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Dissipation of Pendimethalin in Organic Soils in Florida

Dennis C. Odero and Dale L. Shaner*

Understanding the persistence of PRE-applied pendimethalin is important in determining timing of subsequent weed management programs in sugarcane on organic soils in the Everglades Agricultural Area (EAA). Dissipation of oil- and water-based pendimethalin formulations applied PRE at 2, 4, and 8 kg ai ha^{-1} were compared in 2011 and 2012 on organic soils in the EAA. The rate of dissipation of both formulations was very similar. Both formulations had an initial rapid rate of dissipation followed by a slower rate of dissipation. However, the initial amount of pendimethalin in the soil was higher with the water-based compared to the oil-based formulation, most likely because of the lower volatility of the water-based formulation. The half-lives (DT_{50}s) of the oil-based formulation were 32, 18, and 10 d and 8, 8, and 12 d at 2, 4, and 8 kg ha^{-1}, respectively, in 2011 and 2012, respectively. The DT_{50}s of the water-based formulation were 20, 13, and 10 d and 12, 12, and 14 d at 2, 4, and 8 kg ha^{-1}, respectively in 2011 and 2012, respectively. These DT_{50} values were attributed to low soil water content as well as the absence of incorporation following application. Our results suggest that dissipation of pendimethalin is rapid on organic soils irrespective of the formulation when applied under dry soil conditions with no incorporation into the soil.

Nomenclature: Pendimethalin; sugarcane, *Saccharum* spp. hybrids.
Key words: Field dissipation, organic soil.

Entender la persistencia de aplicaciones PRE de pendimethalin es importante para determinar el momento de implementación de programas de manejo de malezas subsiguientes en caña de azúcar en suelos orgánicos en el Área Agrícola de los Everglades (EAA). En 2011 y 2012, se comparó la disipación de formulaciones de pendimethalin a base de aceite y de agua aplicadas PRE a 2, 4, y 8 kg ai ha^{-1} en suelos orgánicos en el EAA. La tasa de disipación de ambas formulaciones fue muy similar. Ambas formulaciones tuvieron una tasa inicial de disipación rápida seguida de una tasa de disipación más lenta. Sin embargo, la cantidad inicial de pendimethalin en el suelo fue mayor con la formulación a base de agua en comparación con la formulación a base de aceite. Esto es probable que se debiera a la baja volatilidad de la formulación a base de agua. Las vidas medias (DT_{50}s) de la formulación a base de aceite fueron 32, 18, y 10 d y 8, 8, y 12 d a 2, 4, y 8 kg ha^{-1}, en 2011 y 2012, respectivamente. Las DT_{50}s de la formulación a base de agua fueron 20, 13, y 10 d y 12, 12, y 14 d a 2, 4, y 8 kg ha^{-1}, en 2011 y 2012, respectivamente. Estos valores de DT_{50}s fueron atribuidos al bajo nivel de humedad de suelo y a la ausencia de incorporación después de la aplicación. Nuestros resultados sugieren que la disipación de pendimethalin es rápida en suelos orgánicos independientemente de la formulación cuando se aplica sin incorporación a suelos en condiciones secas.

Sugarcane is cultivated on 163,000 ha in Florida, making the state one of the largest producers of cane sugar in the United States (U.S. Department of Agriculture–National Agricultural Statistics Service [USDA-NASS] 2012). Approximately 82% of sugarcane in Florida is on high organic matter Histosols of the Everglades Agricultural Area (EAA). Efficacious weed control in these organic (muck) soils is essential for profitable sugarcane production. In Florida, growers rely on PRE herbicides to provide early-season weed control at sugarcane planting (August to January) and following harvest (October to March) of the ratoon crop. Pendimethalin is a dinitroaniline herbicide applied PRE for selective control of many annual grasses in sugarcane (Anonymous 2012; Judice et al. 2006; Millhollon 1993; Richard 1997; Viator et al. 2002). It is applied in combination with triazine herbicides to broaden the spectrum of weed control in sugarcane (Jones and Griffin 2009; Judice et al. 2006; Smith et al. 2008; Viator et al. 2002).

Pendimethalin has low water solubility (0.275 mg L^{-1}) and strong adsorption to soil colloids and organic matter (K_{oc} = 17,200 L kg^{-1}) (Senseman 2007). These characteristics are attributed to its

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* Assistant Professor, University of Florida, Everglades Research and Education Center, Belle Glade, FL 33430; Plant Physiologist, Water Management Research, Agricultural Research Service, USDA, Fort Collins, CO 80526. Corresponding author's E-mail: dcodero@ufl.edu

high potential for hydrogen bonding in particular to organic, lipophilic, and proteinaceous substances (Weber 1990). Therefore, the use rate of pendimethalin increases with increasing soil organic matter because of these physical properties. Pendimethalin has low mobility and will persist in soil because of its low leaching potential, attributed to its ability to strongly adsorb to soil colloids and organic matter. As a result, it typically remains in the top 10 cm of the soil profile following PRE application (Alister et al. 2009; Chopra et al. 2010; Lin et al. 2007; Simmons and Derr 2007; Smith et al. 1995). Furthermore, pendimethalin has low volatility (vapor pressure $= 1.25 \times 10^{-3}$ Pa), and thus is less prone to volatilization compared to other dinitroanilines (Parochetti and Dec 1978).

Pendimethalin is degraded primarily by soil microorganisms (Weber 1990) and its persistence is influenced by edaphic and climatic conditions (Barrett and Lavy 1983; Kennedy and Talbert 1977; Lee et al. 2000; Weber 1990). Degradation is faster under anaerobic, warm, and moist conditions (Barrett and Lavy 1983; Savage 1978; Senseman 2007; Walker and Bond 1977; Weber 1990; Zimdahl et al. 1984). However, Kulshrestha et al. (2000) reported enhanced degradation by adapted aerobic microorganisms within the top 15 cm of the soil. The half-life or the time it takes to 50% dissipation (DT_{50}) of pendimethalin ranges from 42 to 101 d under laboratory conditions depending on soil moisture and temperature (Senseman 2007). The average DT_{50} of pendimethalin in the field is 44 d, but will vary with the prevailing soil temperature and moisture conditions (Senseman 2007).

The bioavailability of dinitroaniline herbicides has been shown to decrease with increase in soil organic matter content (Weber 1990). Currently, no data are available on bioavailability and persistence of pendimethalin applied PRE for weed control in sugarcane on organic soils (up to 85% organic matter) of the EAA. Therefore, a study was conducted to determine dissipation of oil- and water-based formulations of pendimethalin on organic soils of the EAA.

Materials and Methods

Study Location. Field studies were conducted in Belle Glade, FL at the Sugarcane Growers Cooperative of Florida (SGCF) Farm and the Everglades Research and Education Center (EREC) in 2011 and 2012, respectively, to evaluate field dissipation of pendimethalin. The soil type at both locations was Dania Muck (Euic, hyperthermic, shallow Lithic Haplosaprists) with pH of 7.1 and 6.6 and organic matter of 68.1 and 73.9% at SCGCF and EREC, respectively. The experiment was arranged in a randomized complete block design with three (2011) or four (2012) replications of treatments.

Herbicide Treatments and Application. Herbicide treatments consisted of oil- (Prowl 3.3 EC, BASF Corporation, Research Triangle Park, NC 27709) and water-based (Prowl H_2O, BASF Corporation, Research Triangle Park, NC 27709) pendimethalin formulations applied PRE on February 1, 2011 and February 27, 2012 on new plant cane fields. Each formulation was applied at 2, 4, and 8 kg ha^{-1} plus a nontreated control. Plots consisted of four sugarcane rows 1.5 m apart and 12 m long in 2011 and two sugarcane rows 1.5 m apart and 7.6 m long in 2012. Herbicides were applied in 2011 with a tractor-mounted sprayer (Lee Spider, LeeAgra Inc., Lubbock, TX 79404) delivering 180 L ha^{-1} at 276 kPa with Teejet® TT11002 nozzle tips (Spraying Systems Co., Wheaton, IL 60187). In 2012, herbicides were applied with a CO_2-pressurized hand sprayer delivering 180 L ha^{-1} at 276 kPa with Teejet® TT11002 nozzle tips. Pendimethalin is surface applied by sugarcane growers in the EAA and is not incorporated into the soil using any equipment. Weather data (temperature, rainfall, and solar radiation) recorded near the experimental areas are provided in Figures 1 and 2.

Soil Sampling, Pendimethalin Extraction, and Analysis. Soil samples were taken at 7, 14, 21, 28, 35, 42, 49, and 56 d after treatment (DAT) in 2011 and 2012. Four soil cores 3 cm in diameter and 0 to 10 cm in depth were taken randomly from each plot at sampling and composited prior to freezing at −20 C until analysis.

Ten grams of field moist soil from each sample were weighed into 30-ml Teflon tubes and 10 ml of water plus 10 ml of water-saturated toluene were added. The tubes were shaken horizontally on a shaker for 2 h and then centrifuged at $1{,}500 \times g$ for 15 min. Two milliliters of the toluene layer was transferred to a 2-ml volumetric tube to which was

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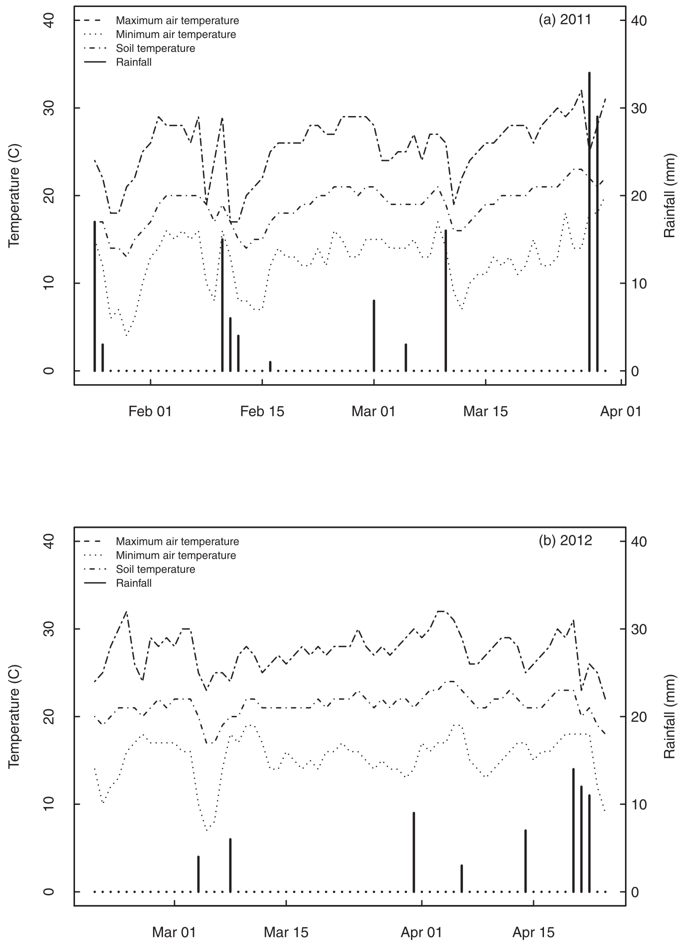

Figure 1. Daily minimum and maximum air temperature, soil temperature, and rainfall during the experiment in 2011 and 2012 (Source: South Florida Water Management District Weather Station. Available at http://erec.ifas.ufl.edu/WD/Ewdmain.htm).

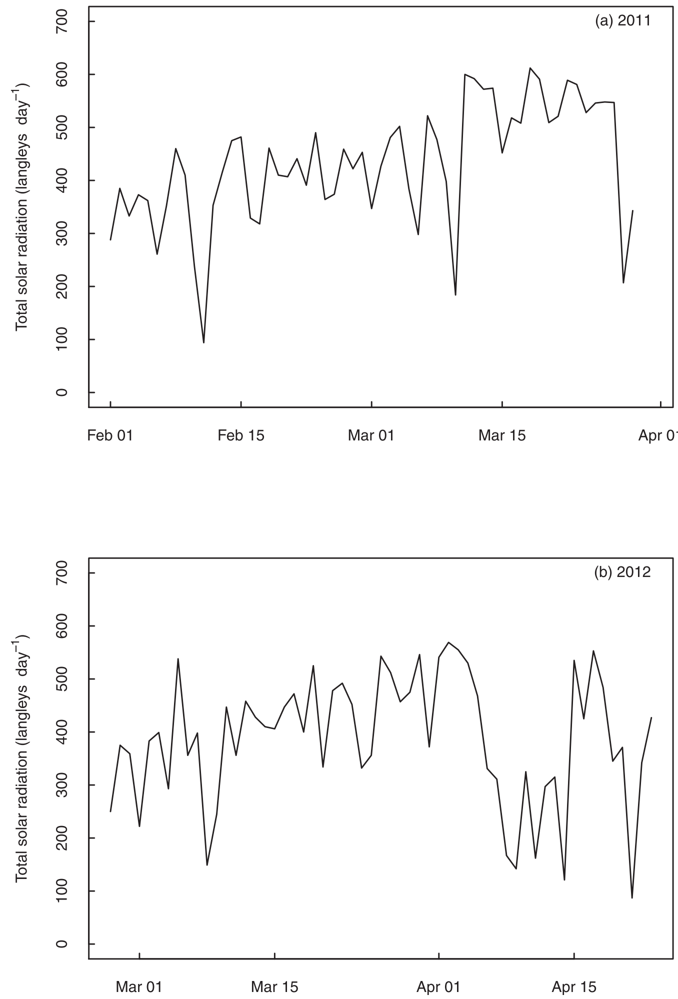

Figure 2. Total solar radiation during the experiment in 2011 and 2012 (Source: South Florida Water Management District Weather Station. Available at http://erec.ifas.ufl.edu/WD/Ewdmain.htm).

added 10 μl of an atrazine internal standard (0.01 mg ml^{-1}). Quality control samples were run with each analysis by spiking soil from the nontreated control plots with 1 or 0.05 mg kg^{-1} of analytical-grade pendimethalin. Recoveries were 85 ± 5%.

The amount of pendimethalin in the toluene extract was determined by gas chromatography and mass spectroscopy (Shimadzu GC-17A and GC-MS QO 5050A, Shimadzu Scientific Instruments, Columbia, MD 21046) by monitoring the masses for atrazine (*m*/*z* 200), and pendimethalin (*m*/*z* 162/252). The program for detecting pendimethalin was as follows: initial oven temperature was 80 C (hold 1 min), which was ramped at 20 C per min to 260 C and then held at 260 C for 2.5 min with a run time of 11.5 min. Under these conditions the retention times of atrazine and pendimethalin were 7.7 and 9.1 min, respectively. The quantification limit was 10 μg kg^{-1} of soil. The soil samples were dried at 80 C and the concentration of herbicide in the soil corrected to dry weight.

Statistical Analysis. Analysis of variance was performed on all data with the use of the MIXED procedure in SAS (SAS version 9.1, SAS Institute Inc., Cary, NC 27513) at 5% level of significance. Data were analyzed separately when there was no significant year effect. Pendimethalin data were fitted to Equation 1 (SigmaPlot Version 11, Systat Software Inc., San Jose, CA 95110) to describe dissipation of the oil- and water-based formulations:

$$Y = Ae^{-kt}, \qquad [1]$$

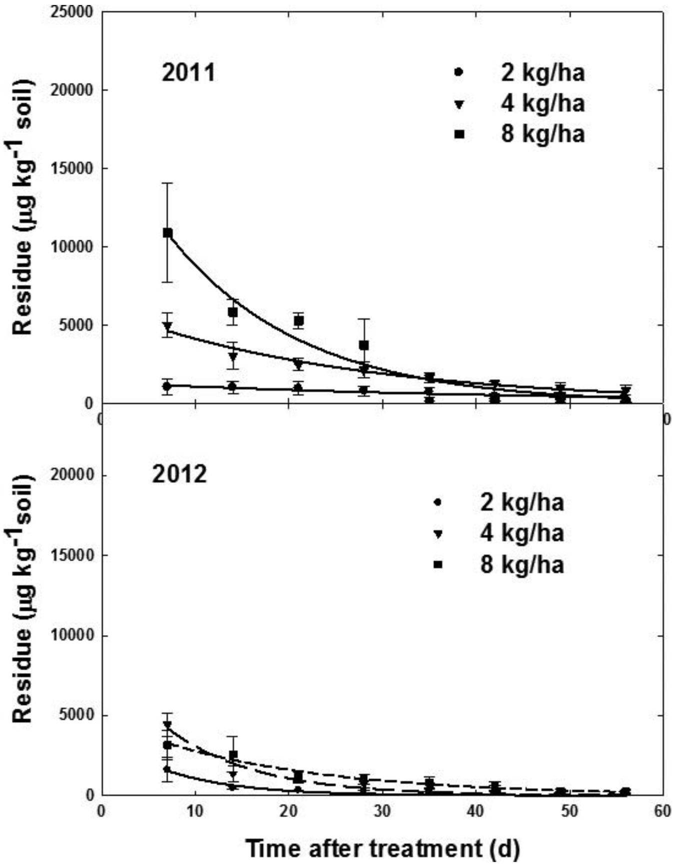

Figure 3. Field dissipation of oil-based formulation of pendimethalin in the top 10 cm of soil in 2011 and 2012 in Belle Glade, FL. Equations: $Y = 1{,}374\ e^{(-0.022t)}$, $Y = 6{,}083\ e^{(-0.038t)}$, and $Y = 17{,}708\ e^{(-0.070t)}$ at 2, 4, and 8 kg ha^{-1}, respectively, in 2011; and $Y = 3{,}774\ e^{(-0.127t)}$, $Y = 4{,}850\ e^{(-0.106t)}$, and $y = 3{,}802\ e^{(-0.055t)}$ at 2, 4, and 8 kg ha^{-1}, respectively in 2012. Error bars represent standard error of the mean.

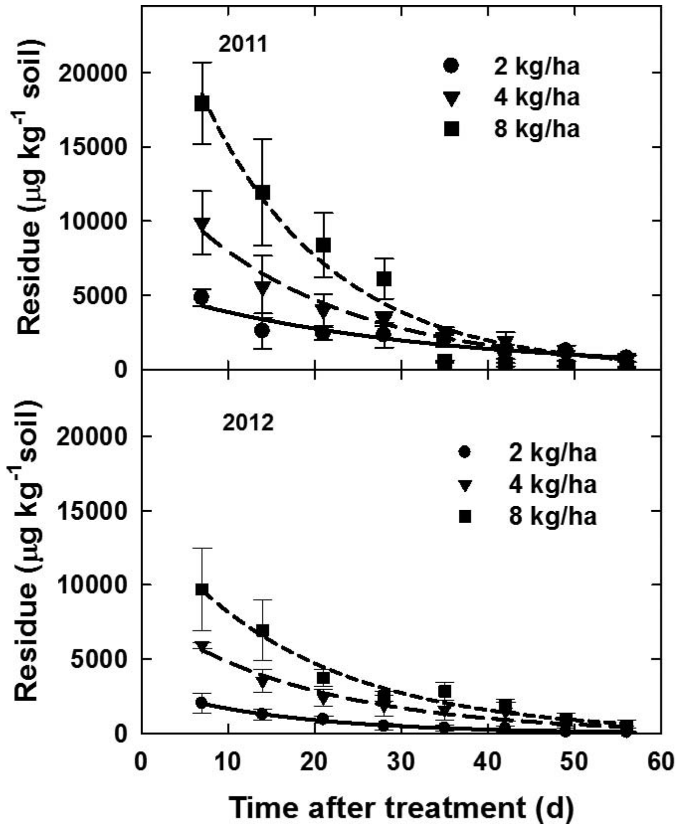

Figure 4. Field dissipation of water-based formulation of pendimethalin in the top 10 cm of soil in 2011 and 2012 in Belle Glade, FL. Equations: $Y = 5{,}449\ e^{(-0.034t)}$, $Y = 13{,}505\ e^{(-0.053t)}$, and $Y = 29{,}817\ e^{(-0.068t)}$ at 2, 4, and 8 kg ha^{-1}, respectively, in 2011; and $Y = 3{,}099\ e^{(-0.062t)}$, $Y = 8{,}080\ e^{(-0.052t)}$, and $Y = 14{,}198\ e^{(-0.055t)}$ at 2, 4, and 8 kg ha^{-1}, respectively, in 2012. Error bars represent standard error of the mean.

where Y is the concentration of herbicide at any time (t, d); A is the amount of herbicide in soil at the first sampling time (mg kg^{-1}); k is the first-order rate constant (d^{-1}); and t is time (d). The DT_{50} values for different rates of each pendimethalin formulation were calculated with the use of Equation 2:

$$DT_{50} = \ln(2)/k. \quad [2]$$

Results and Discussion

There was a significant year effect on dissipation of both pendimethalin formulations; therefore, data are presented by year. The oil-based formulation of pendimethalin had two phases (biphasic) of dissipation both years, with the exception of the lowest rate in 2011 (Figure 3). There was an initial rapid rate of dissipation of pendimethalin within the first 14 d followed by a slow rate of dissipation both years. However, the dissipation curve of pendimethalin at 2 kg ha^{-1} was constant over time. Overall, the initial amount of pendimethalin was higher in 2011 compared to 2012 at all rates of the oil-based formulation. Similarly, the water-based formulation had a biphasic rate of dissipation both years (Figure 4). There was an initial rapid rate of dissipation of pendimethalin within the first 14 d followed by a slower dissipation both years. Similar to the oil-based formulation, the initial amount of pendimethalin was higher in 2011 compared to 2012 for the water-based formulation. Soil and air temperature at the time of herbicide application was 17 and 21

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Table 1. Half-lives (DT_{50}) of field-applied formulations of pendimethalin on organic soils in 2011 and 2012 in Belle Glade, FL.

Formulation	Rate	DT_{50}[a]	
		2011	2012
	kg ha^{-1}	days	
Oil-based	2	31.9	8.1
	4	18.1	7.6
	8	9.9	12.3
Water-based	2	20.4	12.3
	4	13.2	11.5
	8	10.2	13.7

[a] DT_{50} is half-life or the time it takes to 50% dissipation.

C in 2011 and 19 and 23 C in 2012, respectively (Figure 1). Total solar radiation at the time of herbicide application was 208 and 288 langleys day^{-1} in 2011 and 2012, respectively (Figure 2). In addition, a total of 20 mm of precipitation was received 7 d prior to herbicide application in 2011 compared to no precipitation in 2012. The lower initial amount of pendimethalin for both formulations in 2011 compared to 2012 was probably attributed to rapid volatilization from the soil surface as a result of higher soil and air temperatures, and drier soil conditions at the time of application. Lower soil temperatures in the initial period after pendimethalin application has been reported to favor less-rapid loss compared to higher temperatures (Barrett and Lavy 1983; Walker and Bond 1977). Similarly, vaporization of other dinitroanilines (e.g., trifluralin) increase with increasing temperature (Weber 1990). Dinitroanilines undergo photodecomposition when exposed to sunlight (Parochetti and Dec 1978; Savage and Jordan 1980; Weber 1990) which probably contributed to pendimethalin loss. Parochetti and Dec (1978) reported 10% loss of pendimethalin after exposure to unshielded sunlight for 7 d after application. Pendimethalin is degraded primarily by soil microorganisms (Parochetti and Dec 1978; Walker and Bond 1977; Weber 1990). Thus, persistence of pendimethalin in the soil will be primarily affected by factors that alter microbial activity in the soil such as soil organic matter content (Weber 1990). Organic soils of the EAA have large microbial populations (Schueneman and Sanchez 1994). However, microbial breakdown in 2012, which could have resulted in lower initial amount of pendimethalin compared to 2011, was probably insignificant, because the soil was dry and there was no moisture in 7 d prior to and after application. Similarly, Parochetti and Dec (1978) reported that microbial breakdown of pendimethalin was negligible under dry soil conditions 7 d following application. Overall, the initial amount of pendimethalin in the soil was higher with the water-based compared to the oil-based formulation of pendimethalin both years (Figure 3 and 4). This could be attributed to the lower volatility of the water-based formulation. Thus, the water-based formulation may provide better weed control following application based on the initial higher amount of pendimethalin in the soil.

Initial rapid dissipation of pendimethalin followed by a slower rate of dissipation has been reported previously. Walker and Bond (1977) reported that pendimethalin dissipated rapidly in the first 12 to 14 d followed by a slower rate of dissipation as a result of volatilization from the soil surface. Savage and Jordan (1980) reported 41 to 80% loss of pendimethalin from the soil surface within the first 5 to 7 d after application. Similarly, Barrett and Lavy (1983) reported rapid pendimethalin dissipation during the first 14 d after application. The initial rapid dissipation of other soil applied dinitroanilines has been previously reported (Poku and Zimdahl 1980; Serrano et al. 2010; Zimdahl and Gwynn 1977). The rapidly dissipated fraction of pendimethalin does not contribute greatly to weed control; therefore, it is the second phase of dissipation that is important for weed management (Shaner 2012).

The DT_{50} of the oil-based formulation was 32, 18, and 10 d and 8, 8, and 12 d at 2, 4, and 8 kg ha^{-1}, respectively in 2011 and 2012, respectively (Table 1). The DT_{50} of the water-based formulation was 20, 13, and 10 d and 12, 12, and 14 d at 2, 4, and 8 kg ha^{-1}, respectively, in 2011 and 2012, respectively. These DT_{50} values are lower than the reported typical value of 44 d under field conditions (Senseman 2007). The difference in the values is attributed to environmental conditions (soil temperature and water) and organic nature of the soils. Organic muck has been reported to reduce the bioavailability of other dinitroanilines particularly trifluralin (Weber et al. 1974). Soil surface application without any incorporation could have also exacerbated losses by volatilization and photodecomposition. Incorporation can greatly reduce pendimethalin losses from the soil surface (Barrett and Lavy 1983). Pendimethalin has been reported to be more persistent when incorporated in the field (Walker and Bond 1977). Barrett and Lavy (1983) reported that where pendimethalin is not incorporated into the soil, the degree of activity achieved is highly dependent on the changes in the soil water content. In their study, the amount of pendimethalin was reduced where soil water content near the soil surface fluctuated from near saturation to very low levels. A total of 116 and 68 mm of rainfall was received in 2011 and 2012, respectively, during the duration of the study probably explaining the observed low DT_{50} values.

The results of this study show that the rate of dissipation of oil- and water-based pendimethalin formulations was very similar. However, the initial amount of pendimethalin in the soil was higher with the water-based compared to the oil-based formulation. This was attributed to the lower volatility of the water-based compared to the oil-based formulation. The lower DT_{50} values obtained in this study for both pendimethalin formulations under field conditions was attributed to climatic and edaphic conditions in addition to absence of incorporation following application. These results suggest that sugarcane growers on organic soils of the EAA will not observe long residual activity of pendimethalin irrespective of the formulation when applied under dry soil conditions with no incorporation.

Acknowledgments

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