

# Effect of application rate and irrigation on the movement and dissipation of indaziflam

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#### ARTICLE INFO

Article history: Received 29 May 2016 Revised 24 August 2016 Accepted 13 September 2016 Available online 27 September 2016

Keywords: Indaziflam Half-life Sorption Soil properties Dissipation Persistence

#### ABSTRACT

Indaziflam is a new preemergence herbicide for the control of annual grass and broadleaf weeds in various cropping systems including pecan orchards. The objectives of this study were to (1) determine the mobility and dissipation of indaziflam and (2) evaluate herbicide efficacy in a flood-irrigated pecan orchard in southern New Mexico, USA. Indaziflam was applied at 0, 36.5, and 73.1 g/ha in areas with (impacted) and without (unimpacted) tree injury symptoms. Soil samples were collected at 0-15, 15-30, and 30-46 cm depths 26, 63, 90, and 126 days after the first herbicide application. Additional soil samples were collected 4, 30, and 56 days after the second application. Indaziflam was detected in soil samples collected at each depth, suggesting movement with irrigation water. Indaziflam concentrations decreased with increasing soil depth and time. Indaziflam mass recoveries were greater in the unimpacted area than in the impacted area after the first and second applications. Dissipation half-lives of indaziflam in the soil ranged from 30 to 86 days for total indaziflam recovered from the entire soil profile after the first and second applications in both areas. The percent weed control was similar in the impacted and unimpacted areas for both rates of indaziflam on 26 and 63 days after application; however, on 90 days after the application, percent weed control was lower in the impacted than unimpacted area. © 2016 The Research Center for Eco-Environmental Sciences, Chinese Academy of Sciences.

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#### Introduction

Indaziflam is an alkylazine herbicide used for preemergence control of annual grass and broadleaf weeds that inhibits the cellulose biosynthesis of weed species following germination (Alonso et al., 2011). Indaziflam was registered in 2012 for weed control in various agricultural and nonagricultural systems, and limited information is available on the transport and dissipation in soil under field and laboratory conditions. Currently, the literature on the factors that influence the fate and transport of indaziflam has been generated from laboratory studies; therefore, there is a need to evaluate the dissipation of indaziflam under field conditions. In addition, there are no published accounts available on the half-life of indaziflam in the field. The first breakdown product of indaziflam is indaziflam-triazine indanone, which is degraded to indaziflam-carboxylic acid and ultimately to indaziflam-triazinediamine; however, two of the three indaziflam breakdown products (indaziflam-carboxylic acid and indaziflam-triazinediamine) are more mobile than indaziflam (Alonso et al., 2015).

Indaziflam was reported to be low to moderately mobile in six Brazilian oxisols and three U.S. mollisols (Alonso et al., 2011). Similarly, Jhala et al. (2012a) and Jhala and Singh (2012b) reported increased leaching of indaziflam with application

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http://dx.doi.org/10.1016/j.jes.2016.09.002

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rate and amount of rainfall in soil column experiments. Jones et al. (2013a, 2013b) observed a decrease of indaziflam injury to hybrid bermudagrass established in mini-rhizotrons with increasing organic matter content and fraction of fine soil particles. Similarly, Schneider et al. (2015) reported that indaziflam caused phytotoxicity of bermudagrass planted in sandy soil under laboratory conditions decreased with increasing clay and organic matter contents.

Shortly after registration in 2012, indaziflam was extensively used in pecan (Carya illinoinensis (Wangenh.) K. Koch) orchards across the southwestern United States. However, in a few of those orchards, sporadic herbicide injury symptoms were reported 3-4 months after the application date (May 8, 2012). Our previous study in two pecan orchards located in New Mexico and Arizona, where the injury symptoms were detected, indicated a faster dissipation of indaziflam in the New Mexico orchard compared to the Arizona orchard (González-Delgado et al., 2015). Furthermore, the faster rate of dissipation in the New Mexico orchard was attributed to higher sand (77%  $\pm$  7.2%) and lower clay fractions (9%  $\pm$  3.7%) compared to the sand  $(61\% \pm 4.8\%)$  and clay  $(17\% \pm 3.1\%)$ fractions in the Arizona orchard, respectively. Higher sand content in the New Mexico orchard with attendant high soil drainage capacity could have contributed to a faster dissipation of indaziflam compared with the Arizona orchard. This study expects to generate additional information needed to understand the causes of injury to pecan trees that were evaluated by González-Delgado et al. (2015).

We are not aware of studies that have examined the half-life of indaziflam and influence of flood irrigation on the movement of indaziflam under field conditions. Therefore, this field study was conducted in the impacted (injury observed on pecan trees) and unimpacted (no injury observed on pecan trees) areas of the orchard in New Mexico, with the objectives to (1) determine the mobility and dissipation of indaziflam and (2) evaluate herbicide efficacy for two application rates. Indaziflam is classified as low to moderately mobile in the soil (Alonso et al., 2011); therefore, the hypothesis for this study was that indaziflam could move mostly over the soil surface compared to the leaching process after flood irrigations.

#### 1. Materials and methods

#### 1.1. Study site

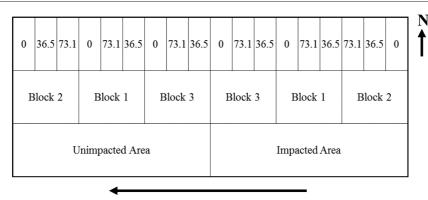
The study site was a pecan orchard located in southern New Mexico, USA (32.412877 N, -106.853516 W) at 1200 m above sea level (González-Delgado et al., 2015). The soil in the orchard is a mixed, thermic Typic Torripsamments with a saturated hydraulic conductivity ranging from  $1.40 \times 10^{-5}$  m/sec to  $4.20 \times 10^{-5}$  m/sec (Soil Survey of Dona Ana County Area, 1980). The orchard was planted with the pecan variety Wichita, which is one of the important commercial varieties adapted to the climate of southern New Mexico and does not require a long growing season (Byford, 2005). A total of 16 cm of precipitation and an average temperature of  $26 \pm 2^{\circ}$ C were recorded between the application day of indaziflam on May 23, 2013 and last day of collecting the soil samples on

November 28, 2013. The orchard was flood irrigated and after the first irrigation using canal water on May 24, 2013, 7 more irrigations were made using well water on June 12, June 29, July 15, Aug. 3, Aug. 26, Sept. 21, and Oct. 6. About 91 cm of total irrigation water was applied. Water flow was from east to west as shown in Fig. 1. Urea nitrogen and ammonium phosphate fertilizers were also applied three times (on April 1, April 24, and June 10).

The orchard was previously treated with indaziflam on May 8, 2012, by the grower, and injury to some pecan trees was observed after July 2012. Injuries to pecan trees were mostly sporadic, and several trees in several rows showed injury symptoms. One of the rows of pecan trees was selected for this study. In this row, four pecan trees suffered extensive damage, and this area was designated as the impacted area. Trees in the contiguous area in the same row but just after the impacted area did not show any injury symptoms; this area was designated as the unimpacted area (Fig. 1). The analysis of soil samples collected from this orchard on March 20, 2013, approximately 11 months after the last application of indaziflam, showed that indaziflam was not detected in 35 out of the 36 soil samples collected from the study site (González-Delgado et al., 2015). Indaziflam was detected only in one soil sample (2.6 µg/kg of indaziflam) collected at 7-15 cm depth from the unimpacted area. Thus no (detectable) indaziflam was present in 0-120 cm depth at the start of this field study on May 23, 2013.

For this study, nine contiguous plots of 6 m  $\times$  4 m were delineated in the unimpacted and impacted areas of the orchard (Fig. 1). This plot arrangement was selected to mimic the herbicide application and transport behavior of indaziflam in the flood-irrigated field with respect to the direction of irrigation water flow. The plots were arranged in the order rate 1 (36.5 g/ha), rate 2 (73.1 g/ha), and rate 0 (control), except in the first block (Block 2) in the impacted area where the control was before the treatment plots with respect to the direction of irrigation water flow (Fig. 1). This was done to evaluate if indaziflam can move backwards or laterally with standing water in the field during irrigation. A split plot experimental design was used with 3 replicates of control (no application) and two rates of indaziflam treatments in each of the impacted and unimpacted area.

Treated plots were sprayed twice during the growing season. During the first application, plots were sprayed with the two application rates of 36.5 and 73.1 g/ha of indaziflam on May 23, 2013 (143 DOY; day of the year). The field was irrigated 24 hr after the indaziflam application. The lower of the two rates applied in May was chosen as a precaution to not cause injury to pecan trees in the orchard. During the second application on October 3, 2013 (276 DOY), indaziflam was sprayed to all the previously treated plots at the rate of 36.5 g/ha. The field was irrigated 72 hr after application. The second indaziflam application was made in October to repeat the field experiment before the experimental site became unavailable. The severely injured pecan trees were removed, and new trees were transplanted in 2014 that caused soil disturbance in the experimental plots. Pecan orchards are managed similarly year after year, and similar irrigation, fertilizer application, and tillage strategies are implemented.



Direction of irrigated water

Fig. 1 – Schematic of plots treated with 0, 36.5 and 73.1 g/ha in the unimpacted and impacted areas of the orchard on May 23, 2013.

#### 1.2. Collection and analysis of soil samples

No specific permissions were required and orchard managers agreed to the collection of soil samples. No endangered or protected species were involved in this study. The volumetric water content at field capacity of sandy loam soil was 0.35 cm<sup>3</sup>/cm<sup>3</sup>. Prior to the irrigation, the average volumetric water content of 0–46 cm soil was about 0.13 cm<sup>3</sup>/cm<sup>3</sup>. Thus 11 cm of irrigation water applied during each irrigation was enough to penetrate to the depth of 48 cm from the soil surface. Since indaziflam is a preemergent herbicide, it is expected to be sorbed in the soil and move to depth less than the wetting front depth of 48 cm. On June 13, 2013 (164 DOY), soil samples were collected with a push probe at three depth intervals (0-15, 15-30, and 30-46 cm depth) from three replicate plots distributed in different blocks at each area (Fig. 1). Soil core samples collected at the same depth interval from replicate plots with the same treatment were composited, air-dried, and passed through a 2 mm sieve to determine soil particle size distribution using the hydrometer method (Table 1) (Gee and Bauder, 1986).

Table 1 – Average particle size distribution and soil
texture of samples collected from the unimpacted and
impacted areas of the pecan orchard treated with 36.5 and
73.1 g/ha of indaziflam in New Mexico, USA.

Area	Depth	Sand	Silt	Clay	Soil
	(cm)	(%) <sup>a,b</sup>	(%) <sup>a,b</sup>	(%) <sup>a,b</sup>	texture
Unimpacted	0–15	76.7a	12.3a	10.9a	Sandy Loam
	15–30	76.5a	13.6a	9.8a	Sandy Loam
	30–46	77.3a	12.3a	10.2a	Sandy Loam
Impacted	0–15	77.4a	12.2a	10.3a	Sandy Loam
	15–30	76.8a	13.1a	10a	Sandy Loam
	30–46	76.9a	12.6a	10.4a	Sandy Loam

 $^{\rm a}\,$  Sample size (n = 7) for % of sand, silt and clay fraction in each of the areas.

 $^{\rm b}\,$  Means within the columns with no common letters are significantly different based on the least significant difference (LSD) test,  $p\mbox{-value}\xspace<0.05.$ 

A soil sample for indaziflam analysis was collected from each plot 26 (170 DOY), 63 (207 DOY), 90 (234 DOY), and 126 (270 DOY) days after treatment (DAT) between June 19 (170 DOY) and September 27, 2013 (270 DOY). Indaziflam was not expected to move below a 30 cm depth during the first irrigation; therefore, soil samples were not collected at 30–46 cm depth of plots 26 DAT (170 DOY). Additional soil samples were collected from plots 4 (280 DOY), 30 (306 DOY), and 56 (332 DOY) days after the second indaziflam application on October 3, 2013 (276 DOY). No further soil samples could be collected because of the soil disturbance during planting of new trees in the impacted area.

Soil samples collected at the same depth intervals from replicated plots for each treatment were composited separately. The concentration of indaziflam was determined by Bayer CropScience laboratory (Research Triangle Park, North Carolina). A sample aliquot was amended with an isotopic standard of indaziflam and diluted with deionized water prior to the analysis. An acetonitrile:water (80:20, V/V) solution was added to the soil aliquot to determine the concentration of indaziflam using the microwave assisted extraction method. Samples were analyzed by tandem mass spectrometry liquid chromatography tandem mass spectrometry (LC/MS/MS) with quantification based on the use of internal standards and comparison of peak areas to those of known standards. The detection limit for indaziflam was 0.2 µg/kg. Analysis of means was conducted using the Proc Mix procedure to determine the significant difference of indaziflam concentrations among treatments and depths by the date of sampling using SAS 9.2 (SAS Institute Inc., Cary, NC, USA). Means were separated statistically using Fisher's Protected LSD when the F-test indicated a significance at p = 0.05.

The total potential mass of indaziflam in the soil ( $C_0$ ;  $\mu g/kg$ ) after a single application was calculated for a given depth using Eq. (1) as:

$$C_0 = (A/(BD \times d) \times CF1$$
(1)

where, A (g/cm<sup>2</sup>) is the application rate, BD (g/cm<sup>3</sup>) is soil bulk density, *d* (cm) is soil depth, and CF1 ( $1 \times 10^9 \,\mu$ g/kg) is a conversion factor (Cycoñ et al., 2005). The mass of indaziflam in the soil from Eq. (1) was used to calculate mass recoveries in

the unimpacted and impacted areas separately. The first-order dissipation coefficient (k; day<sup>-1</sup>) was obtained by plotting indaziflam concentration remaining in the soil ( $C_t$ ;  $\mu$ g/kg) and time (t; day) as follows:

$$C_t = C_0 \times e^{-kt} \tag{2}$$

The dissipation half-lives  $(t_{1/2})$  of the indaziflam in the soil were determined using the following Eq. (3) (Kah et al., 2007):

$$t_{1/2} = Ln2/k \tag{3}$$

No information is available on the dissipation half-life of indaziflam under field conditions; therefore, this study aimed to evaluate the dissipation half-life of indaziflam under field conditions. The dissipation coefficient was calculated from indaziflam concentration separately by depth (0–15, 15–30, and 30–46 cm depth) to evaluate whether the dissipation coefficient varies with soil depth. The dissipation half-life was calculated from the recovered indaziflam in the total soil depth (0–46 cm depth) (Rice et al., 2002).

#### 1.3. Evaluation of herbicide efficacy

Herbicide efficacy (percent weed control) was evaluated visually on 26, 63, and 90 days after the first indaziflam application, and scored as percent control of total grass and broadleaf weed species compared to the untreated control plots. The predominant annual weed species in plots included junglerice (Echinochloa colonum L.), barnyardgrass (Echinochloa crus-galli (L.) Beauv.), red sprangletop (Leptochloa uninervia (Presl) Hitch. and Chase), feather fingergrass (Chloris virgata), and palmer amaranth (Amaranthus palmeri). Data from the weed control evaluations were subjected to a multiple comparison test of all treatment means using Tukey's adjustment.

#### 2. Results and discussion

#### 2.1. Indaziflam concentration and percent mass recovery

Indaziflam was detected in soil samples collected at each of the 0–15, 15–30, and 30–46 cm depths from the impacted and unimpacted areas after the first application on May 23, 2013, with 0, 36.5, and 73.1 g/ha. As expected, the indaziflam concentrations decreased with increasing time in both areas due to degradation and probably leaching (Fig. 2a–c). Concentrations at 0–15 and 15–30 cm depths of the unimpacted area treated with 36.5 and 73.1 g/ha were significantly different (p < 0.03 and p < 0.0001, respectively). Indaziflam concentrations in soil samples collected at 0–15 and 15–30 cm depths of the impacted area treated with 73.1 g/ha were significantly different (p < 0.006) for samples collected 63 (207 DOY) days after treatment. Generally, concentrations at 15–30 and 30–46 cm depths in both areas treated with 36.5 and 73.1 g/ha were not significantly different.

Percent mass recoveries of indaziflam were mostly higher in the unimpacted area than in the impacted area (Table 2). One potential explanation for greater percent mass recoveries in the unimpacted area is greater percentage of soil organic matter. Schneider et al. (2015) reported increased sorption of indaziflam

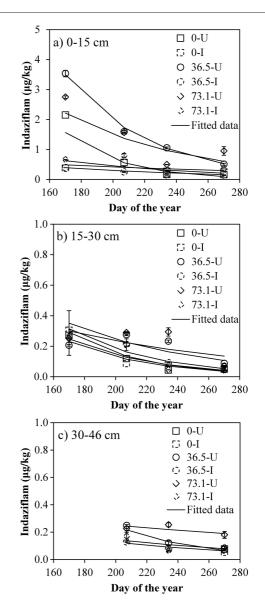


Fig. 2 – Concentration of indaziflam over time at (a) 0–15, (b) 15–30 and (c) 30–46 cm depth in unimpacted (U) and impacted (I) areas treated with 0, 36.5 and 73.1 g/ha. Solid lines are best-fit curves and error bars represent standard error. Standard error values of concentrations at (a) 0–15 cm depth ranged from 0.11 to 0.005, (b) 15–30 cm depth ranged from 0.14 to 0.001 and (c) 30–46 cm depth ranged from 0.02 to  $2.89 \times 10^{-5}$  in both areas treated with 36.5 and 73.1 g/ha.

with increasing organic matter content in a laboratory study on indaziflam phytotoxicity on bermudagrass. Higher amounts of bound metolachlor residues were observed in a surface soil with an organic matter content of 1.5% than in a subsurface soil with an organic matter content of 0.2% (Rice et al., 2002). Similarly, greater sorption of atrazine and terbuthylazine was reported in soils with higher organic matter content (1.2% versus 0.9%) in laboratory experiments (Stipicevic et al., 2015). Rouchaud et al. (1993) reported that the persistence of isoxaben herbicide increased with increasing organic matter content in the field. The average organic matter content (0.65%  $\pm$  0.04%) in the unimpacted area was higher and significantly different

## Table 2 – Percent mass recoveries of indaziflam at different depths in unimpacted and impacted areas treated with 0, 36.5 and 73.1 g/ha.

Depth	DAT <sup>d</sup>	Unimpacted area		Impacted area				
		0 g/ha <sup>b,c,f</sup>	36.5 g/ha <sup>a,f</sup>	73.1 g/ha <sup>a,f</sup>	0 g/ha <sup>b,c,f</sup>	36.5 g/ha <sup>a,f</sup>	73.1 g/ha <sup>a,f</sup>	
0-15 cm	26 <sup>e</sup>	6.4%	20.6%a	8%b	1.1%	2.3%c	1.9%c	
	63 <sup>e</sup>	1.6%	9.1%a	4.5%b	1.5%	1.4%c	2.3%c	
	90 <sup>e</sup>	0.47%	6.1%a	1.4%b	0.78%	1.7%b	0.36%c	
	126 <sup>e</sup>	0.46%	2.9%a	2.7%a	0.72%	0.84%b	1%b	
15-30 cm	26 <sup>e</sup>	0.79%	1.2%a	0.74%a	1.8%	1.6%a	0.73%a	
	63 <sup>e</sup>	0.34%	1.6%a	0.84%bc	0.25%	1.2%ab	0.61%c	
	90 <sup>e</sup>	0.13%	1.3%a	0.86%b	0.13%	0.53%c	0.13%d	
	126 <sup>e</sup>	0.14%	0.51%a	0.2%bc	0.14%	0.31%b	0.11%c	
30-46 cm	26 <sup>e</sup>	NS	NS	NS	NS	NS	NS	
	63 <sup>e</sup>	NS	1.4%a	0.66%b	NS	0.75%b	0.50%b	
	90 <sup>e</sup>	NS	0.70%a	0.74%a	NS	0.45%ab	0.19%b	
	126 <sup>e</sup>	0.22%	0.44%ab	0.52%a	0.16%	0.39%ab	0.26%b	

<sup>a</sup> Sample size (n = 2) for the treatments (36.5 and 73.1 g/ha) at each depth.

 $^{\rm b}\,$  Sample size (n = 1) for the control (0 g/ha) at each depth.

<sup>c</sup> Mass recoveries for control areas were calculated based on the application rate of 73.1 g/ha.

<sup>d</sup> 26, 63, 90 and 126 DAT (days after treatment) are equivalent to 170, 207, 234 and 270 DOY (day of the year), respectively.

<sup>e</sup> Means within the rows with no common letters are significantly different based on the least significant difference (LSD) test, *p*-value <0.05.

<sup>f</sup> NS (not sampled).

(p < 0.05) than the average organic matter content (0.53% ± 0.04%) in the impacted area (González-Delgado et al., 2015). Due to the small difference in organic matter content, sorption capacity of the soil in both areas might not vary as much as for soils with a wider range of organic matter contents. However, this small difference could influence the distribution of soil microorganisms involved in the biodegradation process (Di et al., 1998).

A second potential explanation of lower concentration of indaziflam in the impacted area was the result of indaziflam migration from the impacted area during the lateral movement of irrigation water. Indaziflam was detected in control samples where no application was made at each depth. This indicated that indaziflam moved with irrigation water. After the flood irrigation, water was ponded in the study site for 3 to 4 hr. Indaziflam was also detected in the control plot of block 2 in the impacted area at 0-15 and 15-30 cm depths 26 (170 DOY) days after the first application, although it was located before the treated plots (Fig. 1). Indaziflam likely moved as result of the diffusion and likely subsurface lateral transport from the treated plots to the first control plot in the impacted area. Generally, indaziflam concentrations in control samples from both areas were below 0.6  $\mu$ g/kg, except one sample at 0–15 cm depth of the unimpacted area (2.2  $\mu$ g/kg) 26 DAT (170 DOY). Soil samples were not collected beyond 46 cm depth.

Indaziflam concentrations for samples collected 26 DAT (170 DOY) from control plots in impacted and unimpacted areas were significantly different (p < 0.0004). No significant differences were observed for control samples collected 63 to 126 DAT (207 to 270 DOY).

The results for the second application using the lower of the two rates from the first applications were similar to the first applications. Concentrations from the unimpacted area treated with 0 and 36.5 g/ha were greater than those from the impacted area (Fig. 3). The concentrations from both areas treated the second time with 36.5 g/ha were greater than the concentrations obtained from the same treatments during the first application (May 23, 2013). The greater concentrations observed after the second application could be explained by the longer residence time of indaziflam on the soil surface before irrigation.

The percentage mass recoveries of indaziflam from 0 to 46 cm depth were greater in the unimpacted area than in the impacted area after the second application on October 3. Percentage mass recoveries from the unimpacted and impacted areas treated the second time with 36.5 g/ha were significantly different 4, 30, and 56 DAT (280, 306, and 332 DOY) (Table 3). The results from the second application confirm that indaziflam moved with the irrigated water from the impacted to the unimpacted area. Generally, percent mass recoveries were greater in plots treated with the lower application rate during first application. This could be the result of enhanced microbial activity and vertical and lateral movement of indaziflam in the

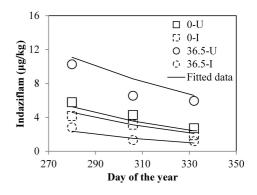


Fig. 3 – Total mass of indaziflam in soil recovered at each soil sampling from 0 to 46 cm depth of the unimpacted (U) and impacted (I) areas treated the second time with 0 and 36.5 g/ha. Solid lines are best-fit curves.

Table 3 – Percent mass recoveries of indaziflam from 0 to 46 cm depth of the unimpacted and impacted areas treated the second time with 0 and 36.5 g/ha.

DAT <sup>d</sup>	Unimpacted area	Impacted area		
	0 g/ha <sup>b,c</sup>	36.5 g/ha <sup>a,e</sup>	0 g/ha <sup>b,c</sup>	36.5 g/ha <sup>a,e</sup>
4	16.8%	59.7%a	12.1%	16.2%b
30	12.5%	38.2%a	9.1%	7.6%b
56	7.9%	34.6%a	5.5%	6.8%b

<sup>a</sup> Sample size (n = 2) for each of the areas treated with 36.5 g/ha of indaziflam.

<sup>b</sup> Sample size (n = 1) for the control (0 g/ha) in each area.

<sup>c</sup> Mass recoveries for control areas were calculated based on the application rate of 73.1 g/ha.

 $^{\rm d}$  4, 30 and 56 DAT (days after treatment) are equivalent to 280, 306 and 332 DOY (day of the year), respectively.

 $^{\rm e}$  Means within the rows with no common letters are significantly different based on the least significant difference (LSD) test,  $p\mbox{-value}\xspace<0.05.$ 

soil with increasing concentration. Indaziflam is a weak acid herbicide that is anionic at soil pH of 5.4 and above (Alonso et al., 2011), and more mobile than nonionic herbicides (e.g., metolachlor and oxyfluorfen). Previous studies reported that anionic herbicides (simazine and terbacil) were more mobile than nonionic herbicides (oxyfluorfen and diuron) (Hodges and Talbert, 1990; Milanova and Grigorov, 1996). Another study reported that indaziflam and simazine showed similar lateral movement potential and greater lateral movement potential than other preemergent herbicides used to evaluate the influence of a simulated storm event in a field experiment (Leon et al., 2016). Anionic compounds move rapidly through the soil profile because they are weakly retained by soil particles as a result of anion exclusion (Celis et al., 2005). Anion exclusion is the repulsion of negatively charged ions from soil particles with a net negative charge (González-Delgado and Shukla, 2011). A shorter persistence of terbuthylazine in the soil was observed at an application rate three times greater than the registered rate (1.0-1.5 kg/ha) due to increased microbial activity (Stipicevic et al., 2015). Similar results were obtained by Singh et al. (1990) for s-triazines and thiocarbamates. Dinelli et al. (1998) and Busse et al. (2001) also reported an increase in microbial activity with increasing application rates of weak acid herbicides (triasulfuron, primisulfuron methyl, rimsulfuron, and glyphosate). In contrast, some studies have reported greater persistence of herbicides with increasing application rates due to suppressed microbial activity (Fuscaldo et al., 1999; Long et al., 2014). Indaziflam is a new herbicide, and to the best of our knowledge there are no studies that examined the effect of indaziflam concentration on soil microbial activity at field scale.

Lower percent mass recoveries in plots treated with the higher application rate could also be due to the greater leaching. Some studies have reported that higher application rates of herbicides contributed to greater leaching (Hunter and Stobbe, 1972; Kotoula-Syka et al., 1993; Sondhia, 2009). The sorption of metolachlor is reported to decrease with increasing concentration as a result of multilayer adsorption with lower binding energy, which lead to greater quantity of metolachlor in the soil solution available for leaching and microbial degradation (Rice et al., 2002; Obrigawitch et al., 1981). Similar results were obtained by Horowitz and Elmore (1991) that observed a strong adsorption of oxyfluorfen to the soil; however, there were not enough adsorbing sites to bind an increased herbicide dose. These studies agree with Jhala et al. (2012a, 2012b) that reported greater leaching of indaziflam with increasing application rate and amount of simulated rainfall in a soil column study under greenhouse conditions.

#### 2.2. Dissipation of indaziflam

Indaziflam content in the soil showed an exponential decrease over time in most of the treatments in both the unimpacted and impacted areas (Fig. 2). Dissipation coefficients (k) were obtained from best fit equations using indaziflam concentrations by depth (Table 4). The dissipation of indaziflam at 30-46 cm depth was expected to be slower due to the decreasing microbial activity and organic matter content with depth. Previous studies also reported that the persistence of acetochlor and metolachlor herbicides increased with increasing depth (Rice et al., 2002; Oliveira et al., 2013). However, a dissipation coefficient value of 0.019 day<sup>-1</sup> at 30–46 cm depth in the unimpacted area treated with 36.5 g/ha was similar to those from upper soil layers (0-15 and 15-30 cm depth). At 15-30 cm depth, dissipation coefficient values of 0.018 day<sup>-1</sup> for 0 g/ha in the unimpacted area, 0.018 and 0.021 day<sup>-1</sup> for 36.5 and 73.1 g/ha in the impacted area, respectively, were larger than those at 0–15 cm depth (Table 4). Dissipation coefficients of herbicides generally decrease with soil depth, but there are reports of increasing dissipation coefficients with soil depth. Higher rates of dissipation with increasing depth could be the result of changes in soil microbial activities in different soil layers (Di et al., 1998; Karpouzas et al., 2011).

Table 4 – Dissipation coefficient (k) values at different depths from unimpacted and impacted areas treated with different application rates (0, 36.5 and 73.1 g/ha) of indaziflam on May 23, 2013.

Area	Depth (cm)	Treatment (g/ha)	k (day <sup>-1</sup> )	r²	
Unimpacted	0–15	0	0.028	88	
		36.5	0.019	99	
		73.1	0.013	55	
	15–30	0	0.018	83	
		36.5	0.008	48	
		73.1	0.012	52	
	30–46	0	NS <sup>d</sup>		
		36.5	0.019	96	
		73.1	0.004	51	
Impacted	0–15	0	0.005	42	
		36.5	0.009	78	
		73.1	0.011	28	
	15–30	0	0.019	82	
		36.5	0.018	95	
		73.1	0.021	83	
	30–46	0	NS <sup>d</sup>		
		36.5	0.01	84	
		73.1	0.009	34	
NS: not sampled.					

## 2.3. Dissipation half-life of total indaziflam recovered from the soil profile

The initial indaziflam concentrations in the soil for the rates of 36.5 g/ha and 73.1 g/ha using Eq. (1) were 17.14 and 34.28  $\mu$ g/kg, respectively. The total masses of indaziflam recovered from the unimpacted area on 26 DAT (170 DOY) with 36.5 and 73.1 g/ha were 3.74 and 3.0  $\mu$ g/kg, respectively (Fig. 4). The masses of indaziflam recovered from the impacted area on 26 DAT with 36.5 and 73.1 g/ha were 0.68  $\mu$ g/kg and 0.91  $\mu$ g/kg, respectively. The mass of indaziflam recovered from soil from both the impacted and unimpacted areas indicated that a large amount of applied indaziflam moved with irrigation water during the first irrigation and supported our hypothesis. The total masses of indaziflam recovered 63 DAT (207 DOY) with 36.5 and 73.1 g/ha were 2.12 and 2.08  $\mu$ g/kg, respectively, from the unimpacted area, and 0.59 and 1.18  $\mu$ g/kg, respectively, from the impacted area.

The total mass of indaziflam recovered from the impacted and unimpacted areas at each sampling time was plotted against the day since application, and Eq. (2) was fitted to the data. The dissipation half-life was calculated using the slope from Eqs. (2) and (3) (Table 5), separately by application rate and areas. The dissipation half-lives for the total mass of indaziflam recovered from the unimpacted area ranged from 30 to 69 days, while those in the impacted area were within the range of 69 to 86 days for the first application. However, for the second application indaziflam half-lives in the unimpacted area ranged from 46 to 63 days and from 41 to 43 days in the impacted area (Table 6). These half-lives were within the half-life range of 30–86 days in the entire soil profile during the first application.

Most of the half-lives obtained in this field study are much smaller than 150 days reported by Environmental Protection Agency (EPA) in the laboratory (US EPA, 2010). This difference suggests that indaziflam dissipation is probably influenced by soil type, experimental and environmental conditions, and movement of herbicide with irrigation. Shaner and Henry (2007) have reported faster dissipation of metolachlor in the field than in the laboratory due to greater microbial degradation and volatilization from the soil under field conditions.

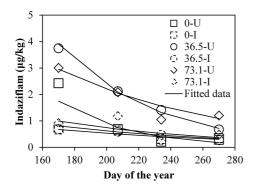


Fig. 4 – Total mass of indaziflam in soil recovered at each soil sampling from 0 to 46 cm depth of the unimpacted (U) and impacted (I) areas treated the first time with 0, 36.5 and 73.1 g/ha. Solid lines are best-fit curves.

Table 5 – Dissipation coefficient (k) and half-life  $(t_{1/2})$  of total indaziflam recovered from a depth of 0–46 cm for each treatment in unimpacted and impacted areas with <u>coefficient of determination (r<sup>2</sup>) ranging from 35% to 99%.</u>

Area	Depth (cm)	Treatment (g/ha)	k (day <sup>-1</sup> )	t <sub>1/2</sub> (day)	r <sup>2</sup>
Unimpacted	0–46	0 36.5 73.1	0.023 0.017 0.010	30 41 69	78 99 79
Impacted	0–46	0 36.5 73.1	0.008 0.009 0.010	86 77 69	71 91 35

However, half-lives in this study could be under-predicted because of the movement of indaziflam with irrigation water within and outside the study site. This study is the first to evaluate the dissipation of indaziflam under field conditions and confirmed that indaziflam is mobile with irrigation water. The dissipation of indaziflam was faster than expected in both areas, probably as result of lateral transport with irrigation water beyond the study site. However, to quantify how much indaziflam can move, future studies should evaluate the leaching of indaziflam under greenhouse and field conditions.

#### 2.4. Evaluation of herbicide efficacy

Over the course of this study, 26 (170 DOY) and 63 (207 DOY) days after indaziflam application, the percent weed control was similar in the impacted and unimpacted areas for both grasses and broadleaf weeds for both application rates. Ninety (234 DOY) days after the application, percent weed control was not significantly different for grasses/weeds in the unimpacted area, but it was significantly lower in the impacted area (Fig. 5a and b). These results disagree with the results of a previous study in southern New Mexico that showed season-long weed control in a pecan orchard with application of indaziflam at 73.1 g/ha (Mohseni Moghadam et al., 2012). The reduction in the percent weed control of indaziflam during the course of the study (i.e., 90 days after the first application) is an indication of indaziflam dissipation from the soil surface, especially in the impacted area. These results are in agreement with the higher concentration of indaziflam in the unimpacted area compared to the impacted area.

Table 6 – Dissipation coefficient (k), half-life $(t_{1/2})$ of total indaziflam recovered from a depth of 0–46 cm and
coefficient of determination $(r^2)$ ranging from 81% to 99% for unimpacted and impacted areas treated the second time with 0 and 36.5 g/ha.

Area	Depth (cm)	Treatment (g/ha)	k (day <sup>-1</sup> )	t <sub>1/2</sub> (day)	r <sup>2</sup>
Unimpacted	0–46	0	0.015	46	99
		36.5	0.011	63	86
Impacted	0–46	0	0.016	43	98
		36.5	0.017	41	81

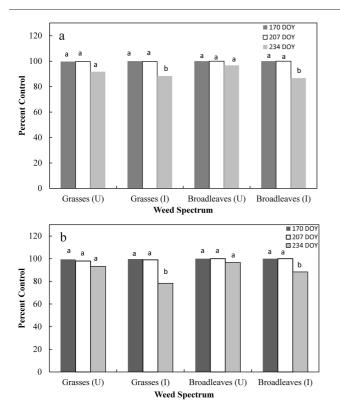


Fig. 5 – Percent grass and broadleaf weed control, compared to untreated control plots, at 26 (170 DOY), 63 (207 DOY) and 90 (234 DOY) days after the first application of indaziflam at (a) 73.1 and (b) 36.5 g/ha in impacted (I) and unimpacted (U) areas the pecan orchard in this study. DOY: day of the year.

#### 3. Conclusions

Indaziflam was detected in soil samples collected at each depth of the impacted and unimpacted areas treated after each application, indicating that indaziflam moved with water. Indaziflam concentrations were greater in the unimpacted area than the impacted area, in agreement with greater organic matter content of the unimpacted area after the first and second applications. Mass recoveries were greater in areas treated with 36.5 g/ha than those treated with 73.1 g/ha after the first application. Dissipation half-life values for total indaziflam recovered from soil samples collected at 0-46 cm depth ranged from 30 to 86 days. The reduced percent weed control of indaziflam in the impacted area also supported indaziflam dissipation from the soil surface. This is the first study that has evaluated the movement and dissipation half-life of indaziflam in a flood irrigated pecan orchard and it provides the needed baseline information for future studies. Further research is needed to quantify the likely saturation of the available sorption sites and stimulation of microbial activity with increasing indaziflam concentration.

#### Acknowledgments

Authors thank Bayer CropScience, Research Triangle Park, North Carolina, USA, for providing analytical data of indaziflam.

This work was supported by grants from Bayer CropScience. Authors also thank Nakayama endowment, New Mexico State University Agricultural Experiment Station, owners of the pecan orchard for support, and Mr. Frank Sholedice for editing the manuscript.

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