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Long-term tillage effects on atrazine and fluometuron sorption in Coastal Plain soils

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Abstract

Conservation tillage (CnT) management practices are known to increase levels of soil organic matter (SOM) in southeastern Coastal Plain soils. Plant residues in CnT systems accumulate at the surface and, with time, will form a layer enriched in SOM. The authors hypothesize that herbicide sorption will be highest in this SOM-enriched zone of CnT systems when compared to sorption at a similar depth in conventional tillage (CT) systems. The objective was to characterize the impact of two different tillage systems, CnT and CT, on sorption of atrazine [6-chloro-*N*-ethyl-*N*'-(1-methylethyl)-1,3,5-triazine-2,4-diamine] and fluometuron [*N*,*N*-dimethyl-*N*'-3-(trifluoromethyl)phenyl urea] in plots of Norfolk loamy sand (fine-loamy, siliceous thermic Typic Kandiudult). The plots have been under CnT and CT management for 18 yrs. Bulk (0–15 cm) and five equal incremental soil samples to a 15 cm depth were collected from 10 CnT and 10 CT plots, and the atrazine (ATR) and fluometuron (FLMT) sorption coefficients (K_d) were measured using batch equilibration. Significantly higher herbicide K_d values occurred in the CnT 0–3 cm samples, indicating that the highest amount of herbicide sorption occurred in the top few cm of soil. This corresponded to the stratified soil organic carbon (SOC) contents in topsoil of the CnT plots. In addition, analyses of covariance using SOC as the covariant to test for tillage effects indicated complex interactions among SOC, tillage, and depth. Those results confirm that tillage and soil depth will affect SOC contents of a Norfolk loamy sand, which correspondly will influence the magnitude of ATR and FLMT sorption.

Keywords: Tillage; Atrazine; Fluometuron; Sorption; Soil

1. Introduction

Conservation tillage practices are used to minimize soil erosion, improve water infiltration, and maintain long-term soil productivity (Logan et al., 1987). It is estimated that 60%–70% of all U.S. cropland will likely be farmed according to some type of CnT by the year 2000 (USDA-ARS, 1988). Conservation tillage is defined by the Conservation Tillage Information Center (CTIC, 1984) as any tillage operation that leaves ≥ 30% surface residue cover after planting. In CnT systems, the soil is left undisturbed from harvest to planting, and plant residues generated from the previous crop and from live vegetation before planting are left on the soil surface. The lack of physical mixing results in stratification of SOM contents, with the top few cm of soil usually having the highest SOM contents (Karlen

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et al., 1989; Lal et al., 1994; Reicosky et al., 1995; Hunt et al., 1996). In contrast, plant residues are physically incorporated into surface soils of CT systems, which promotes decomposition and minimizes the build-up of SOM levels at the surface.

Pesticide distribution patterns within soil are controlled by a complex series of equilibrium interactions between the pesticide and soil components (Locke, 1992). Soil organic and inorganic components will sorb a portion of the pesticide, which results in partitioning between solid and liquid phases. Pesticides in the liquid phase are readily available for weed uptake and leaching. Pesticides sorbed to solid phases are regarded as being slowly available for plant uptake because of strong bonding forces and slow desorption kinetic reactions (Novak et al., 1995). Pesticides sorbed by soil organic or inorganic solid phases are, with time, incorporated into a bound pesticide residue fraction that increases persistence. Sorption is the general term used to describe pesticide binding by soil components with no distinction made between specific processes of adsorption, absorption, and precipitation. The pesticide literature has amply shown that SOC is an important soil component affecting sorption, which regulates weed control efficacy, bioavailability, and persistence (Weber et al., 1993; Stevenson, 1994; Novak et al., 1995).

It has been shown in the Midwest region of the USA that surface soil (bulk samples 0–7.5 cm deep) under no-till (NT) management systems had higher amounts of chlorimuron (a herbicide) sorption when compared to soil under CT (Reddy et al., 1995). The higher amount of chlorimuron sorption in soils under NT systems was attributed to the greater SOC contents.

As the tillage literature has reported that the top few cm of soil in CnT systems are SOC enriched, a greater tillage effect on pesticide sorption may occur if only the top few cm of soil are sampled. Bulking the soil with non-SOC-enriched topsoil obtained from deeper A horizon depths may dilute this effect. Evaluation of tillage effects on pesticide sorption would be particularly germane to southeastern Coastal Plain soils, which are regarded as having a high pesticide leaching potential because of high rainfall amounts, sandy textures, and low SOM levels (Kellog, 1993). The present authors hypothesize that sorption of herbicides in sandy, Coastal Plain soils will be higher in soils under CnT systems than in soils under CT systems, and that the greatest difference will occur in the top few cm of soil. The objective was to evaluate the influence of long-term (18 yrs) CnT vs CT practices on the sorption of ATR and FLMT in plots of Norfolk loamy sand soils. These herbicides are used typically for weed control in corn (*Zea mays* L.) and cotton (*Gossypium hirsutum* L.) in the southeastern U.S. Coastal Plain and are known to have different soil sorption behaviors.

2. Materials and methods

2.1. Site description

The study was conducted using soil samples collected from 10 CnT and 10 CT plots at the Pee Dee Research and Education Center near Florence, SC. The soil in all plots was a Norfolk loamy sand. Each plot measured approximately 0.14 ha with slopes ranging from 0% to 2%. These plots have been managed for 18 yrs; and specific cropping, tillage, and pesticide application histories have been described previously (Karlen et al., 1989; Hunt et al., 1996). Conventional tillage within the plots consisted of multiple diskings (0–15 cm deep) and use of field cultivators (0–5 cm deep) to maintain a relatively weed-free soil surface and to incorporate crop residues, fertilizers, and lime. Conservation tillage within the plots completely eliminated disking and field cultivation for weed control. Both tillage systems received yearly in-row subsoiling (0–30 cm deep) at planting to fracture a root restrictive layer (E horizon) that forms annually within these soils (Busscher et al., 1986). The plots have been in several types of rotations including continuous corn, corn-wheat-soybeans, and corn-wheat-cotton. Bulk soil samples were collected from the Ap horizon (0–15 cm) at three random locations within each plot using a bucket auger and were composited. A bucket auger was also used to collect a core from the Ap horizon (0–15 cm deep), which was subsampled at five depths (0–3, 3–6, 6–9, 9–12, and 12–15 cm) at three random locations within each plot and then composited. A total of 120 soils samples was col-

lected (60 each from CT and CnT plots). Crop residue was removed from the surface before samples were taken. A 250 g portion of each sample was air-dried, crushed, and passed through a 2 mm sieve to remove plant materials. A portion was further ground to pass through a 0.25 mm sieve for SOC determination using a LECO carbon analyzer (LECO Corporation, St. Joseph, MI [1]).

2.2. Atrazine and fluometuron sorption

Sorption of ATR and FLMT by all soils was determined using batch equilibration. Possible background ATR and FLMT concentrations were checked in five randomly selected soil samples. These samples were shaken with 100% methanol for 24 h, filtered, and analyzed for ATR and FLMT as described below. These herbicides were not detected, indicating that background correction was unnecessary. 5 g of 2-mm sieved, air-dried soil were equilibrated for 24 h in 25-ml glass centrifuge tubes containing 20 ml of 1 mg l^{-1} ATR or FLMT (both 99% purity, Chem Service, West Chester, PA) dissolved in 0.005 M $CaCl_2$. Replicate tubes along with controls (herbicide without soil) were shaken at room temperature (22–24°C) using a rocking shaker (Eberbach shaker, Eberbach Corp., Ann Arbor, MI) at 80 cycles min^{-1} (6-cm stroke length). Preliminary kinetic sorption studies had shown that 24 h were sufficient for equilibration to occur, and no herbicide metabolites had been observed. After equilibration, the tubes were centrifuged, and 4 mL of supernatant were filtered using a 0.2 μM nylon filter disk (Supelco, Inc., Bellefonte, PA) and transferred to a glass vial. Herbicide (ATR or FLMT) in the initial and equilibrium solution was quantified by an HPLC system, which consisted of a Waters Nova-Pak 4 μM C_{18} column (Waters Chromatography, Marlborough, MA) using a mobile phase of acetonitrile (AcN) and Millipore Milli-Q H_2O at a gradient of 50:50 AcN:H_2O at 1 mL min^{-1}. Atrazine was detected using UV detection at 220 nm; and FLMT was detected using a fluorescence detector set to excite at 292 nm and to read emission at 345 nm, using a 1.5 s filter, 1000X gain, and ATTN = S. External standards that ranged in concentration from 0 to 2 mg L^{-1} were used to quantify both herbicides. The minimum detectable ATR and FLMT concentrations in the equilibrium solutions using a 25 μL injection volume were 5 and 2.5 ng per injection, respectively. Recovery of ATR and FLMT in controls ranged from 90% to 95% (compared to controls at time 0 h). The difference between initial ATR or FLMT concentration and the equilibrium solution was attributed to sorption by soil. The sorption equilibrium partition coefficient K_d (L kg^{-1}) was calculated as:

$$K_d = X/C \quad (1)$$

where X = mg of ATR or FLMT kg^{-1} of soil and C = mg of ATR or FLMT L^{-1} of equil. soln.

Atrazine K_{oc} values were calculated by normalizing for SOC using:

$$K_{oc} = [K_d/\%SOC] \times 100 \quad (2)$$

2.3. Experimental design and layout

The experimental plan was a split-plot design with tillage (CT vs CnT) as the main plot treatment and soil depth as the subplot treatment. An analysis of variance (ANOVA) was used to compare the effects of tillage on ATR and FLMT K_d values (1) in bulk soils (0–15 cm depth) and (2) in the mean of the five incremental soil depths. In addition, an analysis of covariance (ANOCOVA) was performed with SOC as the covariant because the ANOVA analysis revealed that additional factors may be responsible for the sorption trends. The relationship between mean ATR and FLMT K_d values by tillage with SOC and with soil depth was plotted to determine the slope of the response. Linear regression was used to evaluate the relationship between SOC and ATR or FLMT K_d values by tillage and by depth using the Statistical Analysis System (SAS Institute, Inc., Cary, NC). The ANOVA, ANOCOVA, and relationship between sorption coefficients with

[1] Mention of a trade-mark, proprietary product, or vendor is for information only and does not constitute a guarantee or warranty of the product by the U.S. Department of Agriculture and does not imply its approval to the exclusion of other products or vendors that may also be suitable.

SOC and soil depth were performed also using SAS. Significant tillage effects by soil depth for SOC, ATR K_d, and FLMT K_d were determined using a Students *t*-test and ATR K_{oc} and FLMT K_{oc} by a Mann-Whitney Rank Sum test using Sigma Stat (Jandel Scientific, San Rafael, CA) software.

3. Results

The highest SOC contents, ATR, and FLMT K_d values were found in the surface (0–3 cm deep) of the CnT plots and generally decreased with soil depth (Table 1). These results correspond to a highly significant depth effect and depth x tillage interaction (both $P < 0.0001$) for both ATR and FLMT K_d values, which suggest that the tillage effect is depth dependent. As anticipated, the F test values for the means of bulk (0–15 cm) samples and the means of depth-pooled samples for ATR and FLMT K_d values were not significantly different at the 0.1 level of rejection (P values range from 0.15 to 0.36). It was interesting that the mean ATR and FLMT K_d values and SOC contents at all depths in the CnT systems were more variable (higher SD) than in the CT samples, indicating that more field plot variability occurs in the CnT-managed systems; i.e., tillage mixes enough to homogenize.

Table 2 presents the effects of individual factors and interactions determined by the ANOCOVA analyses. No effect of tillage, when analyzed by itself, was found with respect to herbicide sorption coefficients. Depth and SOC effects varied with herbicide K_d and K_{oc} values. Significant depth, tillage, and SOC interactions on the herbicide sorption coefficients also occurred, indicating complex interactive effects.

Mean ATR and FLMT K_d values were plotted as a function of SOC content and soil depth to investigate interactive and depth relationships (Fig. 1). The plots reveal that SOC and soil depth will influence herbicide sorption coefficients and that these effects varied with tillage. In CnT plots, the highest mean herbicide K_d values occur in the 0 to 3 cm soil depth, which also has the highest SOC content. The relationship between herbicide sorption coefficients and SOC in CT systems is somewhat vague and generally did not change dramatically with soil depth. Regression analyses of mean ATR and FLMT K_d values with SOC and soil depth showed that a quadratic or linear regression equation best described the response (Table 3). In most cases, the model equations were significant ($P < 0.05$) and had high r^2 (> 0.79) values.

The simplistic relationship of explaining the variability in ATR and FLMT K_d values with SOC contents for both tillage systems was evaluated using linear regression analyses. Soil organic carbon contents accounted for 76%–78% of the variability in

Table 1
Mean SOC contents, ATR, and FLMT sorption coefficients (K_d). †

Depth (cm)	SOC ($g \cdot kg^{-1}$) ‡		ATR K_d ($L \cdot kg^{-1}$)		FLMT K_d ($L \cdot kg^{-1}$)	
	CT	CnT	CT	CnT	CT	CnT
0–3	10.2 (1.8) *	16.2 (2.3) *	1.4 (0.4) *	2.6 (0.9) *	1.1 (0.4) *	2.0 (1.1) *
3–6	10.7 (1.7) ‡	11.6 (3.1)	1.4 (0.4)	1.9 (0.9)	1.2 (0.5)	1.8 (1.1)
6–9	9.7 (1.6)	9.2 (3.4)	1.3 (0.6)	1.5 (1.1)	1.3 (0.6)	1.6 (1.1)
9–12	8.0 (1.2)	8.0 (3.6)	1.0 (0.3)	1.3 (0.8)	1.1 (0.4)	1.5 (0.9)
12–15	7.2 (1.7)	7.6 (3.2)	1.0 (0.4)	1.1 (0.7)	1.1 (0.5)	1.3 (0.9)
Bulk (0–15)	9.8 (2.4)	10.6 (3.1)	1.4 (0.6)	1.7 (0.9)	1.3 (0.7)	1.9 (1.1)

† Means calculated from data measured from 10 plots per tillage system.
‡ Values in brackets are standard deviations.
* Significant differences between CT vs. CnT by depth were determined using a Students t-test.

Table 2
Mean squares (n = 100) from the ANOCOVA for the effects of tillage, depth, SOC, and interactions on ATR and FLMT sorption coefficients (K_d and K_{oc}). †

Source	K_d		K_{oc}	
	ATR	FLMT	ATR	FLMT
Tillage	0.67 (0.30)	0.10 (0.15)	12823 (0.30)	6745 (0.30)
Depth	0.002 (0.80)	0.37 (< 0.01)	2968 (0.07)	207 (0.66)
Depth × Tillage	0.54 (< 0.01)	0.07 (0.24)	11323 (< 0.01)	3674 (0.06)
SOC	1.66 (< 0.01)	0.84 (< 0.01)	391 (0.5)	507 (0.49)
SOC × Tillage	0.23 (0.01)	0.02 (0.52)	8553 (< 0.01)	4503 (0.04)
SOC × Depth	0.46 (< 0.01)	0.62 (< 0.01)	1011 (0.28)	5873 (0.02)

† Number in brackets represents probability level of rejection.

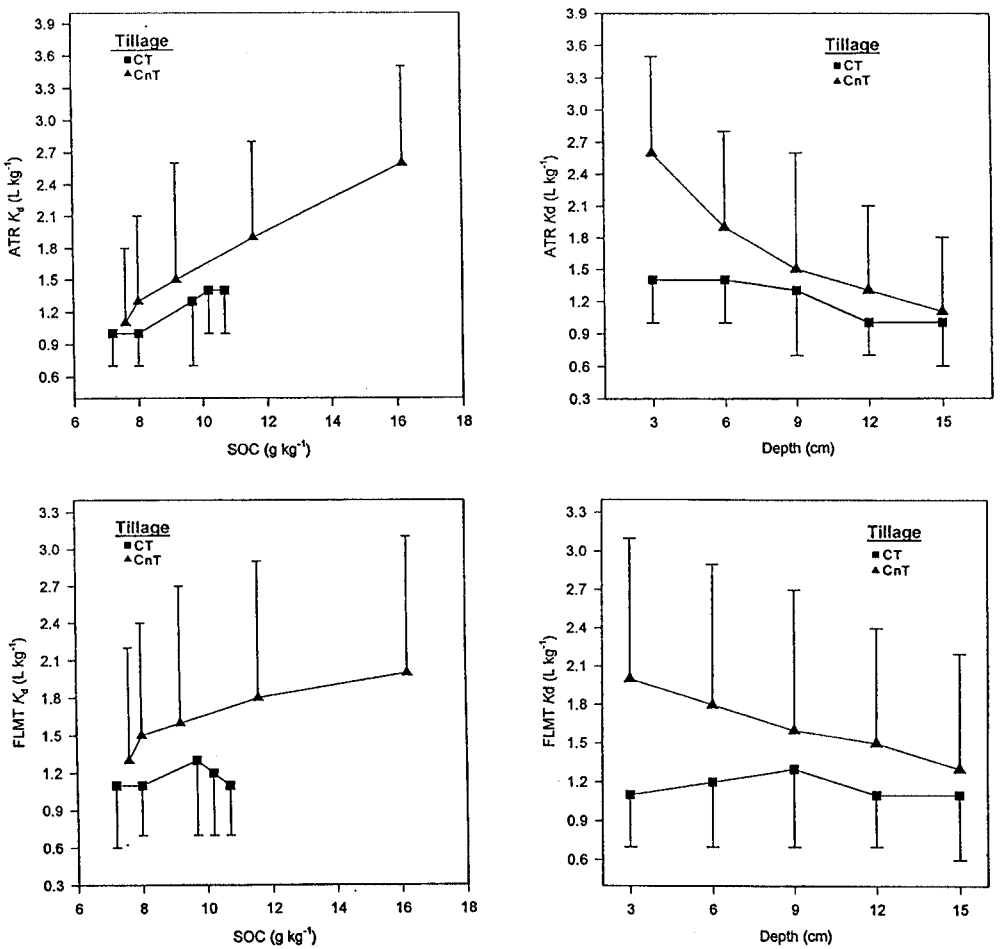

Fig. 1. Relationship between mean ATR K_d (top) and FLMT K_d (bottom) by tillage with SOC contents and soil depth (data points are means and error bars are 1 standard deviation from the mean, unidirectional to avoid overlap).

Table 3
Regression relationships between mean (n = 5) ATR and FLMT sorption coefficients (K_d) with SOC and soil depth in CT and CnT plots. †

Treatment	Variable	Tillage	Equation	r^2
SOC	ATR K_d	CT	$0.826 - 0.057(SOC) + 0.011(SOC^2)$	0.96 *
		CnT ‡	$-0.219 + 0.043(SOC)$	0.95 *
	FLMT K_d	CT	$-0.769 + 0.41(SOC) - 0.021(SOC^2)$	0.32
		CnT	$-0.264 + 0.282(SOC) - 0.009(SOC^2)$	0.96 **
Soil Depth	ATR K_d	CT	$1.519 - 0.125(d)$	0.81 *
		CnT	$3.33 - 0.86(d) + 0.086(d^2)$	0.99 *
	FLMT K_d	CT	$0.98 + 0.199(d) - 0.037(d^2)$	0.79 *
		CnT	$2.16 - 0.171(d)$	0.98 *

† Where SOC = soil organic carbon content (g kg^{-1}); d = soil depth in cm; and *, ** indicate significance at the 0.05 and 0.01 level of rejection, respectively.
‡ Atrazine K_d values were log transformed prior to regression analyses.

ATR K_d values in the CnT and CT systems, respectively. In contrast, SOC only accounted for 50%–58% of the variability between SOC and the FMLT K_d values in the CT and CnT systems, respectively. All linear regression equations (not presented) were highly significant ($P < 0.001$). Atrazine sorption was more strongly influenced (higher r^2 values) by SOC than FLMT sorption.

Table 4
Normalized ATR and FLMT sorption coefficients for SOC content (K_{oc}). †

Depth (cm)	ATR K_{oc}		
	CT	CnT	P §
0–3	130 (21) ‡	159 (35)	0.05
3–6	126 (23)	151 (38)	0.11
6–9	130 (33)	150 (49)	0.35
9–12	122 (27)	158 (25)	0.01
12–15	140 (27)	159 (120)	0.19
bulk 0–15	134 (31)	152 (34)	0.16
	FLMT K_{oc}		
0–3	111 (32)	121 (53)	0.97
3–6	115 (34)	139 (53)	0.27
6–9	128 (46)	157 (50)	0.21
9–12	136 (35)	180 (29)	0.01
12–15	144 (34)	184 (128)	0.91
Bulk (0–15)	130 (37)	165 (50)	0.11

† Values are means from 10 plots per tillage system.
‡ Values in brackets are standard deviations.
§ Significant differences between CT vs. CnT by depth were determined using a Mann–WhitneyRank Sum test.

Normalizing both ATR and FLMT K_d values for SOC resulted in mean K_{oc} values that ranged from 111 to 184 for both herbicides (Table 4). Many of the P values were close to the 0.05% level of rejection when tillage effects on herbicide K_{oc} values were determined. After normalizing for differences in SOC contents, differences in the ATR and FLMT K_{oc} values between tillages and with depth indicate that sorption was influenced by additional soil components (i.e., soil pH, % clay, quality of SOC, etc). Although not significant at the 0.05% level of rejection, higher mean ATR and FLMT K_{oc} values generally occur in soils under CnT rather than CT systems. Similar mean ATR K_{oc} values occur with depth in both tillage systems while, in contrast, FLMT K_{oc} values increase with increasing soil depth.

4. Discussion

The finding of higher SOC contents in the top few cm of soil in CnT plots confirmed the reports of Karlen et al. (1989) and Hunt et al. (1996) that long-term CnT management practices will create a SOC-enriched surface zone in a Norfolk loamy sand soil. In contrast, SOC contents in the CT systems are fairly similar with depth probably as a result of mixing by soil disking. The mean SOC contents with high standard deviations in the CnT plots suggest that the SOC distribution across the landscape may be more spatially variable probably because of a lack of mixing, which reduces the homogenization of the

surface soil layer. Non-uniformly distributed plant debris across the soil surface in CnT systems may also contribute to the high standard deviations in mean SOC contents.

As shown in Table 1, stratified herbicide sorption coefficients occurred with depth in soils under CnT systems. Higher mean ATR and FLMT K_d values were found in the top few cm of soils under CnT systems, which confirmed our hypothesis. In fact, mean ATR and FLMT K_d values in the 0 to 3 cm zone of CnT plots were nearly double the K_d values in the corresponding zone under CT system. This stratified pattern in both ATR and FLMT K_d values was confirmed by the authors ANOVA analyses, which indicated a significant depth and depth x tillage interaction for both herbicide sorption coefficients. Tillage effects (considered separately) were absent when bulk and the depth-pooled mean were compared. Collecting soils in a bulk manner spread out the SOC-enriched surface zone with soil with a lower SOC content, which promoted lower herbicide sorption coefficients. This finding contrasts with Reddy et al. (1995), who reported a tillage effect in bulk (0–7.5 cm deep) Midwest soil samples. The present data strongly indicate that CnT effects on herbicide sorption are depth dependent and were found only in very shallow SOC-enriched surface soil of the Norfolk loamy sand, which is typical for the southeastern Coastal Plain.

The ANOCOVA analyses revealed some significant SOC, tillage, and depth interactions that varied with herbicide. Soil organic carbon, when considered separately, was found to influence ATR K_d values; in contrast, tillage and depth had no effect. The significant interaction between tillage, depth, and SOC strongly implies that tillage and depth will influence SOC content which, in turn, will significantly influence ATR K_d values. The substantial effects of tillage, depth, and SOC on ATR K_d values are shown by plotting ATR K_d values as a function of tillage, depth, and SOC content (Fig. 1). The highest ATR K_d values occurred in soil under CnT, which had the highest SOC contents and also was at a shallow soil depth.

Some treatment effects were found influencing FLMT K_d values that did not influence ATR K_d values. Although FLMT K_d values were significantly influenced by SOC contents, a soil depth-effect was also noted. This indicates that differences in SOC contents between tillage systems did not completely explain differences in FLMT K_d values caused by depth. In summary, tillage and depth will affect SOC contents in the tillage systems, which consequently will influence the magnitude of ATR and FLMT sorption.

Most equations and coefficients of determinations used to describe the SOC content and soil depth response of ATR and FLMT sorption in both tillage systems (Fig. 1) were statistically significant (Table 3). This demonstrates that ATR and FLMT K_d values may be estimated to a 15 cm depth in the CnT plots with a high degree of accuracy. This finding offers researchers the ability to predict a pesticide's sorption with soil depth and then model potential movement in solution. The simplistic linear relationship between ATR and FLMT K_d values and SOC contents accounted for 76%–78% and 50%–58%, respectively, of the variability between these variables. The higher regression coefficients found when using SOC to explain variability in ATR K_d values illustrate the stronger affinity of ATR for SOC when compared to FLMT sorption behaviour. Although it was shown that a fair amount of herbicide sorption may be accounted for by considering SOC content alone, the complex interactions among SOC, depth, and tillage suggest that these interactions are much more important than any single factor influencing herbicide sorption, hence each estimate must be qualified by all parameters.

Pesticide K_d values are typically standardized for differences in SOC contents and provide a common basis to model pesticide leaching potential. The ranges of ATR K_{oc} values were similar to other published sorption data for ATR ($K_{oc} = 160$) by Jury et al. (1987). The FLMT K_{oc} values in the present study were also of similar magnitude for FLMT sorption ($K_{oc} = 143–179$) by Mueller et al. (1992). The ANOCOVA analyses revealed that tillage and SOC, considered individually, did not influence ATR or FLMT K_{oc} values. The effects of SOC were probably nullified as all sorption coefficients were normalized based on SOC contents. As found when comparing treatment effects on K_d values, ATR and FLMT K_{oc} values were influenced by the tillage, depth, and SOC interactions.

Normalizing pesticide K_d values for differences

in SOC allows the evaluation of additional soil components (i.e., pH, % clay, and quality of SOC), which also will influence the magnitude of pesticide sorption. There were some differences between tillage systems when ATR and FLMT K_{oc} values were compared by depth (Table 4). Some tillage effects were found at the $P = 0.05$ level of rejection, and many occurred at the 0.10 level. If $P = 0.1$ were accepted as the rejection level of tillage effects, then in general, higher ATR and FLMT K_{oc} values occur in soils under CnT than CT systems. The authors speculate that as soil in all plots and layers within plots have similar pH values (near pH 7) and clay contents (1%–5%), the difference may be caused by qualitative differences in organic structures that comprise the SOC fraction. As Preston et al. (1987) and Preston et al. (1994) have reported that tillage will affect the qualitative compositional properties of SOC in virgin and cultivated Canadian soils, it is plausible that organic structures in the SOC fraction in CnT systems may be compositionally dissimilar and possess functional groups that can bind more ATR and FLMT. The hypothesis that qualitative variations in SOC will influence pesticide sorption will require additional research.

The soil depth responses of ATR and FLMT K_{oc} values in both tillage systems were dissimilar. Mean FLMT K_{oc} values with depth in both tillage increased dramatically, and mean ATR K_{oc} values were nearly constant. The authors cannot offer a clear explanation for this trend but it may be the result of small qualitative changes in SOC or % clay with depth.

The present data suggest that the interaction of tillage and depth has a substantial effect on the SOC content in the CnT plots of the Norfolk loamy sand soil. As a result of the stratified SOC contents, herbicide sorption coefficients were highest in the top few cm of soil.

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