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Duration of Flumioxazin-Based Weed Control in Container-Grown Nursery Crops

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Flumioxazin is used in nursery production and landscape maintenance industries. In these situations, weed control provided by flumioxazin often lasts longer than that reported in soil. Our objective was to quantify flumioxazin longevity under conditions found in nursery production. Pots were filled with 6 : 1 (v/v) pine bark : sand mixture. This nonsoil media is typical of what is used for nursery crop production. Pots were treated with flumioxazin at either 0.28 or 0.42 kg ai ha^{-1}, and subsequently sown with either hairy bittercress (two winter experiments) or spotted spurge (two summer experiments) at weekly intervals. Weed seed germination, emergence, and seedling establishment in the treated pots was compared with nontreated control and used as a proxy for herbicide activity. Flumioxazin provided approximately 7 wk of complete (100%) hairy bittercress control regardless of rate. However, a rate effect was evident in only one of the two experiments conducted with hairy bittercress. In both experiments with hairy bittercress, marginal and highly variable activity was still evident at 18 wk after treatment. Flumioxazin at 0.28 and 0.42 kg ha^{-1} provided 2- and 4-wk complete spotted spurge control, respectively. No spotted spurge control was evident after about 8 wk. Subjecting this less-variable data to nonlinear regression revealed that the time required for 50% reduction in flumioxazin activity was approximately 5.5 and 6.6 wk for the two rates, respectively. A column leaching study revealed that flumioxazin activity remained localized near the surface (0 to 4 cm). Therefore the dissipation observed was likely the result of in situ degradation and not displacement. The high organic matter content of the nonsoil media contributes to the observed persistence of flumioxazin activity.
Nomenclature: Flumioxazin; hairy bittercress, *Cardamine hirsuta* L.; spotted spurge, *Chamaesyce maculata* (L.) Small.
Key words: Herbicide leaching, herbicide persistence, ornamental plant production, soil-less growth media.

Flumioxazin se usa en las industrias de producción de almácigos y de mantenimiento de paisajes. En estas situaciones el control de malezas brindado por flumioxazin suele durar más que lo reportado en suelo. Nuestro objetivo fue cuantificar la longevidad de flumioxazin bajo las condiciones que se encuentran en la producción de almácigos. Se llenaron macetas con una mezcla 6:1 (v/v) de corteza de pino:arena. Este medio sin suelo es típico en la producción de almácigos de cultivos. Las macetas fueron tratadas con flumioxazin a 0.28 ó 0.48 kg ai ha^{-1}, y subsecuentemente sembradas con *Cardamine hirsuta* (dos experimentos de invierno) o *Chamaesyce maculata* (dos experimentos de verano) en intervalos semanales. Flumioxazin brindó aproximadamente 7 semanas de control completo (100%) de *C. hirsuta* sin importar la dosis. Sin embargo, el efecto de dosis fue evidente solamente en uno de los dos experimentos realizados con esta maleza. En ambos experimentos con *C. hirsuta*, una actividad marginal y altamente variable fue evidente todavía a 18 semanas después del tratamiento. Flumioxazin a 0.28 y 0.42 kg ha^{-1} brindó 2 y 4 semanas de control completo de *C. maculata*, respectivamente. No hubo un control evidente de *C. maculata* después de 8 semanas. Al someter estos datos menos variables a regresión no-lineal, se reveló que el tiempo requerido para reducir en 50% la actividad de flumioxazin fue aproximadamente 5.5 y 6.6 semanas para las dos dosis, respectivamente. Un estudio usando una columna de lixiviación reveló que la actividad de flumioxazin se mantuvo localizada cerca de la superficie (0 a 4 cm). Así, la disipación observada fue probablemente resultado de la degradación in situ y no del desplazamiento del herbicida. El alto contenido de materia orgánica del medio sin suelo contribuye a la persistencia observada de la actividad de flumioxazin.

The herbicide flumioxazin is used in nursery production and landscape maintenance industries, providing both PRE and POST weed control (Altland et al. 2003). It is also registered in soybean and peanut. Flumioxazin inhibits protoporphyrinogen oxidase, an enzyme involved in chlorophyll synthesis (Boger and Wakabayashi 1999; Scalla and Matringe 1994). With respect to its PRE activity, Richardson and Zandstra (2006) evaluated flumioxazin and several other PRE-applied herbicides for weed control in gladiolus (*Gladiolus* spp.) grown for cut flowers. Flumioxazin applied at rates between 0.11 and 0.56 kg ai ha^{-1} provided a favorable balance between comprehensive weed control and minimal crop injury.

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Flumioxazin is reported in the Herbicide Handbook to be "not persistent in soils" (Senseman 2007). This report of limited soil persistence is based upon the laboratory-only research of Ferrell and Vencill (2003). In this research, soil was treated with known amounts of flumioxazin, incubated at 15 and 25 C, and analyzed for flumioxazin over time via solvent extraction and gas chromatographic and mass spectrophotometric detection. The DT_{50} (degradation time for 50% loss of the original amount) was determined to range from only 13 to18 d, depending upon soil type. Dissipation from soil was attributed to microbial activity.

Nursery crop producers in our area have observed that the PRE activity of flumioxazin following the application of registered rates (0.28 to 0.42 kg ha^{-1}) typically lasts for more than 30 d. Wehtje et al. (2010) previously evaluated the interaction of flumioxazin and prodiamine when applied as a tank mixture. This study included a dose–response evaluation;

i.e., flumioxazin was applied at eight rates ranging from 0.02 to 0.43 kg ha^{-1}; weed control was evaluated over time. At 6 wk after application, 0.15 kg ha^{-1} controlled large crabgrass 95%. At 12 wk after application a similar level of control required an initial application of 0.36 kg ha^{-1}. Therefore over a 6-wk period, flumioxazin activity apparently decreased by approximately 55%. This rate of loss in activity appears unexpectedly slow in light of the DT_{50} values in soil as reported by Ferrell and Vencil (2003). We hypothesize that the longevity of activity is longer in nonsoil media than in soil. A literature search was unable to recover any study that focused upon the dissipation rate of flumioxazin from nonsoil or soilless media over time.

Loss of herbicide activity over time is typically assumed to be primarily the result of herbicide degradation. However, leaching can be a contributing factor. In the opinion of the authors, leaching is an unlikely means of flumioxazin dissipation from nonsoil media. Flumioxazin water solubility is only 1.8 mg L^{-1} at 25 C (Senseman 2007). In addition, the absorption of herbicides to the nonsoil media used in nursery production is generally very high (Wehtje et al. 1993, 1994), which would further limit water-based displacement. Data to support the contention that flumioxazin is not leached in nonsoil media also is not available. The primary objective of this study was to determine the dissipation rate of flumioxazin in nonsoil media. The secondary was to evaluate the leaching potential for flumioxazin in nonsoil media and thereby confirm that dissipation is likely due to degradation and not the result of water-based redistribution.

Materials and Methods

General Information. All studies were conducted either in the nursery production area or within a greenhouse; both located within the Paterson Greenhouse Complex of Auburn University, Auburn, AL. Hairy bittercress and spotted spurge were selected as bioassay species because of their prevalence in the nursery production and landscape maintenance industries of the southeastern United States. Seed of these two species had been collected and stored at 5 C. The merits of using bioassay for herbicide residue detection have been reviewed by Lavy and Santelmann (1986). One advantage of bioassay over chemical assay is the assurance that only the parent and phytotoxic molecule is what is actually detected. Bioassay can detect herbicide in soil directly; thus the problems and experimental error associated with solvent extraction as required with most chemical assays are avoided. Bioassays are generally more economical than chemical assays; and they can be very sensitive. Funderburk and Lawrence (1963) reported that a bioassay with duckweed (*Lemna minor* L.) as the test species could detect diquat and paraquat in water at concentrations as low as 0.001 ppm.

The nonsoil media used in all studies was a 6 : 1 (v/v) combination of pine bark and sand. This substrate was amended with a controlled-release granular fertilizer (Polyon® 17N-6P-12K, Harrell's Fertilizer, Inc., 203 West 4th Street, Sylacauga, AL 35105), dolomitic limestone, and a micronutrient fertilizer (Micromax®, O. M. Scott Corp., 14111 Scotts Lawn Road, Marysville, OH 43401) at 8.3, 3.0, and 0.9 kg m^{-3}, respectively. Statistical analysis was performed using the PROC GLM procedure SAS® (Statistical Analysis System Software. Release 8.3, SAS Institute, Inc., Box 8000, SAS Circle, Cary, NC 27513) software. Regression analysis and graphic presentation thereof utilized Prism® (GraphPad Software, Inc., 2236 Avenida de la Playa, La Jolla, CA 92037)software. Flumioxazin was applied as the 51% active, WG formulation (SureGuard® 51WG, Valent U.S.A. Corporation, P.O. Box 8025, Walnut Creek, CA 94596).

Leaching Study. We evaluated leaching using the segmented column technique as described by Wu and Santleman (1975), and also by Weber et al. (1986). A 7.5-cm-diam plastic pipe was cut into 4- and 5-cm sections. Three 4-cm sections, topped with one 5-cm section, were duct taped together to yield a 17-cm-tall column. Fiberglass window screen covered the column bottom. Twenty four columns were prepared. Columns were filled with the pine bark–sand media as described above. Columns were placed in an outdoor nursery production area for 1 wk where they received 1.3 cm of irrigation daily to allow for media settling. Media depth in the top 5-cm section was reduced to approximately 4 cm after column settling and drainage had been achieved. Subsequently, flumioxazin was applied to the surface of 20 columns at 0.42 kg ha^{-1}, the highest registered rate. This treatment was applied within an enclosed-cabinet sprayer calibrated to deliver 280 L ha^{-1} at 193 kPa. Four nontreated columns served as controls. Columns were returned to the production area after treatment where they continued to receive 1.3 cm of irrigation daily. Four columns were randomly selected from the treated columns at 2, 4, 6, 8, and 10 wk after treatment and frozen at −4 C. The nontreated columns were also frozen at 2 wk.

After all columns within a common experiment had been frozen, columns were cut into four sections by cutting through the taped joint with a band saw. Sections were placed onto nursery flats such that the upward surface remained upward. A bioassay was used to evaluate flumioxazin presence within the sections. After thawing, 25 spotted spurge seeds were sown on the media surface of the individual sections. Spotted spurge seedlings were counted 3 wk after sowing. Only seedlings that had at least one true leaf and were visually equivalent to the seedlings in the nontreated control were counted. Seedlings that exhibited any degree of herbicide injury symptoms and were deemed not likely to survive were excluded from the count of surviving seedlings. The experiment was conducted twice. Columns were treated on May 20, 2010, and column segments seeded on August 2 in the first experiment. Columns were treated on July 8, 2010 and segments seeded on September 20 in the second experiment. Data were subjected to ANOVA. Fisher's Protected LSD test at the 0.05 level was used to separate between leaching times within a common column segment, and between column segments within a common time.

Persistence Study. We evaluated persistence using a repeated-sampling-over-time procedure, with bioassay detection. The nonsoil media described above was used to fill 360 8-cm-square plastic pots. Media-filled pots were placed in the nursery production area and irrigated 1.3 cm per day for 3 d to achieve settling. Flumioxazin was applied at 0.28 and 0.42

kg ai ha^{-1} to 120 pots, respectively. The remaining 120 pots remained nontreated and served as controls. Flumioxazin treatments were applied within the enclosed-cabinet sprayer as previously described. All pots were maintained in a greenhouse where they received either 1.3 cm (summer experiments) or 0.6 cm (winter experiments) of irrigation daily. One day after flumioxazin application, six pots were randomly selected from each of the three treatment groups (i.e., 0.28 kg ha^{-1}, 0.42 kg ha^{-1}, and nontreated) and seeded with 25 seeds of either hairy bittercress (winter experiments) or spotted spurge (summer experiments). This procedure was repeated at 1 wk after application and continued on a weekly basis for at least 12 wk for the experiments with hairy bittercress, or 11 wk for the experiments with spotted spurge. The two winter experiments with hairy bittercress began on January 4 and March 7, respectively. Summer experiments with spotted spurge began on June 6 and July 11, respectively.

Seedlings were counted 3 wk after seeding using the same criteria as described above. Number of surviving seedlings in the flumioxazin-treated pots was expressed as a percentage of the average number of seedlings in the six nontreated controls. Subtracting this value from100 yielded a measure of control. Therefore control was 100% if a pot had no surviving seedlings. Conversely, control was 0% if a pot had seedling number equal to or higher than that of the nontreated control. Control data were subjected to ANOVA. Our ultimate objective was to fit the data to the four-parameter log-logistic model (also termed the sigmoid curve), as described by Seefeldt et al. (1995). The four parameters of this model are the maximum, minimum, slope, and I_{50}. Since the I_{50} estimates activity half way between the maximum and the minimum, it is also an estimate of the time required for 50% degradation or dissipation of the original amount applied. This term is herein termed the DT_{50} value.

Results and Discussion

Leaching Study. Preliminary statistical analysis showed no interaction between treatment and experimental repetition. Consequently, data were pooled over the two experimental runs before further analysis. Number of spotted spurge seedlings per section in the four sections of the nontreated control were 4.6, 4.0, 5.3, and 6.1, top to bottom, respectively, and were statistically equivalent (Table 1). Among the treated columns, reduced seedling numbers were only observed in the 0- to 4-cm (surface) section. Number of seedlings in the 0- to 4-cm section was 0, 0, 2.3, 3.1, and 6.1 per section at 2, 4, 6, 8, and 10 wk, respectively. Seedling numbers in the remaining three lower sections were consistently equivalent to each other and to the nontreated control. Therefore we conclude that flumioxazin had not been displaced below the surface section. Furthermore, the dissipation of flumioxazin, which was complete by 10 wk after treatment, is exclusively the result of in situ degradation and independent of any vertical displacement.

Herbicide movement in container nonsoil media has been the focus of only minimal research, and no such research has been conducted with flumioxazin. Simmons and Derr (2007) and Briggs et al. (2011) researched the movement of two dinitroaniline herbicides in container media. Simmons and Derr (2007) conducted a leaching study similar in technique to what we used to evaluate the movement of pendimethalin through pine bark. Pendimethalin was applied to the surface of pine bark-filled columns, and subsequently subjected to 17.5 cm of irrigation applied over a 7-d period. A bioassay with large crabgrass [*Digitaria sanguinalis* (L.) Scop] detected pendimethalin up to 9 cm beneath the surface. Briggs et al. (2011) evaluated prodiamine persistence and displacement in a pine bark–sand substrate. Substrate-filled 3.8-L nursery containers were surface treated with prodiamine, and then subjected to 2.5 cm of daily irrigation for periods of 1 to 60 d. Containers were subsequently split open, separated into four depths, and evaluated for prodiamine through a bioassay with bentgrass (*Agrostis palustris* Huds.). Prodiamine activity was detected at all four depths regardless of irrigation amount. It was speculated that the observed displacement was not the result of prodiamine dissolved in water, but actually the displacement of the very small substrate particles, commonly termed the "fines," onto which the prodiamine had been absorbed. We were unable to detect any flumioxazin leaching, which is in contrast to these two studies.

Persistence Studies. In the first experiment with hairy bittercress as the bioassay species, flumioxazin at 0.28 and 0.42 kg ha^{-1} provided 8 and 7 wk of complete (i.e., 100%) control, respectively (Table 2; Figure 1). These same two rates provided 7 and 8 wk of control, respectively, in the second experiment. Control in both experiments progressively deteriorated after the seventh or eighth week. But even after

Table 1. Column leaching of flumioxazin as quantified with spotted spurge bioassay[a]

	Weeks of potential leaching					
Column segment	2	4	6	8	10	Nontreated
cm from surface	Seedlings/section					
0 to 4	0 C, b[b]	0 C, b	2.3 B, a	3.1 B, a	6.1 A, a	4.6 A, a
4 to 8	3.8 A, a	4.0 A, a	4.1 A, a	4.5 A, a	6.3 A, a	4.0 A, a
8 to 12	3.9 A, a	5.0 A, a	5.8 A, a	4.9 A, a	6.6 A, a	5.3 A, a
12 to 16	4.1 A, a	3.9 A, a	3.8 A, a	5.8 A, a	6.4 A, a	6.1 A, a

[a] Flumioxazin had been applied at 0.42 kg ai ha^{-1} to the column surface. Columns received 1.3-cm daily irrigation during the potential leaching period; columns were subsequently frozen (–4 C). After all columns had been frozen, columns were cut into segments, thawed, and 25 spotted spurge seeds were sown onto the upward-orientated segment surface. Number of established seedlings was determined 3 wk later. Nontreated columns were frozen at 2 wk.

[b] Values within a row and followed by different uppercase letters are significantly different according to a Fisher's Protected LSD comparison at the 0.05 level. Similarly, values within a column followed by different lowercase letters are significantly different.

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Table 2. Duration of PRE control with flumioxazin when applied at the low and high registered rates as determined by bioassay; four separate experiments, all conducted in 2011.

Experiment particulars			Duration of control[a]		Nonlinear regression; parameter estimates[b]			
					Slope		DT_{50}	
Date treated	Bioassay species	Duration of experiment	0.28 kg ha^{-1}	0.42 kg ha^{-1}	0.28 kg ha^{-1}	0.42 kg ha^{-1}	0.28 kg ha^{-1}	0.42 kg ha^{-1}
		wk	wk				wk	
January 4	Hairy bittercress	17	8	7		Unsuccessful[c]		
March 7	Hairy bittercress	12	7	8		Unsuccessful[c]		
June 6	Spotted spurge	11	2	4	−0.67	−0.46*	5.0	6.7*
July 11	Spotted spurge	11	2	4	−0.63	−0.68	6.2	6.6

[a] Number of weeks that control remained numerically 100%.

[b] Data were fitted to the four-parameter log-logistic model. The maximum and minimum parameters were constrained to 100 and 0, respectively, since control declined from 100 to 0% over the time period of the experiments.

[c] Because of excessive variation and failure to reach 0% control, data could not be successful fitted to the four-parameter, log-logistic model.

[d]* Significant difference in parameter value between flumioxazin rate.

17 wk in the first experiment, and 12 wk in the second experiment, and regardless of rate, herbicidal activity was still detected. Flumioxazin rate effect was not significant in the first experiment ($P = 0.87$), but was significant in the second experiment ($P < 0.01$). Because of the excessive variability and since control did not decline to zero, the data from neither of the two experiments could be fitted to the log-logistic model or to any other nonlinear regression model.

Flumioxazin at 0.28 and 0.42 kg ha^{-1} provided 2 and 4 wk of complete control, respectively, in the two experiments with spotted spurge as the bioassay species (Table 2; Figure 2). These data could be fitted to the log-logistic model (Table 2).

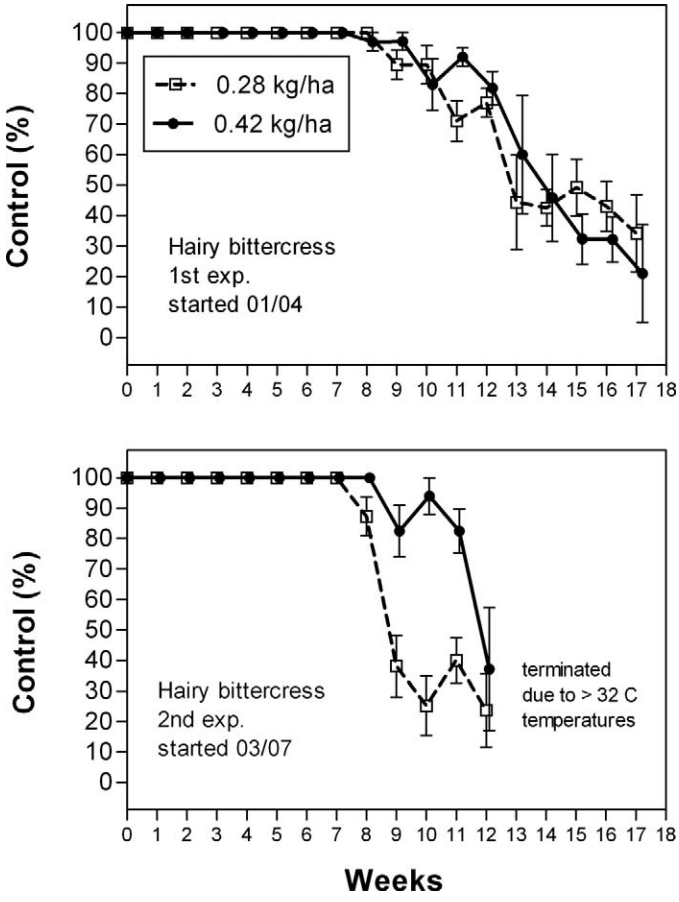

Figure 1. Hairy bittercress control with PRE-applied flumioxazin as influenced by time after application (weeks) and flumioxazin rate. First and second experiments presented top and bottom, respectively. Error bars = standard deviation. Both experiments conducted in 2011.

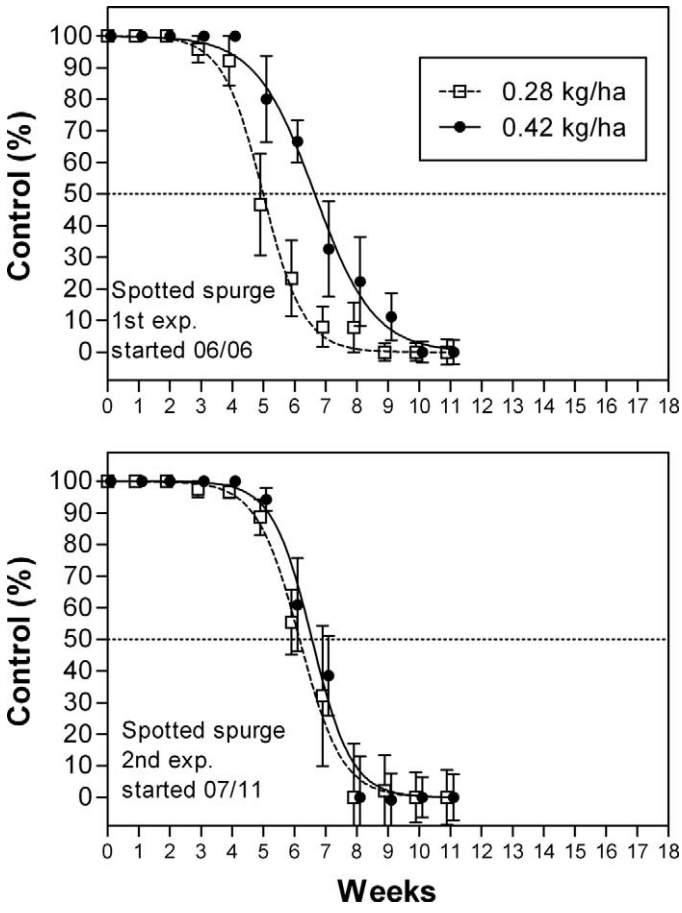

Figure 2. Spotted spurge control with PRE-applied flumioxazin as influenced by time after application (weeks) and flumioxazin rate. First and second experiments presented top and bottom, respectively. Error bars = standard deviation. Both experiments conducted in 2011.

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Since control declined from complete to zero, the upper and lower limits were constrained to 100 and 0, respectively. This allows for more accurate estimations of the remaining two parameters, i.e., DT_{50} and slope (Motulsky and Christopoulos 2004). Flumioxazin activity DT_{50} in the first experiment was 5.0 and 6.7 wk for the 0.28 and 0.42 kg ha^{-1} rates, respectively, and significantly different ($P \leq 0.05$). DT_{50} in the second experiment was 6.2 and 6.6 wk for the same two rates, respectively, and statistically equivalent ($P = 0.32$, Table 2). The DT_{50} values were compared using the lack-of-fit test as described by Seefeldt et al. (1995), which is included in the Prism® software. Therefore the higher rate provided 34% and 6% longer activity half-life in the first and second experiments, respectively. But in averaging across both experiments and rates, the DT_{50} of flumioxazin was 6.13 wk, or approximately 43 d. This activity half-life is over twice as long as what had been reported in soil by Ferrell and Vencill (2003), i.e., only 13 to 18 d.

Ferrell and Vencill (2003) had monitored flumioxazin dissipation in a mineral soil through laboratory-based residue analysis. In contrast, we monitored dissipation in a man-made, organic material-based media. Therefore it cannot be expected that these two studies would yield comparable results. The greater longevity in the nonsoil media suggests that the organic matter is the key factor in extending the flumioxazin activity. Organic matter can have a profound effect on herbicide longevity. Pignatello and Xing (1995) in a review article argued that soil organic matter has a high proportion of meso or micropores (≤ 2 nm). Organic chemical such as herbicides and pesticides in general can enter into, and be held within, these pores. Although a herbicide molecule inside these pores is unavailable for weed uptake and is therefore biologically inactive, it is also protected from microbial-based degradation. Typical bacteria cells range from 500 to 3,000 nm in diameter (Murray et al. 2009). The sorbed herbicide is eventually released back into the water phase due to desorption or eventual organic matter degradation. Once released, it is again available for weed uptake or microbial-based degradation. Pignatello and Xing (1995) surmised that a herbicide is subject to rapid and extensive sorption when introduced into high-organic-matter soil. This is followed by very slow and protracted desorption and dissipation, which typically results in a protracted period of reduced but obviously apparent herbicide activity. Our results with hairy bittercress fit this description.

Data reported herein support our original hypothesis that the longevity of flumioxazin PRE activity is longer in nonsoil media than in soil. Greater longevity occurred in the winter-conducted experiments with hairy bittercress as the test species than in the summer experiments with spotted spurge. We speculate that this difference in longevity can be attributed to hairy bittercress being more sensitive to flumioxazin than spotted spurge, and to reduced microbial activity that likely occurred in winter.

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