs of the TLC plates were obtained by placing X-ray film next to the plate with an exposure period of approximately 3 weeks.

Data Analyses. Freundlich adsorption and desorption isotherm coefficients were calculated by the least-squares technique using the log-transformed equilibrium data. Statistical evaluation included Bartlett's test for homogeneity of variances, comparison of slopes and elevations of the regression lines, and calculation of the 95% confidence intervals for the intercept (log K_f) and standard error of the slope ($1/n$).

Table 2. Influence of Ammonia Application on Soil pH and Solution DOC Content of a Brandt Silty Clay Loam and a Ves Clay Loam after 0 or 8 Days of Incubation

soil type	treatment	soil pH			solution DOC, ppm		
		day 0	day 8	LSD$_{(0.05)}$	day 0	day 8	LSD$_{(0.05)}$
Brandt	control	5.74	5.69	0.03	76	30	2
	medium ammonia	7.81	7.70	0.04	144	207	5
	high ammonia	8.92	8.17	0.62	390	718	8
Ves	control	5.61	5.39	0.14	53	22	1
	medium ammonia	7.60	7.10	0.04	108	155	8
	high ammonia	8.88	7.97	0.19	414	419	ns

RESULTS

Soil Solution pH and DOC Content. Ammonium hydroxide increased soil solution pH by approximately 2 units in the medium and 3 units in the high ammonia treatments, when compared to the 0 N control (Table 2). The solution pH decreased from 0 to 8 days in both soils and all treatments. The pH drop ranged from 0.05 pH unit in the Brandt soil treated with CaCl$_2$ to 0.9 pH unit in the Ves soil treated with the high ammonia rate. Generally, the pH decrease was greater in the Ves than Brandt soil solutions and treatments with the highest initial pH values had the largest pH decrease during the 8-day incubation period. These results are in agreement with those reported by Norman et al. (1987). DOC is made up of organic acids. Therefore, the combination of the effect of organic acids in solution and soil buffering capacity decreased pH.

As the amount of ammonia increased, the amount of DOC in solution increased for both soils when compared to their respective 0.01 M CaCl$_2$ controls (Table 2). After the 8-day incubation, the amount of DOC in the control solution was less than the amount measured at day 0. However, the amount of DOC present in the ammonia treatment solutions generally increased although the pH decreased. These findings suggest that DOC solubilization continued in the ammonia treatments and was pH independent at least over an 8-day time frame.

Atrazine Adsorption and Desorption. Atrazine adsorption isotherms for both soils had $1/n$ values less than 1, indicating that the concentration of atrazine in solution was inversely related to the percentage of soil-adsorbed herbicide (Table 3). The $1/n$ value was consistent between soils, incubation period, and ammonia treatment. K_f values, however, decreased in both soils as ammonia addition increased when soil was not preincubated with ammonia (Table 3; Figures 1 and 2), indicating decreased atrazine adsorption to soil. The decrease in adsorption was most likely due to the increase in pH (Clay et al., 1988a; Yaron et al., 1985).

When the soil was preincubated with 0.01 M CaCl$_2$ for 8 days, atrazine adsorption decreased from 0 to 8 days and matched the decrease observed in both pH and DOC concentration. However, atrazine adsorption was similar in ammonia-treated soil regardless of incubation treatment.

Atrazine desorption increased in ammonia-treated soil as indicated by lower K_f values and higher $1/n$ values when compared to the control (Table 4; Figures 1 and 2). In the medium ammonia treatment, desorption decreased for the Brandt soil but remained constant for the Ves soil from 0 to 8 days. In the high ammonia treatment, atrazine desorption decreased from 0 to 8 days for both soils.

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Table 3. Atrazine Adsorption Isotherms for Brandt Silty Clay Loam and Ves Clay Loam with Ammonia Treatment at 0 and 8 Days of Incubation

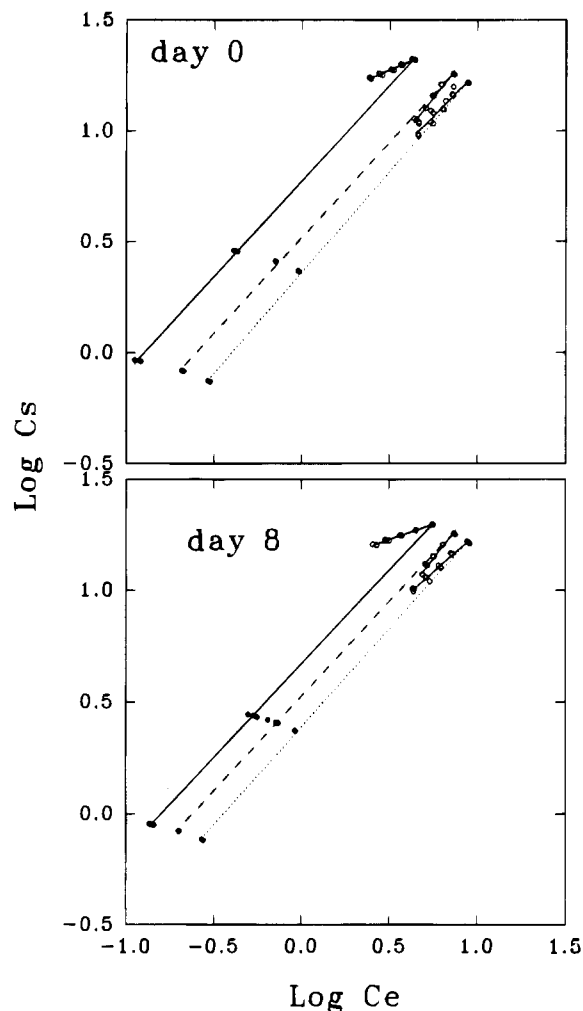| soil type	treatment	day 0		day 8	
		$K_{f,ads}$[a]	$1/n$[b]	$K_{f,ads}$	$1/n$
Brandt	control	5.16 (4.89–5.44)	0.86	3.53 (3.12–3.98)	0.85
	medium ammonia	2.89 (2.43–3.44)	0.86	2.79 (2.56–3.04)	0.85
	high ammonia	2.15 (1.97–2.35)	0.88	2.57 (2.18–3.02)	0.86
Ves	control	5.81 (5.21–6.48)	0.84	4.58 (4.01–5.22)	0.81
	medium ammonia	3.28 (2.98–3.60)	0.85	3.33 (2.97–3.73)	0.83
	high ammonia	2.28 (2.00–2.61)	0.88	2.43 (2.17–2.72)	0.85

[a] Numbers in parentheses are the 95% confidence interval (CI) for K_f; antilogs of log K_f – CI log K_f and log K_f + CI log K_f. [b] The standard error of $1/n$ < 0.01 and $r^2 = 0.99$ for all regression lines.
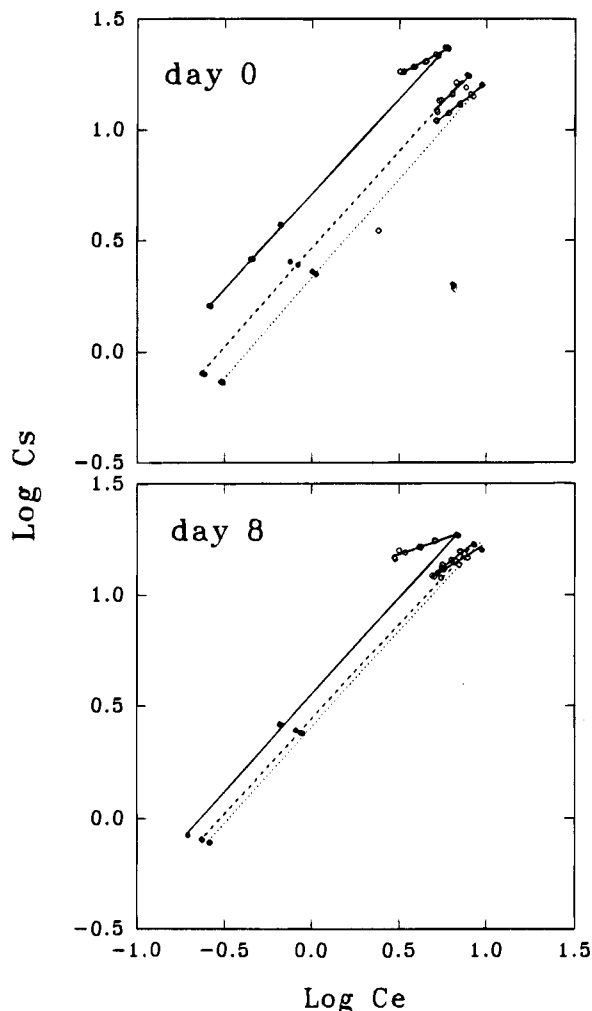
Figure 1. Adsorption and desorption isotherms for atrazine from the Brandt silty clay loam soil pretreated with 0 (—), 1400 (- - -), or 2800 (···) μg of N g^{-1} of soil for 0 or 8 days. Open circles represent the desorption data for atrazine applied at 50.80 μmol L^{-1} for each N treatment.

Figure 2. Adsorption and desorption isotherms for atrazine from the Ves clay loam soil pretreated with 0 (—), 1400 (- - -), or 2800 (···) μg of N g^{-1} soil for 0 or 8 days. Open circles represent the desorption data for atrazine applied at 50.80 μmol L^{-1} for each N treatment.

No hysteretic effect was observed for either soil at day 0 when treated with the medium ammonia rate as evidenced by similar $1/n_{des}$ and $1/n_{ads}$ values (Table 4; Figures 1 and 2). However, atrazine desorption from soil treated with either 0.01 M CaCl$_2$ or high ammonia rate was found to be hysteretic, i.e. $1/n_{des} < 1/n_{ads}$ (Table 4; Figures 1 and 2). Hysteresis means that a portion of atrazine is strongly or irreversibly bound to soil and cannot be readily desorbed. Hysteresis of atrazine has been reported previously (Clay et al., 1988b, 1990a; Bowman et al., 1985; Wauchope and Meyer, 1985).

In this portion of the study, pH and DOC appeared to co-influence atrazine sorption and desorption. Atrazine sorption remained fairly constant between incuba-

tion times in the ammonia-treated soil. If atrazine adsorption was controlled only by pH, then adsorption in the ammonia-treated soils should have increased with incubation treatment because the pH decreased. If only DOC controlled adsorption, then the influence of DOC on adsorption could have been dramatic, depending on the amount and reactivity of DOC in solution. Atrazine adsorption to soil pretreated with DOC has been reported to increase or decrease depending on the source of DOC (Barriuso et al., 1992). DOC could increase atrazine adsorption to soil if removal of DOC from soil particles opened up more atrazine sorption sites, or DOC could decrease atrazine sorption if DOC and atrazine interacted to form soluble complexes. Replacing soil

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Table 4. Influence of Ammonia Application and Incubation Interval on Atrazine Desorption Isotherms for Brandt Silty Clay Loam and Ves Clay Loam Treated with 50.8 μmol L^{-1} Atrazine

soil type	treatment	day 0			day 8		
		$K_{f,des}$[a]	$1/n$[b]	r^2	$K_{f,des}$	$1/n$	r^2
Brandt	control	11.86 (10.74–11.40)	0.42 (0.02)	0.98	11.12 (10.60–11.67)	0.27 (0.02)	0.95
	medium ammonia	3.69 (3.29–4.31)	0.75 (0.10)	0.98	4.62 (4.43–4.81)	0.61 (0.03)	0.98
	high ammonia	4.00 (3.85–4.15)	0.61 (0.02)	0.99	5.55 (4.94–6.23)	0.49 (0.08)	0.81
Ves	control	12.79 (12.48–13.12)	0.34 (0.01)	0.98	12.31 (12.10–12.52)	0.28 (0.01)	0.99
	medium ammonia	2.5 (2.28–2.74)	1.00 (0.06)	0.95	2.39 (2.20–2.61)	1.02 (0.07)	0.94
	high ammonia	2.9 (2.69–3.12)	0.81 (0.06)	0.94	3.57 (3.35–3.80)	0.70 (0.03)	0.98

[a] Numbers in parentheses are the 95% confidence interval (CI) for K_f, antilogs of log K_f − CI log K_f and log K_f + CI log K_f. [b] Numbers in parentheses are the standard error of the slope of the regression line.

Table 5. Solution pH and DOC Concentration of Control and NH$_4$OH Treated Brandt Silty Clay Loam and Ves Clay Loam Soil after 0 or 8 Days of Incubation after Separation from Soil

| soil type	treatment	day 0		day 8	
		pH	DOC	pH	DOC
Brandt	control	6.39 (0.08)[a]	60 (1)	7.47 (0.05)	27 (2)
	+ ammonia	10.11 (0.03)	1014 (33)	9.94 (0.05)	643 (10)
Ves	control	7.55 (0.05)	33 (1)	7.66 (0.04)	14 (1)
	+ ammonia	10.51 (0.01)	3197 (36)	10.24 (0.23)	2948 (4)

[a] Numbers in parentheses are the standard error of mean.

solution with "clean" 0.01 M CaCl$_2$ instead of with soil extract during the desorption equilibration has been reported to reduce both UV absorbance at 200 nm and DOC concentration of desorption solutions (Clay et al., 1988a). However, the changes in the solution composition accounted for less than 1% of the observed hysteresis from a Ves clay loam (Clay et al., 1988a). If pH alone controlled atrazine desorption, then desorption should increase from the medium to the high ammonia treatment. However, this was not observed.

Another explanation for the observed hysteresis in the high ammonia treatments is that hydroxyatrazine may have been formed at the high pH. The hydroxy metabolite has been shown to be more adsorbed and less desorbed than atrazine (Clay and Koskinen, 1990b). However, TLC analysis of the desorption solutions did not show evidence of significant amounts hydroxyatrazine or atrazine−DOC complex being formed (data not shown). However, due to the strong sorption of hydroxyatrazine to soil (Clay and Koskinen, 1990b), its concentration in solution when in the presence of soil would be negligible.

Ammonia−DOC−Atrazine Interactions. Further evaluation of the interactions between ammonia, DOC, and atrazine were conducted in the absence of soil. Higher solution pH and DOC content were obtained with the increased ammonia treatment compared to the sorption experiments (Table 5).

The TLC analysis of the ^{14}C solution treated with DOC extracted from the Brandt silty clay loam with a 5200 μg of N g^{-1} of soil application of ammonia had two bands of radioactivity after a 2-h incubation (Figure 3). One band having an R_f value of 0.80 contained approximately 90% of the ^{14}C and corresponded to parent atrazine. A second minor band, containing approximately 11% of the ^{14}C, had an R_f of 0.53. The R_f value of this band corresponded to hydroxyatrazine (Clay and Koskinen, 1990b).

At day 8, most of the ^{14}C still was located in the parent atrazine band. However, the hydroxyatrazine band increased in intensity and contained approximately 17% of the ^{14}C. Hydroxyatrazine bands also were observed in the ammonia−CaCl$_2$ treatment and
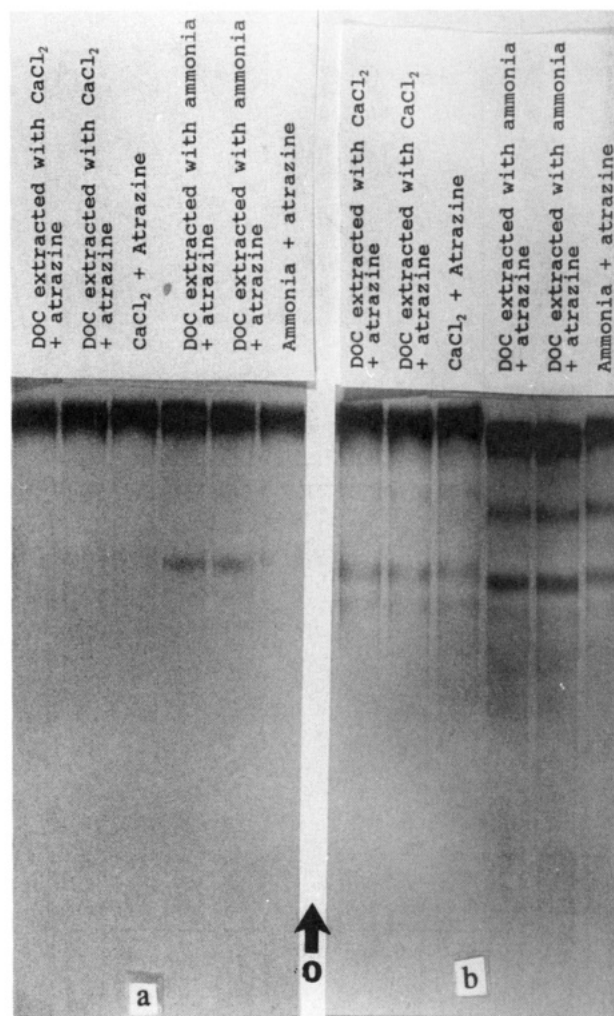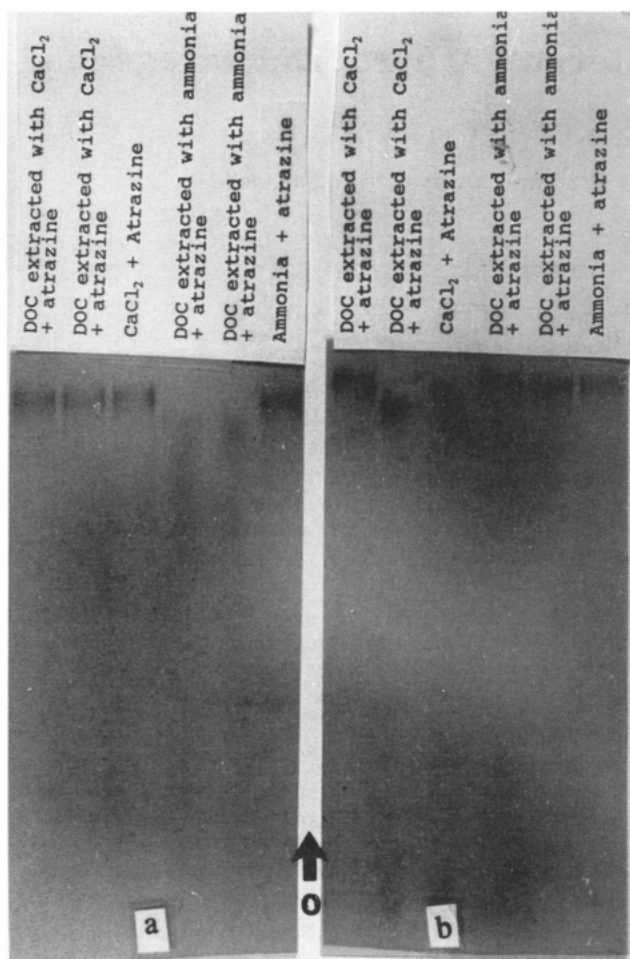

Figure 3. Autoradiograph of the ^{14}C compounds after [^{14}C]atrazine was incubated for 2 h (a) or 8 days (b) with 0.01 M CaCl$_2$, ammonia plus 0.01 M CaCl$_2$, or soil solution from Brandt silt loam soil extracted with either 0.01 M CaCl$_2$ or 0.01 M CaCl$_2$ plus 5200 μg of N g^{-1} of soil added as ammonia. "O" indicates the origin, and the arrow indicates the direction of solvent flow.

contained 8% of the radioactivity. A third band was observed on day 8. This band had an R_f value of 0.64 and contained up to 11% of the ^{14}C. This band was most prevalent in the DOC−ammonia solutions. The identification of this compound was not determined.

Hydroxyatrazine has a strong affinity for soil, and its K_f values for a silt loam soil ranged from 26 (pH 6) to 60 (pH 4.5) (Clay and Koskinen, 1990b). The formation of hydroxyatrazine and other metabolites may explain a portion of the observed hysteresis in high ammonia treatments of the Brandt soil.

Extracts from the Ves clay loam reacted differently to the ammonia treatments than the Brandt extract.

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Figure 4. Autoradiograph of the ^{14}C compounds after [^{14}C]atrazine was incubated for 2 h (a) or 8 days (b) with 0.01 M CaCl$_2$, ammonia plus 0.01 M CaCl$_2$, or soil solution from Ves clay loam soil extracted with either 0.01 M CaCl$_2$ or 0.01 M CaCl$_2$ plus 13 000 μg of N g^{-1} of soil added as ammonia. "O" indicates the origin, and the arrow indicates the direction of solvent flow.

Only parent atrazine bands ($R_f = 0.80$) were observed (Figure 4) when atrazine was incubated for 2 h in either the solution that was extracted from soil with 0.01 M CaCl$_2$ or ammonia solution. Atrazine incubated with soil solution that was extracted with 13 000 μg of N g^{-1} of soil ammonia solution and analyzed at 2 h and 8 days after treatment resulted in smeared bands on TLC plates. These smears indicate that an interaction occurred between DOC and atrazine when high amounts of ammonia and DOC were present in solution. The nature of this interaction was not investigated.

In summary, ammonia application raised soil solution pH and DOC content. Following ammonia application, atrazine adsorption to soil decreased and desorption from soil increased. Both pH and DOC played a role in influencing atrazine adsorption and desorption. The decrease of atrazine adsorption and the increase of atrazine desorption from ammonia treated soil increased atrazine concentration in solution. More atrazine present in solution may increase atrazine weed control efficiency. However, it also may increase the risk of atrazine leaching through soil to contaminate shallow aquifers. If rainfall or irrigation occurs soon (between 0 and 8 days) after atrazine–ammonia application, the atrazine present in solution may have a higher potential to leach with percolating water.

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