Low Sorption and Fast Dissipation of the Herbicide Saflufenacil in Surface Soils and Subsoils of an Eroded Prairie Landscape

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ABSTRACT: Saflufenacil partitioning and dissipation were evaluated in soils from an eroded prairie landscape to provide information on its expected environmental fate. Saflufenacil K_d values followed trends in soil organic carbon content. In surface soils, K_d values ranged from 0.05 to 0.2 L kg^{-1} in the depositional lower slope and from 0.02 to 0.06 L kg^{-1} in the eroded upper slope. In subsurface soils, K_d values were an order of magnitude higher in the lower slope (mean 0.1 L kg^{-1}) than in the upper slope (mean 0.01 L kg^{-1}). Sorption was slightly higher in samples aged 1–8 weeks compared to freshly spiked soils. Mean dissipation half-lives (DT_{50}) were 13 d in surface soils and 32 d in subsurface soils. The observed low sorption and relatively rapid dissipation of saflufenacil suggest that this herbicide will be readily available for degradation or plant uptake in the root zone.

KEYWORDS: degradation, dissipation, herbicide, retention, soil, sorption, subsoil

■ INTRODUCTION

Herbicide interactions with soil control their availability for plant uptake, transport, and degradation. The kinetics and extent of herbicide partitioning and transformation in soil are affected by properties of the herbicide and of the soil. Soils are inherently spatially variable, and soil erosion increases this variability. A study of loess- and till-derived soils throughout the midwestern United States[1] showed that soil loss by erosion affected soil organic carbon and clay contents, hydraulic conductivity, bulk density, and other soil properties, which can affect herbicide fate and transport. In hilly landforms, soil erosion by tillage and water results in complex redistribution of soil and soil carbon.[2]

In hilly cultivated prairie landscapes, convex landscape positions are characterized by removal of mollic material as a result of soil erosion. Eroded profiles have an Ap horizon relatively low in organic carbon, evidence of subsoil incorporation into the tilled layer, and a shallow depth to the C horizon. Deep soil profiles relatively high in soil organic carbon are present in concave landscape positions where soil accumulates through erosion.[3] In these landforms, laboratory assessments of sorption and transformation of a variety of herbicides have been shown to be spatially variable, with more sorption and slower degradation observed in the high-organic-matter soils of the lower slope compared to the eroded upper slope.[4−8] However, our recent work in one eroded prairie landform showed no differences due to landscape position in S-metolachlor retention and dissipation in field studies[9] or laboratory studies[10] despite a 50% difference in organic carbon concentration. Landscape position effects on herbicide fate and transport are not universal and require further investigation.

The herbicide saflufenacil (N′-{2-chloro-4-fluoro-5-[1,2,3,6-tetrahydro-3-methyl-2,6-dioxo-4-(trifluoromethyl)pyrimidin-1-yl]benzoyl}-N-isopropyl-N-methylsulfamide) (Figure 1) was
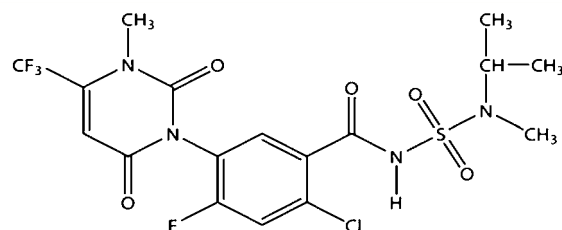

Figure 1. Chemical structure of saflufenacil.

registered in the United States in 2009 and is labeled for the control of broadleaf weeds in corn, soybean, and other crops. Saflufenacil has the following properties: water solubility (pH 7) = 2.1 g L^{-1}; K_{ow} = 368; Henry's law constant = 4.0 × 10^{-20} atm m^{-3} mol^{-1}.[11] It is characterized as having low acute toxicity to humans, fish, birds, and insects.[11] Saflufenacil is used as a crop desiccating harvest aid, in burndown treatments, and as a preemergence herbicide. Information regarding the mode of action, selectivity, and crop tolerance of saflufenacil are available in the peer-reviewed literature,[12−14] but little information has been published regarding the environmental fate of this new herbicide. Field studies in Canada showed that some sensitive crops demonstrated symptoms of carryover injury 1 year following saflufenacil application.[15] Additional information is needed to evaluate saflufenacil's potential for weed control, carryover, leaching, and runoff in a variety of soils in which it is likely to be used. In these studies, we determined saflufenacil

Table 1. Properties of Soils Used in These Experiments

	shoulder/upper backslope			footslope/toeslope		
Depth (cm)	0−10	15−30	45−60	0−10	15−30	45−60
Total C (wt %)	3.6	2.5	2.3	4.8	4.1	1.9
Organic C (wt %)	2.5	1.4	1.2	3.6	3.2	0.8
pH in CaCl$_2$	7.9	8.0	8.1	7.8	7.7	7.9
CEC (meq/100 g)	58.7	48.1	71.7	54.7	62.8	26.8
Total N (g kg^{-1})	1.3	0.6	0.4	3.2	3.0	0.9
Olsen P (mg kg^{-1})	17	11	6	30	27	22
Sand (wt %)	51	60	65	50	52	65
Clay (wt %)	20	16	16	20	18	14
Moisture (wt %)	14.2	12.7	11.8	22.4	20.7	12.4

sorption−desorption and rate of dissipation in surface and subsurface soils collected from an eroded prairie landscape.

METHODS

Chemicals. Technical saflufenacil (purity 99.6%) was kindly provided by BASF Corporation. Solvents were pesticide grade and were used as received. A stock solution of saflufenacil in methanol was prepared at a concentration of 300 μg mL^{-1}. Spiking solutions were prepared by diluting the stock solution with methanol.

Soils. Soils were collected from an eroded prairie landform typical of the Prairie Pothole Region. Soils are calcareous, formed in glacial till. The field site in west central Minnesota, USA (45.65° N, 95.83° W) has been cultivated for about 100 years and is affected by long-term cultivation and cropping. Using a shovel, soils were collected from one location in the eroded upper slope (at the boundary of shoulder and upper backslope positions) and one location in the depositional area (boundary of footslope and toeslope) in depth increments of 0−10, 15−30, and 45−60 cm. Soils were passed through a 2 mm sieve, then frozen until use. Selected physical and chemical properties were determined. In the upper slope, soil nutrient and organic carbon concentrations decline sharply with depth, whereas in the lower slope, soil properties are relatively consistent throughout the top 30 cm (Table 1).

Traditional Sorption Determinations. Triplicate aliquots of field-moist soil (10 g dry weight equivalent) were weighed into 50-mL glass centrifuge tubes. Additional tubes were prepared with no soil. Saflufenacil in 0.01 M CaCl$_2$ solution (10 mL) was added to soils to achieve concentrations of 0.03, 0.1, 0.3, and 1.0 μg saflufenacil/g of dry soil. A soil concentration of 0.1 μg g^{-1} corresponds to a standard saflufenacil application rate[13] of 63 g ai ha^{-1} and incorporation to 5-cm depth, assuming a soil bulk density of 1.3 g cm^{-3}. Samples were vortexed, then placed on a reciprocating shaker operating at 190 cycles min^{-1} for 20−24 h (temperature 20−23 °C), and centrifuged at 760g for 10 min. Using a disposable glass pipet, 3 g of the aqueous phase were transferred to a disposable glass test tube. Acetonitrile (3 mL) was added to the CaCl$_2$ extract to match the LC mobile phase. Approximately 3 mL of acetonitrile/CaCl$_2$ solution was drawn into a glass syringe and filtered using a 0.45 μm Teflon-membrane syringe filter. The first 1 mL of solution was used to condition the filter, and the remainder was eluted into a glass vial for analysis.

Sorption coefficients were determined for each soil and each spiking concentration using $K_d = C_{sorbed}/C_{solution}$, where C_{sorbed} is determined from the decrease in saflufenacil concentration in tubes containing soil compared to those containing no soil, and $C_{solution}$ is the concentration of saflufenacil in the aqueous extract after equilibration with soil. Sorption coefficients were normalized to the concentration of organic carbon in each sample to determine K_{oc}.

Dissipation and Sorption of Aged Residues. Traditional sorption determinations used a slurry of 1:1 soil/water. To evaluate saflufenacil sorption and dissipation under more realistic moisture conditions, spiked field-moist samples were incubated and extracted using sequential solvent extraction. Triplicate aliquots of soil (10 g dry weight equivalent) were weighed into 50-mL glass centrifuge tubes. Soils were spiked with 100 μL of saflufenacil solution to achieve concentrations of 0.1 and 1 μg saflufenacil/g of dry soil. After spiking, samples were left for 1 h in the fume hood to allow the methanol to evaporate, then soil samples were mixed, capped, and placed in an incubator (25 °C) for 1, 2, 4, and 8 weeks. Soil moisture as collected was 0.12−0.22 g g^{-1} (Table 1). Soil moisture was not adjusted and remained constant during incubation.

At the end of the incubation period, triplicate samples were removed and 10 mL of 0.01 M CaCl$_2$ solution was added. Samples were equilibrated, centrifuged, transferred, diluted, and filtered as described for the traditional sorption determinations. To the soil/CaCl$_2$ solution in the centrifuge tube, 3 g of acetonitrile was added. Samples were vortexed to mix, placed on reciprocating shaker for at least 2 h, then centrifuged. An aliquot (3 g) of the supernatant was transferred to a glass test tube. This process was repeated, the acetonitrile/CaCl$_2$ solutions were combined, and the extract was filtered as for the aqueous extract.

Saflufenacil that was not extracted using CaCl$_2$ solution, but was extracted with acetonitrile−water, was considered sorbed. The ratio of saflufenacil concentration in the organic extract to the saflufenacil concentration in the aqueous extract is analogous to a sorption coefficient. Straightforward comparisons of the ratio of herbicide concentrations in organic and aqueous extracts were used to compare sorption in aged samples to sorption in freshly spiked soils. Saflufenacil dissipation rates were determined by fitting a first-order kinetic model to the concentration of saflufenacil remaining in soil at each incubation time.

Desorption. Desorption was determined for samples spiked with 0.1 and 1 μg saflufenacil/g of dry soil. Samples were spiked and extracted with 0.01 M CaCl$_2$ as for the sequential solvent extraction experiment (with no incubation). An aliquot (3 g) of the aqueous supernatant solution was removed and replaced with 3 g of fresh 0.01 M CaCl$_2$. Samples were vortexed, placed on a reciprocating shaker for 24 h, and centrifuged. Again, a 3-g aliquot of the supernatant was removed and replaced with 3 g of fresh CaCl$_2$. After three desorption steps were thus completed, the soil was extracted with acetonitrile as described for the sequential solvent extraction study.

LC/MS/MS Analysis. Because analysis is resource-intensive, only two of the three replicate samples were analyzed. Concentrations of saflufenacil were determined using a Shimadzu (Kyoto, Japan) high performance liquid chromatograph coupled with an Applied Biosystems (Carlsbad, CA) API 3200 triple quadrupole. A Columbus 5 μm C8 110A, 50 × 2.00 mm column (Phenomenex;Torrance, CA) was used for separation. The column was maintained at 40 °C. The mobile phase was isocratic, 50% water (0.1% formic acid) and 50% acetonitrile, at a flow rate of 0.2 mL min^{-1}. The sample injection volume was 50 μL. Samples were maintained at 15 °C in the autosampler to minimize decomposition.

Tuning parameters were optimized by direct infusion. Saflufenacil was detected using the mass spectrometer operated in turbo spray negative ionization multiple reaction monitoring (MRM) mode with the following mass spectrometer conditions: curtain gas interface, 30 psi; ion source voltage, −4500 V; gas 1, 30 psi; gas 2, 30 psi; ion source temperature, 350 °C; collision gas, medium; dwell time, 200 ms. The MRM transitions (m/z) used were: M − H 498.8 > 347.7 and 498.8 >

327.7. Comparing saflufenacil spikes in water versus soil extracts, no matrix effects (e.g., signal enhancement or suppression) were observed. For quantitation, calibration was against external standards; the calibration curve was linear between 10 and 1000 ng mL^{-1} (r = 0.996). Recoveries from spiked soil extracts were 80−110%.

The limit of blank was determined (mean + 1.645 × standard deviation of blanks = 0.2 ng mL^{-1}) from 8 blank centrifuge tubes that were treated in the same manner as the soil samples. The limit of detection was 1.7 ng mL^{-1}, determined as the limit of blank + 1.645 × standard deviation of a very low standard (n = 26). Only three samples (upper slope surface soil samples incubated for 8 weeks) were below the limit of detection.

■ RESULTS AND DISCUSSION

Sorption Coefficients. Sorption in upper slope soils did not change with initial concentration. For lower slope soils, K_d was lower at the highest initial concentration (1 μg g^{-1}) compared to the other initial concentrations. Saflufenacil sorption did not fit the nonlinear Freundlich equation (no improvement in r^2 compared to a linear isotherm). Therefore, K_d values are discussed.

In the surface soils, individual K_d values ranged from 0.02 to 0.2 L kg^{-1}, and the mean K_d was plotted in Figure 2. These
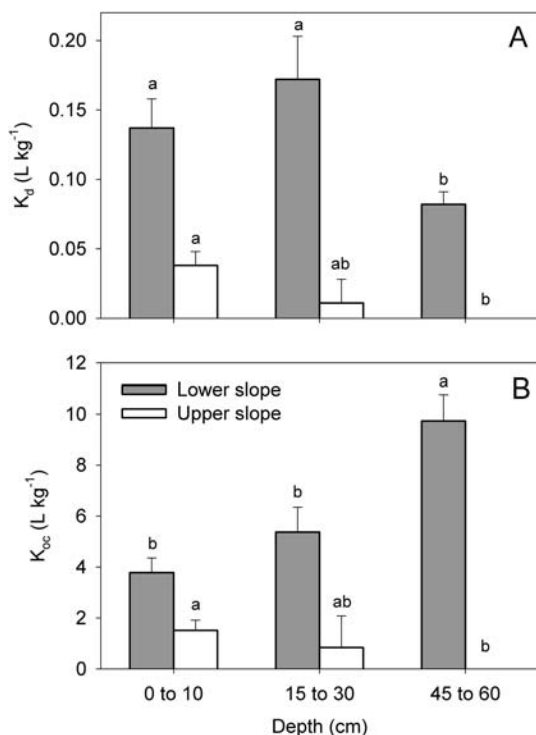

Figure 2. Sorption coefficients (A) and normalized sorption coefficients (B) for saflufenacil. Values are the mean of duplicate samples at four nominal concentrations. Letters indicate significant differences within the same landscape position.

sorption coefficients are similar to those found for the weak acid herbicide aminocyclopyrachlor (0.04−0.12 L kg^{-1})[16] but much lower than those measured for the acetanilide S-metolachlor (3 L kg^{-1})[10] in soils from the same field site. Our results are consistent with those of Hixson,[17] who reported K_d values of 0.06−2.4 L kg^{-1} for saflufenacil sorption to seven surface soils, including a Chernozem of pH 7.5 from Saskatchewan (K_d = 0.07 L kg^{-1}).

Saflufenacil sorption to soils from 45 to 60 cm depth was significantly lower than sorption to surface soils (0−10 cm).

Trends in saflufenacil sorption with depth followed trends in soil organic carbon content. For soils from the upper slope, organic carbon concentrations were generally low and decreased with depth (Table 1), as did saflufenacil sorption (Figure 2A). In contrast, organic carbon concentrations were high throughout the top 30 cm of the lower slope profile, then decreased sharply in soils at 45−60 depth (Table 1); saflufenacil sorption followed the same pattern (Figure 2A).

At each depth, we observed more saflufenacil sorption to soils from the lower slope than to those from the upper slope (Figure 2A), again in agreement with trends in soil organic carbon concentration. At this site and another eroded site in the same county, previous assessments have shown more herbicide sorption in the lower slope compared with the upper slope for the weak acids 2,4-D,[18] aminocyclopyrachlor, and picloram,[16] but no or small differences for S-metolachlor[10] and glyphosate.[18] Together with previous observations, these results concur with Farenhorst et al.,[18] who noted that soil properties and terrain attributes tend to have a stronger influence on herbicides demonstrating low sorption (including saflufenacil) than on more highly sorbed herbicides.

Using a series of bivariate regressions, saflufenacil K_d values were found to be positively (α = 0.05) correlated with parameters indicating higher organic matter (organic carbon concentration, total N, Olsen P, and moisture; r = 0.58, 0.74, 0.78, and 0.70, respectively) and negatively correlated with parameters indicating soil loss by erosion (pH and sand content; r = −0.79 and −0.50, respectively). Sorption of the weak acid 2,4-D (pK_a 2.64) to calcareous soils with pH > 7 showed similar trends.[18] Saflufenacil is a weak acid (pK_a = 4.41) and sorption is likely to increase at low pH. All soils used in these studies had pH > 7, so all saflufenacil will be anionic, and small variations in soil pH are not expected to affect sorption. Hixson[17] saw no significant correlation between native soil pH and saflufenacil K_d, but sorption did decrease by a factor of 5 when soil pH was increased through the addition of CaO.

Despite the correlation between soil organic carbon and K_d, normalizing saflufenacil sorption coefficients to the fraction of organic carbon in the soil (K_{oc}) did not reduce the variation in sorption coefficients. In the lower slope, differences in soil organic carbon concentration are larger than differences in saflufenacil sorption (Figure 2B). Values of K_{oc} for the lower slope are higher than those for the upper slope (Figure 2B), suggesting that the organic matter in the depositional area has a stronger affinity for saflufenacil than does organic matter in the eroded upper slope. Metolachlor showed an opposite trend[10] and K_{oc} values for aminocyclopynachlor and picloram were the same in the upper slope and lower slope.[16] Differences in organic matter sorptivity across this landscape appear to be compound-specific, emphasizing the need for compound- and soil-specific information to enable accurate predictions of pesticide fate.

Desorption. In desorption experiments, solution concentrations decreased in proportions equivalent to the sequential dilution of the solution. During each desorption step, saflufenacil remaining in the centrifuge tube was diluted through addition of fresh CaCl$_2$ solution. The solution concentration that would result from dilution was calculated and compared to the solution concentration measured in the desorption study. Solution concentrations following triplicate desorption steps were always within 20% of the calculated dilution (Figure 3), with a mean ratio of 1.04 ± 0.03. These

results indicate for the small amounts of saflufenacil that were sorbed, desorption from freshly spiked soil was not hysteretic.
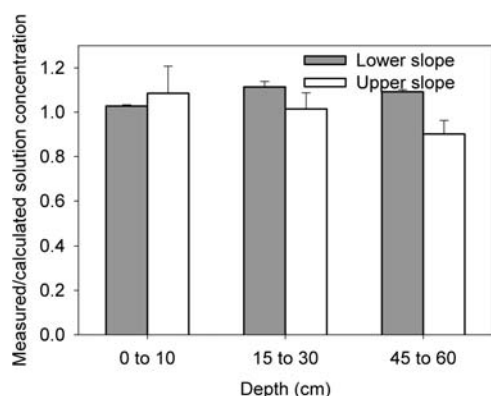

Figure 3. Ratio of measured saflufenacil solution concentration following 3 desorption steps to the solution concentration calculated based only on dilution during the desorption experiment. Values are the mean ± standard error of samples spiked with 0.01 and 0.1 $\mu g\ g^{-1}$. A value of 1 indicates no hysteresis in saflufenacil partitioning compared to freshly spiked soils.

Change in Sorption with Aging. We used sequential solvent extraction to evaluate changes in saflufenacil sorption in field-moist soils. More sorption is indicated by higher ratio of saflufenacil concentrations in the organic extract to that in the aqueous extract. In our study, sorption tended to be lowest at time 0, increased in samples aged 1 week, then remained constant through 8 weeks of incubation (Figure 4). The aging
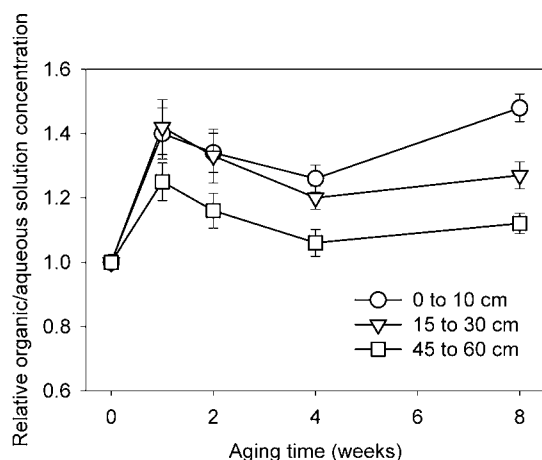

Figure 4. Ratio of saflufenacil concentrations in organic extract to concentrations in the aqueous extract. Values are relative to freshly spiked soils and are the mean (± standard error) across soils (upper and lower slope) and two initial concentrations (0.1 and 1 $\mu g\ g^{-1}$). Values greater than 1 indicate more sorption in aged samples than in freshly spiked soils.

effect was stronger for surface soils than for subsurface soils (Figure 4). Saflufenacil concentrations in samples spiked with 0.1 $\mu g\ g^{-1}$ and aged 4 to 8 weeks were very low, especially in surface soils, owing to the rapid dissipation of saflufenacil in these soils. There is therefore substantial scatter in the data, but the ratio of saflufenacil concentrations in organic and aqueous extracts was 1.1−1.5 times higher for aged samples than for freshly spiked soil (Figure 4). Differences in sorption with aging

followed trends in sorption coefficients: surface soils showed the most saflufenacil sorption and a significant (α=0.05) aging effect; subsurface soils from 45 to 60 cm depth demonstrated very low sorption and no significant aging effect; and soils collected from 15 to 30 cm depth were intermediate in both sorption coefficients and aging effects.

The magnitude of the aging effect was similar to that observed for a variety of pesticides. Previous reports of aging effects range from no change in sorption to increasing sorption by a factor of 7, depending on pesticide/soil combination. For weak acids like saflufenacil, pH can influence the aging effect, but in our study all soils had pH >7 (Table 1). No aging effect was observed for the diketonitrile metabolite (DKN) of isoxaflutole in soils at pH 7−8, but aging increased sorption by a factor of 1.8 in soil with pH 5.7.[19] In soil with pH 5.4, sorption of imazamox increased by a factor of 1.5 when aging increased from 1 to 6 months.[20] Dicamba, which like saflufenacil is weakly sorbed and rapidly degraded, did not demonstrate a significant aging effect until <15% of the applied dicamba remained.[21] Sorption increased with aging by a factor of >2 for nicosulfuron,[22] the urea metabolite of imidacloprid,[23] the dicamba metabolite 3,6-DSCA,[21] and sulfonylaminocarbonyltriazolinone herbicides.[24] Sorption of triazine herbicides increased by 1.4−3 times after incubations of 1 to 2 weeks, and then remained relatively constant in incubations of 4−8 weeks.[25,26] We observed the same trend, with increased saflufenacil sorption 1 week after spiking, but no subsequent increase with longer incubations of up to 8 weeks.

The reason for increased sorption with increased residence time in soil is unclear. Retention mechanisms could change with time, with pesticides shifting to stronger binding sites or diffusing to less accessible sorption sites. Sorption is often concentration-dependent, with more sorption at lower concentrations (Freundlich $1/n$ value < 1). In such cases, degradation during the incubation results in lower concentrations and higher K_d consistent with the nonlinear sorption isotherm. A combination of mechanisms may be manifested in observed aging effects.

Dissipation. The rate of saflufenacil dissipation was determined from the amount of saflufenacil extracted in samples aged for 0, 1, 2, 4, and 8 weeks as part of the sorption study. First-order dissipation rates were 0.05 ± 0.01 d^{-1} in surface soils and 0.022 ± 0.007 d^{-1} in subsurface soils (Figure 5), translating to mean DT_{50} of 13 d in surface soils and 32 d in subsurface soils. These results are consistent with manufacturer-reported mean DT_{50} of 15 d in lab studies and 17 d in field studies.[27] The dissipation rate was the same for the two initial concentrations (0.1 and 1 $\mu g\ g^{-1}$), for soils from the upper and lower slope, and for samples collected from 15−30 and 45−60 cm. Degradation has also been shown to be slower in subsurface soils than in surface soils for a variety of herbicides including chloroacetamide,[28,29] phenoxycarboxylic acid,[30,31] urea,[4,32,33] 1,2,4-triazinone,[4] 1,3,5-triazine,[4,28] diphenyl ether,[34] and sulfonylurea[35] herbicides.

The degradation rate of herbicides is primarily dependent on the presence and activity of a microbial population capable of degrading the herbicide, but is also dependent on the availability of the herbicide to the organisms. In most cases, decreased degradation with increasing soil depth has been directly correlated to decreased microbial populations (MPN, bacterial plate counts, biomass, activity, respiration numbers, etc.)[4,29−32] as opposed to decreased availability. Decreased degradation rates with increased soil depth have also attributed
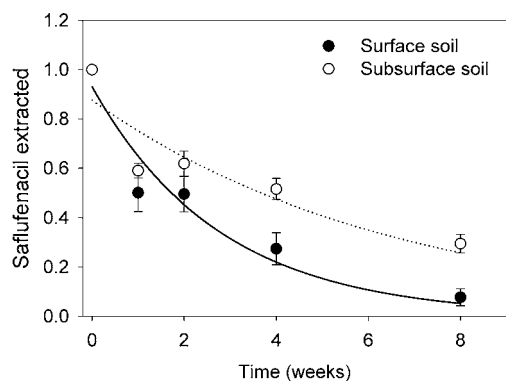
Figure 5. Saflufenacil dissipation in surface and subsurface soils. Values are the mean ± standard error across soils (upper and lower slope) and two initial concentrations (0.1 and 1 μg g^{-1}). Surface soils are 0−10 cm; subsurface soils are mean of 15−30 and 45−60 cm depth. Lines are first-order kinetic model.

to the time required for adaptation of the degrading population in subsurface soils.[33]

In the case of saflufenacil, the slower dissipation rate in subsurface soils is presumably due to lower populations or lower activity of degrading microorganisms (or a combination of the two) in subsurface soil as compared to surface soil. The results of this study have shown that bioavailability does not limit saflufenacil dissipation: sorption was low and easily desorbed using an aqueous extraction in freshly spiked soils and in samples aged up to 8 weeks. These results suggest that saflufenacil in the root zone will be readily available for degradation or plant uptake.

In these studies, saflufenacil was found to interact little with soils typical of the northern U.S. Corn Belt. Although sorption coefficients were greater in soils from the depositional lower slope than in soils from the eroded upper slope, sorption coefficients were low ($\leq$0.2 L kg^{-1}) in all cases. Sorption to soil is not expected to limit saflufenacil uptake. Under static laboratory conditions, saflufenacil dissipated with a DT$_{50}$ of about 2 weeks in surface soil and 1 month in subsurface soils. These results suggest that under typical conditions, saflufenacil will not present significant carryover in the U.S. Corn Belt. Its low sorption coefficient could make saflufenacil prone to leaching shortly after application so, like all agrochemicals, it should be judiciously used to avoid water contamination.

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Notes
The authors declare no competing financial interest.

■ ACKNOWLEDGMENTS

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