Research Article

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Soil residue analysis and degradation of saflufenacil as affected by moisture content and soil characteristics†

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Abstract

BACKGROUND: Saflufenacil dissipation in soils under different moisture conditions is not available in the scientific literature. The objective of this study was to evaluate saflufenacil degradation and persistence in soils from rice regions under field capacity (non-flooded) and saturated (flooded) conditions.

RESULTS: The accelerated solvent extraction (ASE) residue analytical method developed to conduct the study resulted in recovery greater than 80% for the combinations of soils and moisture conditions. Saflufenacil degradation was faster at field capacity for all soils, except for Morey soil. Herbicide half-life was 28.6, 15.0 and 23.1 days under field capacity treatments and 58.8, 36.9 and 79.7 under saturated conditions for Nada, Crowley and Gilbert soils respectively. A half-life no longer than 80 days was observed for the combination of soils and moisture treatments.

CONCLUSION: An ASE method was developed and used to extract saflufenacil from soil samples. Half-life averaged among soils was 59 and 33 days for saturated and field capacity respectively. Saflufenacil persistence in the environment was 2–3 times longer under flooded conditions for most of the soils studied.

Keywords: accelerated solvent extraction; field capacity; flooded; half-life; Sharpen®

1 INTRODUCTION

Saflufenacil is a new protoporphyrinogen IX oxidase (PPO or Protox) inhibitor[1] that controls several broadleaf weed species, as indicated in the approved label of Sharpen®. Sharpen® has been recently registered for commercialization in the United States. Research has demonstrated that saflufenacil can be safely used in pre-emergence applications with several winter and summer crops,[2–5] justifying pre-emergence approval in multiple crops. Additionally, the herbicide is currently recommended in preplant burndown application programs, providing an alternative for difficult to control and herbicide-resistant broadleaf weeds.[6–8] The number of cases of reported weed resistance to glyphosate, the primary herbicide used for burndown applications, has increased considerably from 51 in 2005 to 138 in 2011 (www.weedscience.org). In recent cases, broadleaf weeds such as Palmer amaranth (*Amaranthus palmeri*), common waterhemp (*Amaranthus tuberculatus*) and *Conyza* species have accounted for a significant number of resistant weeds. Therefore, saflufenacil has potential to be widely used in combination with other burndown herbicides to help manage broadleaf resistance problems in production areas.[6,7]

Additionally, a supplemental label for Sharpen® was approved during 2011 for preplant burndown applications in rice (CDMS: www.cdms.net/LDat/ld99E010.pdf). Studies investigating alternative usage patterns for saflufenacil had demonstrated that rice was tolerant to pre-emergence applications.[9] Also, saflufenacil had been effectively used to control hemp sesbania (*Sesbania exaltata*) in post-emergence applications,[10,11] with potential to expand the weed control spectrum in combination with other rice herbicide programs.[9,10] In spite of these findings, saflufenacil is not recommended for pre-emergence application in rice and post-emergence application in any row crop. Rice production is different from other major crops, as plants can be cultivated under flooding conditions.[12] Therefore, saflufenacil behavior and fate in the soil have to be well understood, considering the environmental

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aspects of the rice production ecosystem, before this herbicide can be approved for further label expansion.

Saflufenacil has potential to be used in multiple agricultural systems. However, there has been no work published in the scientific journals on the degradation and persistence of saflufenacil in soils, especially under the flooded conditions found in an irrigated rice field. Degradation is one of the key processes affecting a pesticide's environmental impact.[13] Dissipation patterns of a pesticide would be expected to differ in lowland and upland environments. The soil profile in a lowland flooded rice paddy undergoes microbiological and chemical transformation,[14] which can affect degradation rates. For instance, anaerobic microorganisms predominate in the soil community as oxygen is depleted after a flooding event in rice production.[14]

As saflufenacil is a new herbicide that has demonstrated promise in a number of agricultural scenarios, it is important to have more data regarding the environmental fate of this material. At the moment, results indicating saflufenacil dissipation in soils under different moisture conditions are not available. Thus, information regarding saflufenacil degradation in soils from different geographic regions will provide information for more effective agronomic and environmental management. The objective of this study was to evaluate saflufenacil degradation and persistence as well as microbial activity in soils from different rice regions under field capacity (non-flooded) and saturated (flooded) conditions.

Table 1. Soil sample characterization and gravimetric water content (θ_g) for soils collected in Eagle Lake (EL) and Beaumont (BM), Texas, and in Crowley (CR) and Gilbert (GB), Louisiana[a]

Parameters	Soil sample locations			
	EL	BM	CR	GB
Soil characterization				
Soil series name	Nada	Morey	Crowley	Gilbert
Texture class[b]	FSL	L	SiL	SiC
Sand (%)	56.8	29.4	8.3	6.4
Silt (%)	33.6	46.5	77.3	48.6
Clay (%)	9.6	24.1	14.4	45.0
Organic carbon (%)	0.79	1.21	1.18	3.52
pH	6.5	7.8	5.7	5.3
CEC (meq/100 g)	6.9	26.3	12.2	44.2
Gravimetric water content (mg water g soil $^{-1}$)[c]				
Field capacity (−33 kPa)	159(±15)	264(±20)	326(±2)	415(±10)
Saturation (0 kPa)	363(±10)	523(±22)	614(±41)	807(±22)

[a] Samples were analyzed by the Soil Characterization Laboratory located at Texas AgriLife Research and Extension, College Station, TX. Gravimetric water content was estimated using −33 kPa (field capacity) and 0 kPa (saturation) as the water potential.
[b] FSL, fine sandy loam; L, loam; SiL, silt loam; SiC, silty clay.
[c] Values within parentheses represent the standard deviation of four replications.

2 MATERIALS AND METHODS

2.1 Soils

Four samples were collected in rice-producing areas located in Eagle Lake and Beaumont, Texas, and in Crowley and Gilbert, Louisiana, by sampling the top horizon of the soil (15 cm upper layer). Samples were brought to the laboratory, air dried and then passed through a 2-mm sieve for removal of particles and non-decomposed plant residues. The prepared soil samples were stored at room temperature (24 ± 0.8 °C) during the studies. A representative subsample was submitted for analysis at the Texas AgriLife Research and Extension Soil Characterization Laboratory located in College Station, Texas. Particle size distribution, total organic carbon, pH and cation exchange capacity results for the Nada, Morey, Crowley and Gilbert soil series are presented in Table 1.

2.2 Soil moisture treatments

A water retention curve was used to determine the amount of water to be added in the field capacity and saturated treatments. A sample of each soil was placed inside a ring positioned over a suction plate. Water was applied on the plate and samples were allowed to saturate. The chamber containing the suction plate was sealed, and negative pressure (−33 kPa) was applied to estimate the field capacity moisture.[15] Water content for the saturated treatment was determined by applying no pressure (0 kPa) on the plate. After 24 h, samples were removed from the suction plate and weighed to acquire the wet weight. Subsequently, samples were oven dried at 105 °C for 48 h to obtain the dry weight. For each soil sample and water potential, the gravimetric water content (θ_g) was calculated using the following equation:

$$\theta_g = \frac{\text{wet soil weight} - \text{dry soil weight}}{\text{dry soil weight}} \quad (1)$$

Results (in mg water g soil^{-1}) are listed in Table 1. The amount of water to generate the moisture treatments was calculated according to the soil sample size (g).

2.3 Saflufenacil degradation

Air-dried samples of each soil (10 g) were placed in a round-bottom centrifuge tube, rewetted to reestablish microbial activity and preincubated in the dark for 14 days prior to herbicide and moisture treatment applications. All samples were rewetted to bring the soil moisture to 50% of the field capacity. The amount of saflufenacil to be added to the samples (μg g soil^{-1}) was estimated on the assumption that a 15 cm furrow slice in the area of a hectare would have approximately 2 250 000 kg of soil. Additionally, it was assumed that the herbicides would be mainly concentrated in a 5 cm layer (750 t of soil). Saflufenacil was applied in preincubated samples at a rate of 2000 g ha^{-1}, and therefore corresponded to 2.67 μg g soil^{-1}. The herbicide rate was approximately 15 times higher than the maximum recommended amount to be applied in a cropping season (150 g ha^{-1}) according to the Sharpen® registration label. A higher rate of saflufenacil was chosen to allow quantification with the analytical instrumentation employed. Preliminary quality control assurance indicated no detectable saflufenacil in the soil samples.

An analytical standard for saflufenacil (99.5% purity) was provided by BASF Corporation (Research Triangle Park, NC). Stock solution for spiking soils was prepared in HPLC-grade acetonitrile, using the analytical standard, and stored at ∼3 °C. Solution containing saflufenacil (20 μL) was pipetted and thoroughly mixed with the preincubated soil samples, inside the centrifuge tubes, before adding the water treatments. The amount of water added in the field capacity and saturation treatments was calculated according to results described in Table 1 for a 10 g sample size. As all samples were rewetted to bring moisture to 50% of field capacity during the preincubation step, only the remaining amount to

achieve the field capacity and saturation moisture content was added during sample preparation prior to incubation. However, in the saturated treatments, an extra 1.5 mL of water was added to generate an aqueous layer, simulating a flooded rice paddy. Tubes were then loosely capped and incubated at 24.8 ± 0.5 °C in the dark. Moisture content was adjusted twice a week by replenishing the water amount lost, if any, after weighing the preincubated and incubated tubes. Upon experiment initiation, samples were removed at 0, 1, 3, 7, 14, 21, 30 and 45 days. The 0 day samples were used to estimate extraction efficiency. In these samples, herbicide was added and mixed with air-dried soil (without preincubation) 24 h before extraction to allow equilibration.[16] Moisture treatments were applied immediately prior to the extraction. Experimental samples were prepared, starting from the longest incubation time, and the remaining timings were planned such that all samples of one replication were harvested on the same date. Harvested samples were immediately frozen. Replications were staggered over time during the experiment so that replications could be extracted on separate days.

2.4 Soil extraction procedure and analysis

An accelerated solvent extraction (ASE) method was developed, according to Lancaster *et al.*[16], to extract saflufenacil from soil samples. Centrifuge tubes containing samples were removed from the freezer and placed in a water bath (~40 °C) for 5–10 min to initiate the defrosting process. Subsequently, 2 and 4 g of Hydromatrix® (inert diatomaceous earth; Agilent Technologies Inc., Santa Clara, CA) were combined with field capacity and saturated soil sample treatments respectively. Hydromatrix® facilitated the removal of samples from the centrifuge tubes by absorbing the moisture. Incubated samples mixed with Hydromatrix® were then transferred into the ASE extraction cells (22 mL) assembled with a glass fiber filter at the bottom. Empty spaces at the top of the cells were filled with washed sand until the cell volume reached capacity (Ottawa sand; EMD Chemicals Inc., Gibbstown, NJ). The ASE method (ASE 200; Dionex Corporation, Sunnyvale, CA) used acetonitrile solvent for three static extraction cycles of 5 min each at 10.3 MPa cell pressure and 50 °C. The extraction cells were preheated for 2 min before being filled up with acetonitrile. Subsequently, the cell was heated and pressurized for 5 min to achieve thermal equilibrium. Immediately after this, static cycles were initiated, where pressure and temperature were maintained at the desired specifications for 5 min. At the end of each cycle, the cell was partially flushed and fresh solvent corresponding to 60% of the cell volume was introduced. Finally, solvent was purged from the cell by a stream of N_2 gas for 60 s and discharged into the collection vial.

A quantity of 1 g of sodium chloride was added to the collection vial containing the extraction solution. The vial was then manually shaken for 1 min. The organic and aqueous phases were allowed to separate for 15 min.[17] The upper layer (organic, 25–30 mL) was transferred to a graduated tube, while the aqueous layer was transferred to a waste container. The organic phase was evaporated using a water bath temperature of 50 °C and a vortexing force generated by nitrogen gas flowing directly in the sample tube (TurboVap® LV Evaporator; Zymark Center, Hopkinton, MA). Samples were evaporated to concentrate saflufenacil in solution and facilitate quantitative analysis. The final volume was measured in the graduated tube and ranged from 1 to 4 mL, depending on the incubation time. An aliquot of the final volume was removed and placed in an HPLC vial for analysis.

Extractions from soil were analyzed using an HPLC equipped with a photodiode array detector (Waters Corporation, Milford, MA). A method was developed using a Symmetry® C18 analytical column (5 μm, 4.6 mm × 250 mm). An isocratic mobile phase was prepared using 65% acetonitrile, 34.5% deionized water and 0.5% formic acid. The mobile phase was filtered with a 0.45-μm filter and degassed before usage. Samples were analyzed for 10 min using a flow rate of 1 mL min^{-1}. The sample injection volume was 20 μL. The retention time for saflufenacil was 5.95 ± 0.05 min. Samples were analyzed at 271 nm. Calibration standards were prepared in acetonitrile at 1, 2, 5, 10 and 15 μg mL^{-1}. These concentrations encompass the expected range of responses in the final sample aliquots. The R^2 for the calibration curves prepared during the study was above 0.998. Calibration standards were included in every analyzed sample set.

2.5 Carbon dioxide (CO_2) evolution under saturation and field capacity soil moisture

Microbial CO_2 evolution was determined hourly for 30 days by adapting the procedure described previously.[18] Samples (30 g) were preincubated after rewetting the air-dried soil to bring moisture to 50% of field capacity. After 14 days of preincubation, samples were treated with saflufenacil (2.67 μg g $soil^{-1}$) using stock solution and the procedure previously detailed. Soil samples for the field capacity treatments were placed in 50-mL plastic beakers containing holes in the bottom. Soil was added over a filter paper that covered the beaker holes. Beakers were then placed inside a chamber, and the remaining amount of water to achieve the field capacity moisture content was added to each soil. Furthermore, 10 mL of water was added to the chamber to allow bottom-up soil rewetting during the course of the experiment. Samples for the saturation treatments were placed in a glass beaker inside the chamber. Water was added to achieve saturation, based on results in Table 1. Furthermore, 4.5 mL of water was added to each soil to create an aqueous layer simulating a rice field. Samples were incubated at 24.0 ± 0.8 °C. Sealed chambers were coupled with an infrared CO_2 detector (ADC 225MK3; BioScientific Ltd, Great Amwell, England) allowing continuous reading of carbon mineralization.[18]

2.6 Statistical analysis

The studies were conducted as a randomized complete block design. Treatments formed by the combination of soils and moisture contents were repeated in time. Carbon mineralization and saflufenacil degradation studies were conducted using four and five replications respectively. SAS® Enterprise Guide® (v.4.2 software; SAS Institute, Cary, NC) was used to perform the regression analysis. The validity of the models was verified by assessing the normality of the errors and homogeneity of the variance using the software. The concentration of saflufenacil over time was divided by the initial concentration and log transformed before adjustment of the first-order equations. Means of remaining parameters were separated by overlapping 95% confidence intervals.

3 RESULTS AND DISCUSSION

3.1 CO_2 mineralization under saturated and field capacity moisture conditions

Under saturated conditions, CO_2 production was relatively constant throughout the study, where mineralization rates

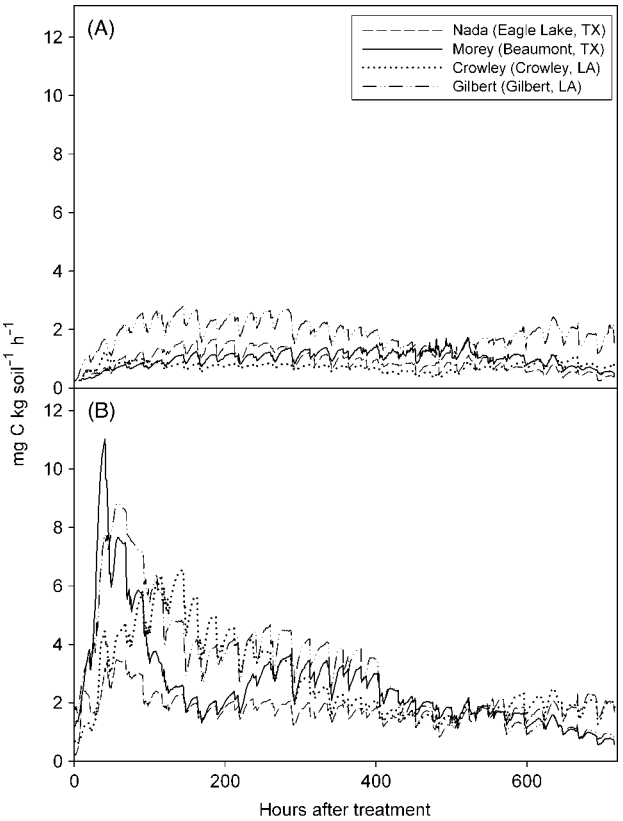

Figure 1. Carbon evolution in rice soils under saturated (A) and field capacity (B) moisture conditions over a period of 716 h (~30 days). Saflufenacil was applied at 2000 g ha^{-1} (2.67 μg g soil^{-1}). Results are the average of four replications.

remained below 3 mg C kg soil^{-1} h^{-1} (Fig. 1A). Hourly carbon mineralization was higher under field capacity conditions for all soils within the initial 500 h after treatment. Microbial activity rapidly increased in the field capacity treatments, indicating that preincubation procedures were effective in reactivating the soil microorganisms. Maximum rates of carbon mineralization were observed within the first 100 h (~4 days) after incubation for all soils, except for the Crowley series where the maximum was observed at approximately 140 h (~6 days) after treatment (Fig. 1B). Maximum mineralization rates ranged from approximately 3.5 to 11 mg C kg soil^{-1} h^{-1}, depending on the soil. Carbon dioxide production returned to basal respiration levels between 425 to 475 h after incubation. In a similar experiment using fluometuron, CO_2 production returned to basal respiration levels after 450 h.[18] Daily cycles of carbon mineralization were observed under both moisture regimes. These cycles can be associated with fluctuations in room temperature that ranged from 23.3 to 24.8 °C.

Carbon dioxide can be produced by various forms of microbial metabolism such as fermentation,[19] anaerobic respiration[20,21] and aerobic respiration.[22] Soil flooding rapidly depletes oxygen from the soil environment by aerobic bacterial consumption and chemical oxidation reactions.[14] Therefore, the microbial community would shift to anaerobic with a prevalence of fermentative bacteria and methanogenic archea microorganisms after flooding.[14] Consequently, under these circumstances, anaerobic respiration and fermentation would potentially lead to production of CO_2 and methane.[14] As fermentation results in partial degradation of organic skeletons, less CO_2 can be generated with this metabolic process. Anaerobic respiration would result in complete oxidation of organic molecules, but it requires the presence of alternative electron acceptors.[19,21,22] Therefore, CO_2 evolution results indicated a shift in microbial population between the moisture treatments. Aerobic respiration would predominate in the field capacity treatments, while anaerobic degradation of organic carbon would be the metabolic process acting under saturated conditions.

Table 2. Saflufenacil recovery from soil samples used to control efficiency of accelerated solvent extraction (ASE) method (50 °C, 10.3 MPa, acetonitrile, three static cycles)

	Soil moisture treatments	
	Field capacity	Saturation
Soil series	% Recovery[a]	
Nada	88 (84–92)	91 (83–99)
Morey	93 (86–100)	90 (84–96)
Crowley	89 (81–97)	94 (88–100)
Gilbert	83 (76–90)	88 (78–98)

[a] Values within parentheses represent 95% confidence intervals of five replications.

3.2 Saflufenacil extraction efficiency

Accelerated solvent extraction (ASE) is a technique that has been successfully used on a wide array of matrices for extraction of organic molecules.[16,23–25] Currently there is no published method in the scientific journals for the extraction of saflufenacil from soil samples. Methodology developed to conduct this study resulted in recovery higher than 80% for a combination of soils and moisture conditions (Table 2). No differences among treatments were observed by overlapping 95% confidence intervals. Therefore, the ASE procedure was determined to be an effective method for extracting saflufenacil from a range of soil matrices with a relatively low volume of solvent.

3.3 Saflufenacil degradation

The overall concentration of saflufenacil decreased more rapidly at field capacity than at saturation. Differences between moisture treatments were observed 7 days after treatment in the Morey and Gilbert soils, and 14 days after incubation in the Crowley soil (Fig. 2). However, differences in saflufenacil concentration persisted only until 21 days after treatment in the Morey series. The only difference observed in the Nada soil was at 30 days after incubation. Therefore, no differences between moisture treatments within any soil were observed at 1 and 3 days after incubation. The maximum rate of microbial activity was observed from 4 to 6 days after incubation when soils were kept at field capacity. Therefore, differences in saflufenacil concentration between soil moisture treatments were perceived after observation of maximum carbon mineralization, indicating the importance of microbial activity on saflufenacil degradation.

Saflufenacil dissipation patterns were subsequently linearized by log transformation to estimate degradation rates. Saflufenacil concentration 1 day after incubation was no different from the concentration at experimental initiation. However, significantly less saflufenacil was observed 3 days after incubation when compared with 0 days (data not shown). Thus, a 1-day period was

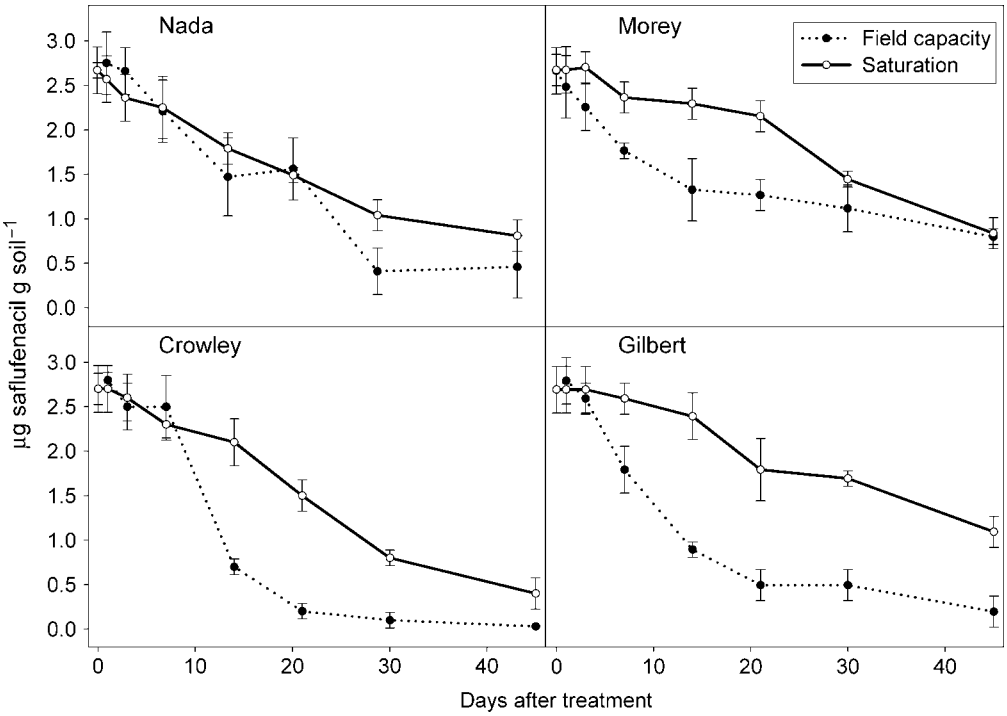

Figure 2. Patterns of saflufenacil degradation under saturation and field capacity moisture conditions for Nada, Morey, Crowley and Gilbert soil series. Saflufenacil was applied at 2000 g ha^{-1} (2.67 µg g soil^{-1}). Bars represent 95% confidence intervals of five replications.

defined as the lag phase. First-order regressions for all soils and moisture treatments were fitted, starting at 1 day after incubation. Saflufenacil degradation was faster at field capacity for all soils, except for the Morey soil (Table 3). Herbicide half-life was 2.1, 2.5 and 3.4 times shorter under field capacity treatments for Nada, Crowley and Gilbert soils respectively. Saflufenacil half-life was similar for moisture treatments in the Morey soil. Half-life averaged among soils was 59 and 33 days for saturated and field capacity treatments respectively.

Pesticides are degraded by biological, chemical and photochemical processes.[13] In these experiments, saflufenacil degradation resulted from chemical and biological degradation as samples were incubated in the dark. Microbiology activity depended on soil moisture, as previously indicated by carbon mineralization results. Predominance of aerobic respiration resulted in faster dissipation of saflufenacil at field capacity in most of the soil series. Anaerobic metabolism resulted in slower degradation of saflufenacil under the saturated treatments. Currently, information regarding saflufenacil degradation and persistence is not available in the scientific journals. However, information available from the Environmental Protection Agency (EPA) website reported that saflufenacil has an aerobic soil half-life ranging from 7 to 35 days (http://nepis.epa.gov/Exe/ZyPDF.cgi?Dockey=P1008R8C.pdf). Results obtained in this study for the field capacity treatments of three of the four soil samples corroborated this statement, as saflufenacil half-life ranged from 15 to 28.6 days. However, the Morey soil had a longer half-life (64 days).

A saflufenacil report available at EPA also indicated that herbicide degradation is slower in acidic to neutral water bodies), as half-life ranged from 28 to 70 days (http://nepis.epa.gov/Exe/ZyPDF.cgi?Dockey=P1008R8C.pdf). Microbial activity in water bodies would be distinct from aerobic soils, but perhaps more similar to a soil flooding condition owing to the limited oxygen availability. Chemical degradation in water bodies seemed to be pH dependent (http://nepis.epa.gov/Exe/ZyPDF.cgi?Dockey=P1008R8C.pdf). In the soil, flooding irrigation resulted in gradual stabilization of pH around the neutral range.[26] Therefore, differences in soil pH among soils would be minimized in the saturation treatments. Degradation patterns of pesticides under simulated rice field conditions were available in the literature, but no alternative moisture treatment was included for comparison.[27,28] In studies considering contrasting moisture conditions (aerobic and anaerobic), half-life results were pesticide dependent. Atrazine and etofenprox degradation followed a similar trend to saflufenacil, as biotransformation was slower under anaerobic conditions.[29,30] For etofenprox, differences between moisture conditions diminished with increasing incubation temperature.[30] However, studies investigating the degradation of parathion and clomazone demonstrated that these pesticides dissipated more rapidly under anaerobic conditions.[31,32]

Microbial degradation of saflufenacil appeared to be the primary degradation mechanism under the controlled conditions of this study. Both aerobic and anaerobic microbial populations encountered in the soil series were able to dissipate saflufenacil relatively rapidly over time. The Crowley series had the shorter half-life among soils in both moisture treatments (Table 3). The Nada, Morey and Crowley series under saturation conditions had the longer half-life, which was similar to the Morey series when moisture was kept at field capacity. Saflufenacil is currently recommended for preplant burndown and pre-emergence in row crops. Under these conditions, herbicide would be applied primarily in aerobic conditions, which would favor dissipation according to results from this laboratory study. In rice, this herbicide is currently recommended only for preplant burndown applications. A supplemental label indicated that saflufenacil must be applied at least 15 days before planting and 45 days prior to establishment of permanent flood

Table 3. First-order rate constant (k), half-life ($t_{1/2}$), 95% confidence limits of saflufenacil and adjusted coefficient of determination (R^2) under saturation and field capacity soil conditions[a]

Soil moisture	Soil series	k ($\lvert\beta_1\rvert$)	$t_{1/2}$ (days)[b]	95% confidence limits (days)[b]	R^2
Saturation	Nada	0.0120	58.8	51.9–68.4	0.86
	Morey	0.0114	61.8	54.3–71.7	0.87
	Crowley	0.0193	36.9	32.3–43.0	0.85
	Gilbert	0.0088	79.7	68.3–94.6	0.83
Field capacity	Nada	0.0251	28.6	22.1–41.0	0.58
	Morey	0.0110	64.0	53.5–80.7	0.74
	Crowley	0.0495	15.0	13.4–17.0	0.89
	Gilbert	0.0314	23.1	19.4–28.5	0.76

[a] Saflufenacil was applied at 2000 g ha^{-1} (2.67 μg^{-1}).
[b] A 1-day lag phase was added to the estimated half-life and 95% confidence limits.

(CDMS: www.cdms.net/LDat/ld99E010.pdf). Therefore, saflufenacil application would be mainly performed with soils under aerobic conditions. In dry-seeded rice production, flooding is established when the crop reaches the 4 to 5-leaf stage.[33] As a result, alternative herbicide usage timings in rice could be considered when managing saflufenacil application before flooding if faster degradation is required to minimize environmental risks. Furthermore, it is important to consider that, under field conditions, moisture content would not be constant after applications of herbicides as in a controlled experimental environment. Drying and wetting cycles that frequently occur under field conditions have been demonstrated to impact upon microbial activity.[34] Hence, this fluctuation in microbial metabolism may result in changes in pesticide dissipation.

4 CONCLUSIONS

Results from this study indicated that carbon mineralization was affected by field capacity and saturated (flooded) moisture conditions. Half-life averaged among soils was 59 and 33 days for saturated and field capacity respectively. Saflufenacil persistence in the environment was 2–3 times longer under flooded conditions for most of the soil series. An effective method for extracting saflufenacil from soil samples was developed for performing experiments using accelerated solvent extraction.

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REFERENCES

1 Grossmann K, Niggeweg R, Christiansen N, Looser R and Ehrhardt T, The herbicide saflufenacil (Kixor™) is a new inhibitor of protoporphyrinogen IX oxidase activity. *Weed Sci* **58**:1–9 (2010).
2 Knezevic SZ, Datta A, Scott J and Charvat LD, Tolerance of winter wheat (*Triticum aestivum* L.) to pre-emergence and post-emergence application of saflufenacil. *Crop Prot* **29**:148–152 (2010).
3 Sikkema PH, Shropshire C and Soltani N, Tolerance of spring barley (*Hordeum vulgare* L.), oats (*Avena sativa* L.) and wheat (*Triticum aestivum* L.) to saflufenacil. *Crop Prot* **27**:1495–1497 (2008).
4 Soltani N, Shropshire C and Sikkema PH, Response of corn to preemergence and postemergence applications of saflufenacil. *Weed Technol* **23**:331–334 (2009).
5 Soltani N, Shropshire C and Sikkema PH, Sensitivity of leguminous crops to saflufenacil. *Weed Technol* **24**:143–146 (2010).
6 Owen LN, Mueller TC, Main CL, Bond J and Steckel LE, Evaluating rates and application timings of saflufenacil for control of glyphosate-resistant horseweed (*Conyza canadenis*) prior to planting no-till cotton. *Weed Technol* **25**:1–5 (2011).
7 Waggoner BS, Mueller TC, Bond JA and Steckel LE, Control of glyphosate-resistant horseweed (*Conyza canadensis*) with saflufenacil tank mixtures in no-till cotton. *Weed Technol* **25**:310–315 (2011).
8 Davis VM, Kruger GR, Young BG and Johnson WG, Fall and spring preplant herbicide applications influence spring emergence of glyphosate-resistant horseweed (*Conyza canadensis*). *Weed Technol* **24**:11–19 (2010).
9 Camargo ER, Senseman SA, McCauley GN and Guice JB, Rice tolerance to saflufenacil in a clomazone weed control program. *Int J Agron* DOI: 10.1155/2011/402461 (2011).
10 Camargo ER, Senseman SA, McCauley GN and Guice JB, Rice (*Oryza sativa* L.) response and weed control from tank-mix applications of saflufenacil and imazethapyr. *Crop Prot* **31**:94–98 (2012).
11 Meier JR, Smith KL, Bullington JA and Doherty RC, Program approaches for hemp sesbania (*Sesbania exaltata*) control in drill-seeded rice. *Proc Rice Tech Work Group* **33**:p149 (2010).
12 Das A and Uchimiya H, Oxygen stress and adaptation of a semi-aquatic plant: rice (*Oryza sativa*). *J Plant Res* **115**:315–320 (2002).
13 Villaverde J, Kahl M and Brown CD, Adsorption and degradation of four acidic herbicides in sons from southern Spain. *Pest Manag Sci* **64**:703–710 (2008).
14 Liesack W, Schnell S and Revsbech NP, Microbiology of flooded rice paddies. *FEMS Microbiol Rev* **24**:625–645 (2000).
15 Lee DJ, Senseman SA, O'Barr JH, Chandler JM, Krutz LJ, McCauley GN *et al.*, Soil characteristics and water potential effects on plant-available clomazone in rice. *Weed Sci* **52**:310–318 (2004).
16 Lancaster S, Senseman S and Carson K, Accelerated solvent extraction of fluometuron from selected soils. *J AOAC Int* **90**:1142–1145 (2007).
17 Schenck FJ, Callery P, Gannett PM, Daft JR and Lehotay SJ, Comparison of magnesium sulfate and sodium sulfate for removal of water from pesticide extracts of foods. *J AOAC Int* **85**:1177–1180 (2002).
18 Lancaster SH, Haney RL, Senseman SA, Kenerley CM and Hons FM, Microbial degradation of fluometuron is influenced by Roundup WeatherMAX. *J Agric Food Chem* **56**:8588–8593 (2008).
19 Dodla SK, Wang JJ, Delaune RD and Breitenbeck G, Carbon gas production under different electron acceptors in a freshwater marsh soil. *Chemosphere* **76**:517–522 (2009).
20 Hong YG and Gu JD, Bacterial anaerobic respiration and electron transfer relevant to the biotransformation of pollutants. *Int Biodeter Biodegr* **63**:973–980 (2009).
21 Lovley DR and Coates JD, Novel forms of anaerobic respiration of environmental relevance. *Curr Opin Microbiol* **3**:252–256 (2000).
22 Dettling MD, Yavitt JB and Zinder SH, Control of organic carbon mineralization by alternative electron acceptors in four peatlands, central New York State, *USA. Wetlands* **26**:917–927 (2006).

23 Ding YJ, Zhang WH, Gu C, Xagoraraki I and Li H, *Determination of pharmaceuticals in biosolids using accelerated solvent extraction and liquid chromatography/tandem mass spectrometry*. *J Chromatogr A* **1218**:10–16 (2011).
24 Gentili A, Perret D, Marchese S, Sergi M, Olmi C and Curini R, Accelerated solvent extraction and confirmatory analysis of sulfonamide residues in raw meat and infant foods by liquid chromatography electrospray tandem mass spectrometry. *J Agric Food Chem* **52**:4614–4624 (2004).
25 Hossain MB, Barry-Ryan C, Martin-Diana AB and Brunton NP, Optimisation of accelerated solvent extraction of antioxidant compounds from rosemary (*Rosmarinus officinalis* L.), marjoram (*Origanum majorana* L.) and oregano (*Origanum vulgare* L.) using response surface methodology. *Food Chem* **126**:339–346 (2011).
26 Savant NK and Kibe MM, Influence of continuous submergence on pH, exchange acidity and pH-dependent acidity in rice soils. *Plant Soil* **35**:205–208 (1971).
27 Jabusch TW and Tjeerdema RS, Microbial degradation of penoxsulam in flooded rice field soils. *J Agric Food Chem* **54**:5962–5967 (2006).
28 Doran G, Eberbach P and Helliwell S, Sorption and degradation of fipronil in flooded anaerobic rice soils. *J Agric Food Chem* **57**:10 296–10 301 (2009).
29 DeLaune RD, Devai I, Mulbah C, Crozier C and Lindau CW, The influence of soil redox conditions on atrazine degradation in wetlands. *Agric Ecosyst Environ* **66**:41–46 (1997).
30 Vasquez ME, Holstege DM and Tjeerdema RS, Aerobic versus anaerobic microbial degradation of etofenprox in a California rice field soil. *J Agric Food Chem* **59**:2486–2492 (2011).
31 Sethunat N and Yoshida T, Parathion degradation in submerged rice soils in Philippines. *J Agric Food Chem* **21**:504–506 (1973).
32 Tomco PL, Holstege DM, Zou W and Tjeerdema RS, Microbial degradation of clomazone under simulated California rice field conditions. *J Agric Food Chem* **58**:3674–3680 (2010).
33 Way MO, *Texas Rice Production Guidelines*. Texas AgriLife Research, Texas AgriLife Extension and United States Department of Agriculture, Beaumont, TX (2010).
34 Chowdhury N, Burns RG and Marschner P, Recovery of soil respiration after drying. *Plant Soil* **348**:269–279 (2011).

