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# Effect of Clay, Soil Organic Matter, and Soil pH on Initial and Residual Weed Control with Flumioxazin

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**Abstract:** Greenhouse studies were conducted to evaluate the effects of soil organic matter content and soil pH on initial and residual weed control with flumioxazin by planting selected weed species in various lab-made and field soils. Initial control was determined by planting weed seeds into various lab-made and field soils treated with flumioxazin (71 g ha$^{-1}$). Seeds of *Echinochloa crus-galli* (barnyard grass), *Setaria faberi* (giant foxtail), *Amaranthus retroflexus* (redroot pigweed), and *Abutilon theophrasti* (velvetleaf) were incorporated into the top 1.3 cm of each soil at a density of 100 seeds per pot, respectively. Emerged plants were counted and removed in both treated and non-treated pots two weeks after planting and each following week for six weeks. Flumioxazin control was evaluated by calculating percent emergence of weeds in treated soils compared to the emergence of weeds in non-treated soils. Clay content was not found to affect initial flumioxazin control of any tested weed species. Control of *A. theophrasti*, *E. crus-galli*, and *S. faberi* was reduced as soil organic matter content increased. The control of *A. retroflexus* was not affected by organic matter. Soil pH below 6 reduced flumioxazin control of *A. theophrasti*, and *S. faberi* but did not affect the control of *A. retroflexus* and *E. crus-galli*. Flumioxazin residual control was determined by planting selected weed species in various lab-made and field soils 0, 2, 4, 6, and 8 weeks after treatment. Eight weeks after treatment, flumioxazin gave 0% control of *A. theophrasti* and *S. faberi* in all soils tested. Control of *A. retroflexus* and *Chenopodium album* (common lambsquarters) was 100% for the duration of the experiment, except when soil organic matter content was greater than 3% or the soil pH 7. Eight weeks after treatment, 0% control was only observed for common *A. retroflexus* and *C. album* in organic soil (soil organic matter > 80%) or when soil pH was above 7. Control of *A. theophrasti* and *S. faberi* decreased as soil organic matter content and soil pH increased. Similar results were observed when comparing lab-made soils to field soils; however, differences in control were observed between lab-made organic matter soils and field organic matter soils. Results indicate that flumioxazin can provide control ranging from 75–100% for two to six weeks on common weed species.

**Keywords:** adsorption; efficacy; flumioxazin; herbicide bio-assay; persistence; weed management



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## 1. Introduction

Interactions between a soil-applied herbicide and soil medium are complex. A relative equilibrium is reached soon after the application of a herbicide to soil [1,2]. The portion of herbicide that is not sorbed to the soil particle surface is generally considered available for weed control [3,4]. Therefore, if herbicide adsorption increases, subsequent weed control decreases. The amounts and types of particles in the soil and the soil's pH can greatly affect herbicide adsorption. Herbicide adsorption in soil is often evaluated by deriving a $K_d$ value, a measure of the amount of herbicide in solution to herbicide adsorbed to soil particles [2]. A $K_d$ value is often adjusted for soil organic matter (SOM) due to the magnitude of its role in herbicide binding [2,5,6]. Soil organic matter can differ greatly

in functional group type and abundance, depending on the origin of the SOM, soil pH, climate, and the microbial community [7,8]. Soil organic matter is not the sole sorbent for many herbicides. Soil clay particles, due to their net negative charge, can interact with herbicides in many ways. Clay is reported as the major adsorption surface for certain herbicides [5].

Flumioxazin, a non-ionic protoporphyrinogen oxidase-inhibiting herbicide (PPO, EC 1.3.3.4, herbicide group [HG] 14), is labeled for preemergent use in many crops, including alfalfa (*Medicago sativa* L.), cotton (*Gossypium hirsutum* L.), peanuts (*Arachis hypogaea* L.), potato (*Solanum tuberosum* L.), soybeans (*Glycine max* L.), and wheat (*Triticum aestivum* L.) [9,10]. Flumioxazin has been used widely in crop systems to manage acetolactate synthase (ALS, EC 2.2.1.6, HG 2), photosystem II (PSII, EC 1.10.3.9, HG 5), 5-enolpyruvyl-shikimate synthase (EPSPS [glyphosate], EC 2.5.1.19, HG 9), 4-hydroxyphenylpyruvate dioxygenase-inhibiting (HPPD, EC 1.13.11.27, HG 27), herbicide-resistant, and herbicide-susceptible weed species due to its different sites of action [11–14]. Flumioxazin control has been shown to vary between and within studies on different weed species [15–19]. While the species were similar in each experiment, the soil types in which flumioxazin was applied greatly differed. Observed differences in initial and residual weed control with flumioxazin could be due to variations in the chemical and physical properties of the soil to which it is applied.

Non-ionizable herbicides such as flumioxazin form relatively few associations with soil particles [20,21]. Adsorption of non-ionizable herbicides to soil particles is mainly attributed to hydrophobic binding, which is the result of a decrease in entropy due to partitioning of the hydrophilic herbicide in the hydrophobic regions of soil [20,22]. Soil organic matter and the interlayer of clays provide the conditions necessary to adsorb flumioxazin due to the hydrophobicity of the particles. However, SOM and clay particles are, to some degree, subject to alterations in chemical and physical structure due to solution pH. The effects of soil pH on SOM can be diverse and are due to the effects of speciation on SOM functional groups [22,23]. Ferreira et al. [24] found SOM to increase in hydrophobicity at soil pH values lower than five, which could increase flumioxazin adsorption.

Batch equilibrium and field experiments have been conducted to evaluate the adsorption of flumioxazin. Researchers found the adsorption of flumioxazin to be correlated with SOM and certain types of clay particles [1,25]. These experiments focused extensively on herbicide adsorption, with weed control effects only implied [26]. Field studies are often conducted to determine the effect of soil type on weed control but are complicated due to soil variations within a plot, differences in weather, spatial variation in weed population and density, and a lack in range of soil parameters tested [4,22,27]. Experiments conducted in controlled environments with various soils could provide insight into the initial control and length of pervasive weed control when treated with flumioxazin. Thus, the objective of the experiment was to determine the initial and residual control of flumioxazin as influenced by various soil characteristics on select weed species. The hypotheses of the experiment were that soil pH would have no effect while increasing clay SOM content will reduce flumioxazin initial and residual control due to the non-ionic properties of the herbicides.

## 2. Materials and Methods

### *2.1. Flumioxazin Initial Control*

Three separate greenhouse experiments were conducted at Michigan State University to investigate the effect of clay content, SOM, or soil pH on flumioxazin efficacy. The studies were arranged in a randomized complete block design with a factorial arrangement of treatments, four replications, and repeated in time. Experimental factors included: three soil characteristics, four weed species, and two herbicide treatments. Base soil components utilized in the study were collected from the top 13 cm of respective field soils in uniform areas with no history of flumioxazin application. Soil components were autoclaved prior to use to ensure soil sterility as flumioxazin is susceptible to rapid microbial degradation [28].

Soil particle size distribution, pH, cation exchange capacity (CEC), and SOM content were determined for each soil used (Table 1).

**Table 1.** Properties of the soils used to evaluate the efficacy of flumioxazin on *Echinochloa crus-galli*, *Setaria faberi*, *Amaranthus retroflexus*, and *Abutilon theophrasti* [a].

| Soil | Sand | Silt | Clay | SOM | pH | CEC |
|---|---|---|---|---|---|---|
| Sand | 97.7 | 0.07 | 2.1 | 0.1 | 10 | 0.6 |
| 10% Clay | 88.2 | 0.4 | 11.2 | 0.2 | 9.3 | 1.5 |
| 20% Clay | 78.8 | 0.6 | 20.4 | 0.2 | 9 | 2.9 |
| 30% Clay | 66.5 | 2.6 | 30.7 | 0.2 | 8.8 | 3.7 |
| 40% Clay | 56.2 | 3.4 | 40.2 | 0.2 | 8.7 | 4.9 |
| 50% Clay | 43.7 | 5.4 | 50.6 | 0.3 | 8.7 | 5.4 |
| 60% Clay | 29.4 | 9.6 | 60.7 | 0.3 | 8.4 | 6.4 |
| 70% Clay | 18.8 | 9.6 | 71.1 | 0.5 | 8.3 | 6.9 |
| 0.5% SOM | 93.3 | 0.7 | 5.4 | 0.6 | 9.1 | 2.8 |
| 1% SOM | 92.7 | 0.7 | 5.4 | 1.2 | 8.8 | 3 |
| 2% SOM | 91.7 | 0.7 | 5.4 | 2.2 | 7.8 | 17.1 |
| 3% SOM | 90.9 | 0.7 | 5.4 | 3 | 7.8 | 24.2 |
| 4% SOM | 89.8 | 0.7 | 5.4 | 4.1 | 7.3 | 30 |
| 8% SOM | 85 | 1.7 | 5.4 | 7.9 | 7.1 | 35 |
| 16% SOM | 77.4 | 1.4 | 5.4 | 15.8 | 6.9 | 75.8 |
| 32% SOM | 62 | 0.9 | 5.4 | 31.7 | 6.6 | 121.6 |
| pH 4 | 68.4 | 10.8 | 16.8 | 4 | 4.07 | 6.6 |
| pH 5 | 69.8 | 11 | 15.4 | 3.8 | 4.93 | 7.2 |
| pH 6 | 68.2 | 12.6 | 14.3 | 4.9 | 6.07 | 5.5 |
| pH 7 | 70.1 | 10.4 | 16.8 | 2.7 | 7.07 | 7.1 |

[a] Abbreviations: SOM, soil organic matter; CEC, cation exchange capacity.

### 2.2. Preparation of Soils

A kaolinite clay (Plus White Clay, Charles B. Chrystal Co., Inc., New York, NY 10007), hereafter referred to as clay, was added to sand (Premium play sand, The Quikrete Companies, Atlanta, GA 30329) on a dry weight-to-weight basis to achieve a titration of soil clay content. Soils ranged from 0% clay to 70% clay varying by 10%, resulting in eight unique test soils. Organic soil was obtained from the Michigan State University Muck Farm (42.82° N, 84.37° W) in Laingsburg and is described as a Houghton muck soil (Euic, mesic Typic Haplosaprist) derived from reed sedge plant materials containing 82% organic matter by mass. The organic soil was passed through a 2-mm sieve to remove large debris prior to mixing and was added to sand on a dry weight-to-weight basis to achieve 0.5, 1, 2, 4, 8, 16, and 32% SOM.

A soil of pH 4.8 from a blueberry field consisting of a Pipestone-Kingsville soil (complex, sandy, mixed, mesic Typic Endoaquod) was adjusted to desired pH values of 4, 5, 6, and 7 using NaOH (to alkalify) and $H_3PO_4$ (to acidify). As with the organic soil, the base soil was passed through a 2-mm sieve prior to acid or base treatment to remove debris and large particles. Calculated amounts of acid and base were dissolved in 3 L of de-ionized water and added to 8 kg of soil to make a soil solution. The soil solution was mixed thoroughly and spread over a large surface area to allow for rapid drying to prevent prolonged anaerobic conditions. The soil was mixed every 3 hours until gravimetric water had evaporated. Soils adjusted with NaOH were subjected to salinity analysis by determining electrical conductivity using a 1:1 soil to water ratio. The conductivity of soils ranged from 0.4 to 0.7 mmhos $cm^{-1}$, this was considered to be non-saline for a soil type of loamy sand and able to support normal crop production [22]. Soil pH was tested after completion of the experiment to determine pH stability and was found to vary for all soils by 0.02 to 0.16.

The evaluated weed species consisted of *Abutilon theophrasti* Medik. (velvetleaf), *Amaranthus retroflexus* L. (redroot pigweed), *Echinochloa crus-galli* L. Beauv. (barnyard grass), and *Setaria faberi* Herrm. (giant foxtail). The *A. theophrasti*, *E. crus-galli*, and *S. faberi*

populations were collected from maize (*Zea mays* L.) and soybean fields at the Michigan State University Agronomy Farm (42.71° N, 84.47° W). The *A. retroflexus* population was obtained from a commercial source (Azlin Seed Service, Leland, MS 38756, USA). Seeds of each species were planted at a density of 100 seeds per pot to obtain a target population of 50 seedlings, respectively. Soil was added to 7 by 7 by 6.4 cm pots with the top 1.3 cm of soil being added after mixing with one of the four weed species. After planting, the soil was brought to field capacity and then either left non-treated or treated with flumioxazin (Valor SX 51 WDG, Valent U.S.A. Corporation, Walnut Creek, CA 94596, USA) at 71 g ai $ha^{-1}$ with a track sprayer delivering 187 L $ha^{-1}$. Pots were kept in a greenhouse maintained at 25 ± 5 °C with a 16-h photoperiod of natural sunlight supplemented with high-pressure sodium lighting to provide 1000 µmol $m^{-2}$ photosynthetic photon flux. The day after application, 0.64 cm of water was added over the top of all pots to simulate incorporation by rainfall with subsequent moisture provided by sub-irrigation and weekly topical watering of 0.64 cm.

Emerged weeds were counted and removed from pots two weeks after planting using forceps, carefully removing the growing point to minimize soil mixing. Seedling counts were taken for an additional seven weeks after initial removal with 96% of weed emergence taking place between planting and the first two seedling counts across species. Weed control was calculated using the equation:

$$y = 100 - \left(\left(\frac{t}{n}\right)100\right) \quad (1)$$

where, $y$ is percent control, $t$ is the number of weeds that emerged in treated soil, and $n$ is the average emergence of weeds in the respective non-treated soil. Weed control was analyzed with SAS 9.1 (Statistical Analysis Systems Institute, Inc., Cary, NC 25712, USA) using PROC MIXED to test for significant interactions between the fixed effects of experiments, weed species, and soil characteristics ($p < 0.05$). The experimental block was considered a random effect. No significant differences were found between experiment runs; therefore, weed control data were pooled across experiment runs. Due to significant species by soil interactions, and the large main effect of soil characteristics, weed control was evaluated separately by soil characteristics with comparisons amongst species. Weed control for species affected by soil differences was fit with trend lines using Sigma Plot software (Systat Software, Inc. version 12.5, San Jose, CA 95131, USA) and was modeled using either linear or inverse first-order, regression. Inverse first-order regression fit is described as:

$$y = b + \frac{a}{x} \quad (2)$$

where, $y$ is the weed control achieved at level $x^{-1}$ with asymptote b.

### *2.3. Flumioxazin Residual Control*

Two separate greenhouse experiments were conducted at Michigan State University to investigate the effect of SOM and soil pH on flumioxazin residual control. The SOM and soil pH studies were arranged in a randomized complete block with a factorial arrangement of treatments and were repeated in time. Experimental factors included: five soils, four weed species, two herbicide treatments (treated with flumioxazin or non-treated), and five planting times (0, 2, 4, 6, and 8 weeks after treatment [WAT]) with three replications. The soils utilized for the study were either field soils collected based on desired properties with no prior history of flumioxazin application or soils that were artificially adjusted to provide a range of values for the characteristics to be investigated. Soils collected from the field were taken from uniform areas in respective locations from the top 13 cm of soil. The adjusted soils will hereafter be referred to as "lab soils". Soil particle size distribution, pH, cation exchange capacity, and SOM content were determined for each soil used (Table 1). Field soils were investigated to determine if results from the lab soils were representative of expected field results.

### *2.4. Organic Matter Soils*

Organic soil was obtained from the Michigan State University Muck Farm (42.82° N, 84.37° W) and is described as Houghton muck soil (Euic, mesic Typic Haplosaprists) derived from reed sedge plant materials containing 82% organic matter by mass. The organic soil was passed through a 2-mm sieve to remove large debris prior to mixing and was added to sand on a dry weight-to-weight basis to achieve 0, 1, and 3% SOM. Field soils of 3% SOM (Capac loam, fine-loamy, mixed, mesic Aeric Ochraqualfs) and the unadjusted organic soils were also included for comparison.

### *2.5. pH Soils*

A Capac loam (fine-loamy, mixed, mesic Aeric Ochraqualf) with a pH 6 was collected from a field in a corn-soybean rotation and was adjusted to the desired pH levels of 5 and 7 using $H_3PO_4$ and $Ca(OH)_2$, respectively. As with the organic soil, the base soil was passed through a 2-mm sieve prior to acid or base treatment to remove debris and large particles. To adjust the pH, calculated amounts of acid and base were dissolved in 3 L of de-ionized water and added to 8 kg of soil to create a soil solution. Once in a solution, soil was mixed thoroughly and spread over a large surface area to allow for rapid drying to prevent prolonged anaerobic conditions. The soil was mixed every 3 h until gravimetric water had evaporated. Soils of Capac loam (different from the pH experiment soil) with a pH 5 and a Spinks loam (mixed, mesic Psammentic Hapludalf) pH 7 were collected from fields in corn-wheat-soybean rotations currently planted with wheat under-seeded with red clover (*Trifolium pratense*) were also included for comparison.

Tested weed species were the same as the initial control experiment apart from excluding *E. crus-galli* due to poor germination and including *Chenopodium album* L. (common lamsbsquarters) to include another small-seeded broadleaf species. Seeds of each species were planted at 150 seeds per pot, respectively, except for *A. theophrasti,* which was planted at 65 seeds per pot to obtain a target population of 50 seedlings. Lab soil and field soil were added to 7.0 by 7.0 by 6.4 cm pots brought to field capacity and were either non-treated or treated with flumioxazin at 71 g ai $ha^{-1}$ with a track sprayer delivering 187 L $ha^{-1}$. Once treated, pots were placed in a greenhouse maintained at 25 ± 5 °C with a 16-hour photoperiod of natural sunlight supplemented with high-pressure sodium lighting to provide 1000 µmol $m^{-2}$ photosynthetic photon flux. Weed seeds were planted by scattering one of the four species onto the soil surface then incorporating them with forceps to a depth of 0.5 to 1 cm at 0, 2, 4, 6, and 8 WAT. At each timing, weeds were planted into treated and non-treated soil to evaluate residual control of flumioxazin and to assure that emergence of weeds in treated soils was due solely to chemical control and not changes in the soil. The day after application, 0.64 cm of water was added over the top of all pots to simulate incorporation by rainfall with subsequent moisture provided by sub-irrigation and weekly topical watering of 0.64 cm.

## 3. Data Collection and Analysis

Emerged weeds were counted and removed from pots weekly using forceps carefully removing the growing point to minimize soil mixing. Seedling counts were taken for three weeks after each respective planting with 94% of weed emergence taking place between planting and the first count at one week after planting. Weed control was calculated using Equation (1).

Weed control was analyzed with SAS 9.1 (Statistical Analysis Systems Institute, Inc., Cary, NC 25712, USA) using PROC MIXED to test for significant interactions between the fixed effects of runs, weed species, time after application, and soil effect on weed control ($p < 0.05$). Block was considered a random effect. No significant differences were found between experiment runs; therefore, weed control data were pooled across experiment runs. Due to significant species by soil by time interactions, and the large main effect of soil characteristic, weed control was evaluated separately by soil characteristic with comparisons among species. Weed control for species as affected by soil differences was fit

with trend lines using Sigma Plot software (Systat Software, Inc. version 12.5, San Jose, CA 95131, USA) and was modeled using either linear or logistic regression. Logistic regression fit to data is described as:

$$y = a/(1 + (x/b)^c) \quad (3)$$

where, $y$ is weed control achieved at level $x$ with an upper asymptote of $a$ (with a forced upper limit of 100) and slope of $c$ with the point of inflection $b$ [28]. The time that elapsed in weeks until a 50% reduction in weed control was observed ($I_{50}$) and was calculated for each soil and weed species using the respective regression equation to compare weed control between species and soils.

## 4. Results and Discussion

### *4.1. Flumioxazin Initial Control*

Flumioxazin control varied by soil characteristics and weed species (Table 2). Control of weeds with flumioxazin as affected by SOM was best modeled by linear regression, while the effect of pH on control was best modeled by inverse first-order regression with the coefficient of determination values ranging from 0.31 to 0.78.

**Table 2.** Seedling count of weed species averaged by soil and regression equation used to model weed control for clay, SOM and pH soils [a].

| Soil | Species | Emergence | Model [b] | $r^2$ |
|---|---|---|---|---|
| Clay | | | | |
| | ABUTH | 26 | NS | NS |
| | AMARE | 29 | NS | NS |
| | ECHCG | 88 | NS | NS |
| | SETFA | 18 | NS | NS |
| SOM | | | | |
| | ABUTH | 41 | $y = 94.64 - 0.69b$ | 0.31 |
| | AMARE | 70 | NS | NS |
| | ECHCG | 86 | $y = 95.69 - 1.06b$ | 0.66 |
| | SETFA | 28 | $y = 92.03 - 0.89b$ | 0.48 |
| pH | | | | |
| | ABUTH | 32 | $y = 139.24\ (256.33/b)$ | 0.78 |
| | AMARE | 22 | NS | NS |
| | ECHCG | 65 | NS | NS |
| | SETFA | 20 | $y = 126.43 - (200.38/b)$ | 0.53 |

[a] Abbreviations: SOM, organic matter; ABUTH, *Abutilon theophrasti*; AMARE, *Amaranthus retroflexus*; ECHCG, *Echinochloa crus-galli*; SETFA, *Setaria faberi*; NS, not significant; [b] Regression models fit to data include linear ($y = a + bx$) and inverse first-order regression ($y = b + a/x$).

Clay content did not affect flumioxazin control of any of the tested species (Figure 1). Flumioxazin has been shown to form relatively weak associations with clay particles. Weak adsorption of flumioxazin by clay particles has been suggested to be due to an electronegative region on the molecule causing repulsion with negative surfaces like clay particles [1]. Therefore, due to low adsorption by clay particles, flumioxazin availability is dependent on soil water content [1]. Since soils were maintained at or near field capacity through the duration of the experiment, 100% weed control was observed for all tested species.

Soil organic content effect on weed control varied by species, with control decreasing as SOM content increased (Figure 2). Sebastian et al. [18] found a similar response of decreased flumioxazin control on *Kochia scoparia* L. (kochia) as the SOM increased in similar laboratory experiments. A similar negative correlation of flumioxazin availability and SOM was reported by Schutte et al. [29]. Increasing SOM content reduced control of all species except *A. retroflexus* and was significantly different due to high sensitivity to flumioxazin ($p < 0.05$). The decrease in weed control as SOM increased, as determined from the slope of the regression equations, were 1.06, 0.89, and 0.69 for *E. crus-galli*, *S. faberi*, and *A. theophrasti*, respectively. Control of *E. crus-galli*, *S. faberi*, and *A. theophrasti* at 3% SOM, common to soils in Michigan, could be greater than 85% for all tested species. Conversely, *A. retroflexus*

was completely controlled across the SOM contents tested. Differences in control of weed species due to SOM content are likely due to increased adsorption by hydrophobic bonding to SOM, decreasing herbicide concentration in solution available for control [3,21,22]. The relative control of weed species by flumioxazin across various SOM contents was as follows, from greatest to least control: *A. retroflexus*, *A. theophrasti*, *S. faberi*, *E. crus-galli*, similar to reports by others; however, the level of control observed at various SOM contents differed from the presented experiment. Wilson et al. [10] found that flumioxazin applied at 53 g ai $ha^{-1}$ (lower than the flumioxazin rate [71 g ai $ha^{-1}$] used in this experiment) to SOM content of 0.8% provided 90 and 56% control of *A. retroflexus* and *E. crus-galli*, respectively, 4 WAT in the field. At 0.8% SOM, the observed control of *A. retroflexus* and *E. crus-galli* was 100 and 95%, respectively, which could be due to the higher flumioxazin rate used in the experiment. Niekamp and Johnson [9] observed that flumioxazin at a rate of 71 g ai $ha^{-1}$ applied to soil containing 2.5% SOM provided 82 and 63% control of *A. theophrasti* and *S. faberi*, respectively, 7 WAT in the field. At 2.5% SOM, the observed control of *A. theophrasti* and *S. faberi* was 93 and 90%, respectively. Lastly, Taylor-Lovell et al. [19] found 99% control of *A. theophrasti* at 105 g ai $ha^{-1}$ of flumioxazin, while only 74 to 78% control of *S. faberi* when SOM was 5.6% in the field. At 5.6% SOM, the observed control of *A. theophrasti* and *S.* faberi was 91 and 87%, respectively. The observed control reported in field studies was generally lower than what was observed here, suggesting that differences in control could be due to the continual emergence of weeds, whereas weed emergence was finite in this experiment during a short period of time. Differences between studies also could be due to environmental effects, including rain and soil moisture which have a large impact on flumioxazin control [1,18].

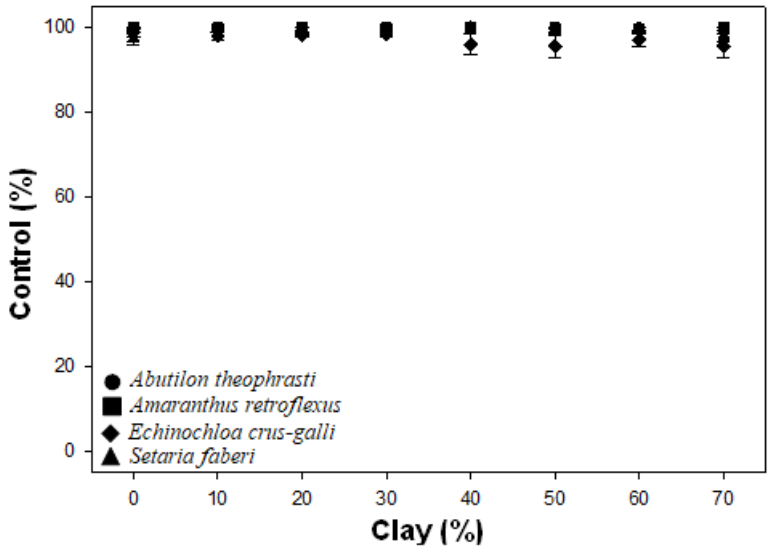


**Figure 1.** Control of flumioxazin on select weed species as affected by clay content. Control of flumioxazin was not affected by clay content across weed species ($p < 0.05$). The vertical bars represent the standard error of means.

The effect of soil pH on weed control varied by species (Figure 3). The control of *S. faberi* and *A. theophrasti* was significantly decreased when soil pH was below 6, with control reduced to 76 and 75%, respectively, at a soil pH of 4. Ferreira et al. [25] demonstrated at a pH of 5.5 or lower, SOM increased in hydrophobicity which could potentially cause increased herbicide adsorption, which could explain the reduction in control of *S. faberi* and *A. theophrasti*. In contrast, the control of *A. retroflexus* and *E. crus-galli* was not affected by soil pH. The control of *A. retroflexus* was 100% at pH values lower than 6 because the species is highly susceptible to flumioxazin. *Echinochloa crus-galli* was controlled at pH values less than 6, regardless of observations of relative tolerance to flumioxazin at higher SOM.

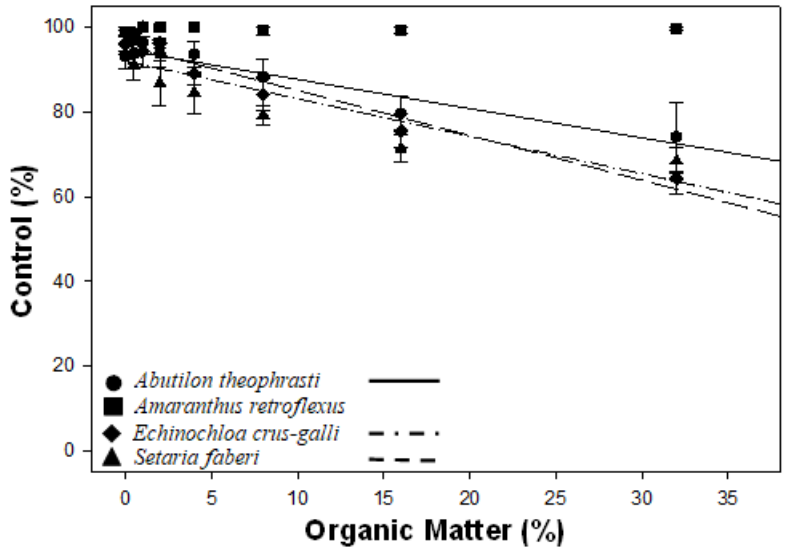


**Figure 2.** Control of flumioxazin on select weed species as affected by soil organic matter content. Fitted lines are calculated by the linear regression equation ($y = a + bx$) for *Abutilon theophrasti*, *Echinochloa crus-galli*, and *Setaria faberi* ($p < 0.05$). Flumioxazin control on *A. theophrasti*, *E. crus-galli*, and *S. faberi* decreased with increasing soil organic matter content. Flumioxazin control on *Amaranth retroflexus* did not change across soil organic matter content ($p > 0.05$). The vertical bars represent the standard error of means.

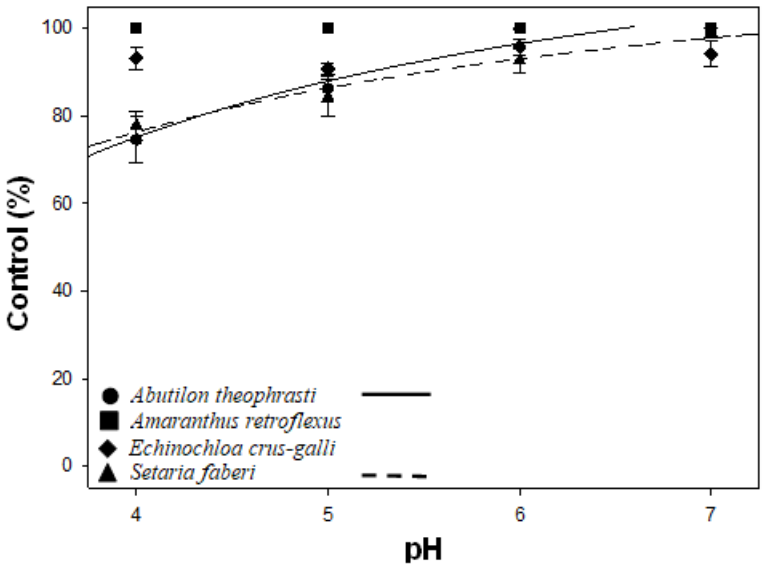


**Figure 3.** Control of flumioxazin on select weed species as affected by soil pH. Fitted lines are calculated by the inverse first-order regression equation ($y = b + a/x$) for *Abutilon theophrasti* and *Setaria faberi* ($p < 0.05$). Flumioxazin control on *A. theophrasti* and *S. faberi* increased with increasing soil pH. Flumioxazin control on *Amaranth retroflexus* and *Echinochloa crus-galli* did not change across soil pH ($p > 0.05$). The vertical bars represent the standard error of means.

Control of weed species in field studies was generally lower than that observed in the greenhouse experiment. Taylor-Lovell et al. [19] found at 105 g ai ha$^{-1}$ of flumioxazin and a soil pH of 6, control of *A. theophrasti* was 99%, similar to the experiment findings of 97% control with 71 g ai ha$^{-1}$ of flumioxazin. Taylor-Lovell et al. [19] observed 74–78% control of *S. faberi*, while here, 93% control was observed. Niekamp and Johnson [9] observed that flumioxazin added to soil with a pH of 6.5 at a rate of 71 g ai ha$^{-1}$, provided 82 and 63% control of *A. theophrasti* and *S. faberi*, respectively, 7 WAT. At a pH of 6.5, the observed

control of *A. theophrasti* and *S. faberi* was 100% and 96%, respectively, with the same rate of flumioxazin.

### *4.2. Flumioxazin Residual Control*

Weed control with flumioxazin varied by soil type and generally decreased over time (Table 3). Residual weed control was best modeled as a logistic response (24 of 40 models) with 4 models sufficiently explained by linear regression and 12 with no significant regression model (Tables 4 and 5). Models that were significant ranged in coefficient of determination values from 0.72 to 0.98 but in most cases were 0.9 or higher.

**Table 3.** Properties of the tested lab and field soils to evaluate the effect of soil organic matter and pH on flumioxazin residual control on *Abutilon theophrasti*, *Amaranthus retroflexus*, *Chenopodium album*, and *Setaria faberi* [a].

| Soil | Sand | Silt | Clay | pH | SOM | CEC |
|---|---|---|---|---|---|---|
| Lab Soil pH 5 | 50.4 | 31 | 16.8 | 5.1 | 2.8 | 21 |
| Field Soil pH 5 | 49.8 | 36.8 | 16.8 | 4.9 | 2.3 | 6.6 |
| Field Soil pH 6 | 37.7 | 33.0 | 23.5 | 6 | 3.2 | 19.6 |
| Lab Soil pH 7 | 48.2 | 29.4 | 20.8 | 7 | 3 | 19.7 |
| Field Soil pH 7 | 40 | 29.6 | 27.8 | 7.1 | 2.6 | 12 |
| Lab Soil 0% SOM | 97.7 | 0.1 | 2.1 | 10 | 0.1 | 0.6 |
| Lab Soil 1% SOM | 92.7 | 0.7 | 5.4 | 8 | 1.2 | 3 |
| Lab Soil 3% SOM | 90.9 | 0.7 | 5.4 | 7.6 | 3 | 24.2 |
| Field Soil 3% SOM | 37.7 | 33 | 23.5 | 6 | 3.2 | 19.6 |
| Organic Soil (Muck) | 7.3 | 9.2 | 1.8 | 6.4 | 81.7 | 142.3 |

[a] Abbreviations: SOM, soil organic matter; CEC, cation exchange capacity.

**Table 4.** Seedling emergence of various weed species averaged over time, $I_{50}$, and regression equation used to model weed control for organic matter soils [a].

| 9 | Species | Emer | $I_{50}$ | Model [b] | $r^2$ |
|---|---|---|---|---|---|
| LS 0% SOM | | | | | |
| | ABUTH | 36.7 | 5.7 | $y = 100/1 + (x/5.67)^{9.85}$ | 0.98 |
| | AMARE | 39.7 | NS | NS | NS |
| | CHEAL | 33.5 | NS | NS | NS |
| | SETFA | 41.6 | 6.3 | $y = 96.48/1 + (x/6.29)^{10.36}$ | 0.98 |
| LS 1% SOM | | | | | |
| | ABUTH | 37.5 | 5.4 | $y = 96.86/1 + (x/5.51)^{4.29}$ | 0.95 |
| | AMARE | 38.9 | NS | NS | NS |
| | CHEAL | 35.5 | NS | NS | NS |
| | SETFA | 40.4 | 4.6 | $y = 97.13 - 10.22x$ | 0.90 |
| LS 3% SOM | | | | | |
| | ABUTH | 38.9 | 4.4 | $y = 93.63 - 10x$ | 0.97 |
| | AMARE | 38.3 | 11.6 | $y = 100/1 + (x/11.57)^{3.63}$ | 0.72 |
| | CHEAL | 35.5 | 13 | $y = 100/1 + (x/13.04)^{2.88}$ | 0.92 |
| | SETFA | 40.6 | 3.5 | $y = 90.7 - 11.65x$ | |
| FS 3% SOM | | | | | |
| | ABUTH | 34.5 | 2.9 | $y = 97.76/1 + (x/2.92)^{1.82}$ | 0.81 |
| | AMARE | 32.5 | NS | NS | NS |
| | CHEAL | 32.6 | NS | NS | NS |
| | SETFA | 38.5 | 2.8 | $y = 92.1/1 + (x/3.03)^{2.4}$ | 0.94 |
| Organic Soil | | | | | |
| | ABUTH | 35 | 1.9 | $y = 77.42/1 + (x/2.44)^{2.6}$ | 0.95 |
| | AMARE | 38.9 | 6.9 | $y = 100/1 + (x/6.92)^{9.28}$ | 0.97 |
| | CHEAL | 34.8 | 7.3 | $y = 100/1 + (x/7.27)^{7.29}$ | 0.98 |
| | SETFA | 37.5 | 1.7 | $y = 80.09/1 + (x/1.99)^{3.12}$ | 0.93 |

[a] Abbreviations: Emer, emergence; SOM, soil organic matter; FS, field soil; LS, lab soil; ABUTH, *Abutilon theophrasti*; AMARE, *Amaranthus retroflexus*; CHEAL, *Chenopodium album*; SETFA, *Setaria faberi*; $I_{50}$, weeks until a 50% reduction in control; NS, not significant; [b] models fit to data include linear ($y = a + bx$) and logistic regression ($y = \frac{a}{1+\left(\frac{x}{b}\right)^c}$).

**Table 5.** Seedling emergence of various weed species averaged over time, $I_{50}$, and regression equation used to model weed control for soil pHs [a].

| Scheme 50. | Species | Emer | $I_{50}$ | Model [b] | $r^2$ |
|---|---|---|---|---|---|
| LS pH 5 | | | | | |
| | ABUTH | 34.9 | 4.49 | $y = 94.37/1 + (x/4.53)^{14.76}$ | 0.95 |
| | AMARE | 33.1 | NS | NS | NS |
| | CHEAL | 32.6 | NS | NS | NS |
| | SETFA | 41.8 | 5.60 | $y = 93.55/1 + (x/5.74)^{5.58}$ | 0.9 |
| FS pH 5 | | | | | |
| | ABUTH | 34.1 | 4.49 | $y = 93.98/1 + (x/4.53)^{14.63}$ | 0.98 |
| | AMARE | 34.2 | NS | NS | NS |
| | CHEAL | 32 | NS | NS | NS |
| | SETFA | 39 | 5.03 | $y = 95.68/1 + (x/5.11)^{5.55}$ | 0.96 |
| FS pH 6 | | | | | |
| | ABUTH | 34.5 | 2.85 | $y = 97.76/1 + (x/2.92)^{1.82}$ | 0.81 |
| | AMARE | 32.5 | NS | NS | NS |
| | CHEAL | 32.6 | NS | NS | NS |
| | SETFA | 38.5 | 2.82 | $y = 92.1/1 + (x/3.03)^{2.4}$ | 0.94 |
| LS pH 7 | | | | | |
| | ABUTH | 35.8 | 3.25 | $y = 97.67/1 + (x/3.27)^{9.26}$ | 0.97 |
| | AMARE | 34 | 5.86 | $y = 100/1 + (x/5.86)^{5.94}$ | 0.95 |
| | CHEAL | 34.3 | 5.93 | $y = 100/1 + (x/5.93)^{6.69}$ | 0.97 |
| | SETFA | 42.9 | 3.83 | $y = 90.63 - 10.61x$ | 0.96 |
| FS pH 7 | | | | | |
| | ABUTH | 35.2 | 2.78 | $y = 99.13/1 + (x/2.79)^{3.31}$ | 0.98 |
| | AMARE | 32 | 5.64 | $y = 100/1 + (x/5.64)^{5.71}$ | 0.88 |
| | CHEAL | 31.8 | 5.75 | $y = 100/1 + (x/5.75)^{5.24}$ | 0.87 |
| | SETFA | 38 | 2.37 | $y = 92.98/1 + (x/2.62)^{1.53}$ | 0.91 |

[a] Abbreviations: Emer, emergence; FS, field soil; LS, lab soil; ABUTH, *Abutilon theophrasti*; AMARE, *Amaranthus retroflexus*; CHEAL, *Chenopodium album*; SETFA, *Setaria faberi*; $I_{50}$, weeks until a 50% reduction in control; NS, not significant; [b] models fit to data include linear ($y = a + bx$) and logistic regression ($y = \frac{a}{1+\left(\frac{x}{b}\right)^c}$).

### 4.3. Organic Soils

Organic matter content significantly influenced flumioxazin residual control (Figure 4). Weed control at 0 WAT ranged from 77 to 100%, with $I_{50}$ values ranging from 1.7 to 13 (Table 4). Lab soil with 0% SOM (100% sand material) had relatively no effect on weed control, and not until 4 WAT was control reduced for *S. faberi* and *A. theophrasti* (Figure 3). Lab soil with 1% SOM resulted in decreased control of *A. theophrasti* after application, and by 2 WAT, control was reduced by 23%. Control of *S. faberi* was affected by SOM content, with reduced control at 2 WAT and 1% SOM compared to 4 WAT for 0% SOM. Control of *C. album* and *A. retroflexus* was not reduced during the duration of the experiment at 1% SOM lab soil; however, control was reduced when SOM was 3%, and seeds were planted 4 WAT. Residual control of *S. faberi* and *A. theophrasti* was greater at 1% SOM than at 3% SOM and was also greater in the field soil than the lab soil. The control of *C. album* and *A. retroflexus* in field soil showed no differences; however, reduced control was observed as SOM changed in lab soil (Figure 3).

Digression between results of lab and field soil at 3% SOM could be due to the type of organic matter in each soil. The SOM in the organic soil used to adjust the lab soil could have a greater affinity for flumioxazin (more hydrophobic) than the SOM found in the field soil [8,30–32]. Differences in weed control in the two soils could also be due to the microbial populations associated with the soil, with populations in the lab soils derived from the organic soil more apt to metabolize flumioxazin [33,34] or cause a synergistic control of weeds [35]. Lastly, control was greatly affected by the organic soil with $I_{50}$ values of 7.3, 1.7, 6.9, and 1.9 for *C. album*, *S. faberi*, *A. retroflexus*, and *A. theophrasti*, respectively (Table 4).

Weed control at 0 WAT for the organic soil was 80 and 77% for *S. faberi* and *A. theophrasti*, respectively, which was the lowest initial control observed for either species (Figure 4).

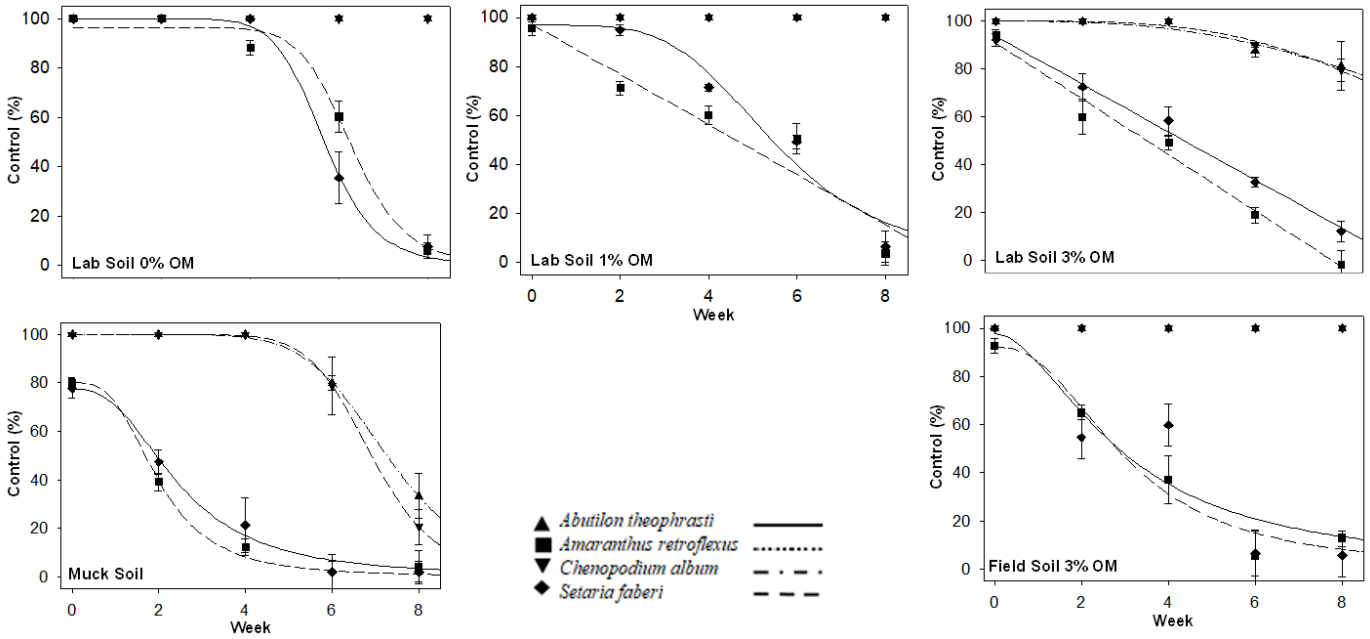


**Figure 4.** Weed control of *Abutilon theophrasti* (▲), *Amaranthus retroflexus* (■), *Chenopodium album* (▼), and *Setaria faberi* (♦) with flumioxazin as affected by percent soil organic matter over time for lab and field soils. Fitted lines for expected control are calculated by linear ($y = a + bx$) or logistic regression ($y = a/(1 + (x/b)\hat{}c)$) for *Abutilon theophrasti*, *Amaranthus retroflexus*, *Chenopodium album*, and *Setaria faberi*. Error bars represent the standard deviation of the mean.

Observed weed control generally decreased as SOM content increased. Control of weed species in response to SOM was similar for the large seeded-broadleaf and grass weed species (*A. theophrasti* and *S. faberi*) and the small-seeded broadleaves (*A. retroflexus* and *C. album*). However, control of *A. theophrasti* tended to be slightly higher than *S. faberi* control except for at 1% SOM lab soil, where the $I_{50}$ value for *S. faberi* was 0.58 greater than *A. theophrasti*.

### 4.4. Soil pH

The residual control of flumioxazin varied greatly by soil pH and species (Figure 4). Initial weed control across all tested species ranged from 91 to 100%, and $I_{50}$ values ranged from 2.37 to 5.93 (Table 5). In both lab and field soils at pH 5, control of *C. album* and *A. retroflexus* remained at 100% for 8 WAT, while *S. faberi* and *A. theophrasti* control decreased over time (Figure 5). Control of *S. faberi* and *A. theophrasti* at pH 5 in lab and field soil began to decrease at 2 WAT with $I_{50}$ values of 5.1 and 4.8, respectively. Minimal differences were observed between the lab and field soil at pH 5 for weed control, with the greatest difference observed in $I_{50}$ values for *S. faberi* being 0.57. The control of *C. album* and *A. retroflexus* was 100% for the duration of the study at a soil pH of 6, similar to the control of these species at a soil pH of 5. Control of *S. faberi* and *A. theophrasti* at pH 6 decreased with a 47 and 37% reduction in $I_{50}$ values, respectively. Loss in weed control from pH 5 to 6 could be due to an increase in the ability of the microbial metabolism or hydrolysis of the herbicide [24,32,35,36]. The control of weed species in pH 7 lab and field soils was similar and only differed by $I_{50}$ values being 0.18 to 0.48 lower for all species except *S. faberi* (1.46) in the field soil. The control of *A. retroflexus* and *C. album* did not decrease until 4 WAT at pH 7, regardless of being lab or field soil. However, at pH 7 lab and field soil, control of *A. theophrasti* and *S. faberi* were similar to the control at pH 6. Reduction in control of *C. album* and *A. retroflexus* but not *S. faberi* and *A. theophrasti* could be due to a slight decrease in herbicide concentration caused by hydrolysis at the higher pH [37]. This

putative decrease in herbicide concentration, however, was not enough to cause a control reduction in the large-seeded species (*A. theophrasti* and *S. faberi*).

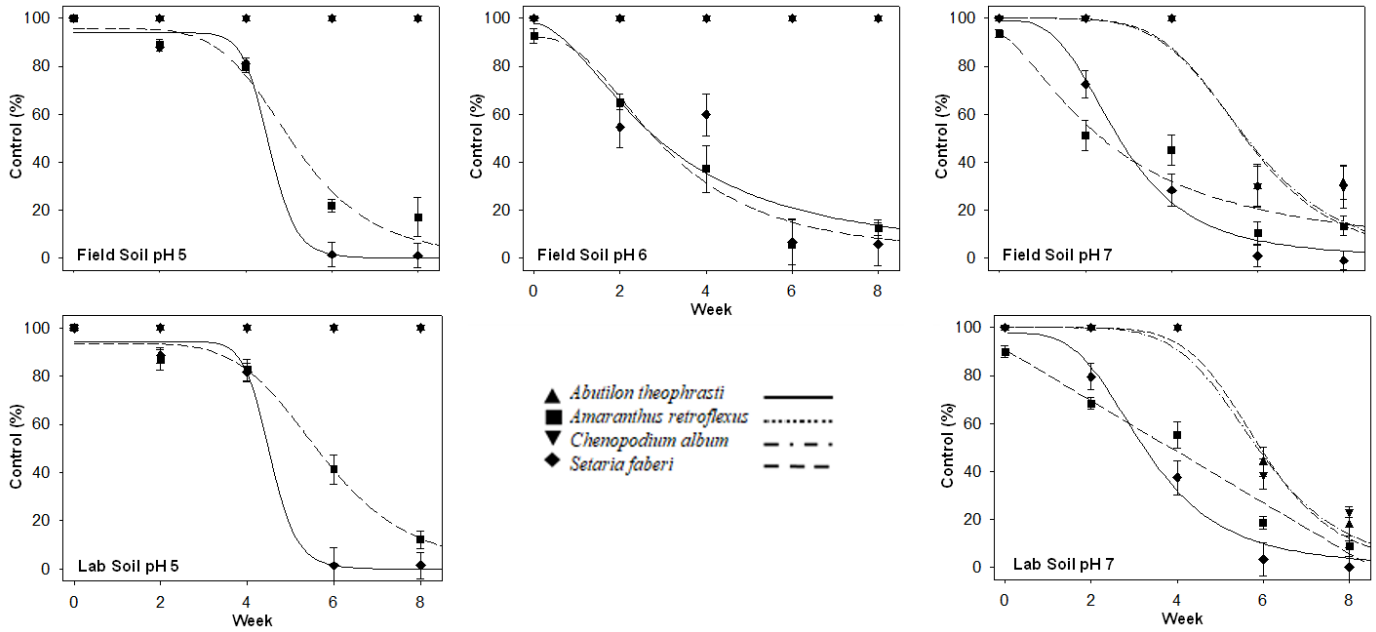


**Figure 5.** Weed control of *Abutilon theophrasti* (▲), *Amaranthus retroflexus* (■), *Chenopodium album* (▼), and *Setaria faberi* (♦) with flumioxazin as affected by soil pH over time for lab and field soils. Fitted lines for expected control are calculated by linear ($y = a + bx$) or logistic regression ($y = a/(1 + (x/b)\hat{}c)$) for *Abutilon theophrasti*, *Amaranthus retroflexus*, *Chenopodium album*, and *Setaria faberi*. Error bars represent the standard deviation of the mean.

Control of all species decreased over time as soil pH increased (Figure 5). The reduction in control of *C. album* and *A. retroflexus* only occurred at the highest soil pH tested; however, control of *S. faberi* and *A. theophrasti* decreased when pH was raised from 5 to 6. When comparing species responses to soil pH, it was observed that similar to SOM soils, *C. album* and *A. retroflexus* had similar responses while *S. faberi* and *A. theophrasti* responded similarly. The reason for differences between the two pairs of weed species and similarities within the pairs could be attributed to seed size, which has been shown to influence herbicide uptake [38].

## 5. Conclusions

The results indicate that SOM content and pH value can adversely impact the initial control effect of flumioxazin on weed species, while clay content does not interfere with flumioxazin control. Additionally, this research shows that increasing SOM and solution pH decreases flumioxazin residual control. Thus, the results reject the null hypotheses for the effect of SOM content and pH on flumioxazin control of select weed species; additionally, the results fail to reject the null hypothesis of the effect of clay control in select weed species with flumioxazin.

Understanding the effects of different soil characteristics on the adsorption of flumioxazin will allow for soil and weed species to have specific herbicide recommendations. If the prevalent weed species are small-seeded broadleaves (i.e., *A. retroflexus or C. album)*, a use rate of 71 g ai $ha^{-1}$ will provide 100% control regardless of SOM content and soil pH for approximately six weeks. However, if the prevalent weed species are grasses or large-seeded broadleaves (i.e., *A. theophrasti*), flumioxazin may need to be tank-mixed with an efficacious herbicide to achieve >85% control for a duration longer than two weeks (dependent or independent of SOM). While most crops are grown in soils with 3–5% OM, there are areas in the world where crops are grown in soils containing low or high OM, as tested in this research. These results provide information regarding flumioxazin control

on pervasive row crop weeds with ubiquitous and anomalous soil characteristics. Adjusting herbicide recommendations based on soil type, and prevalence of weed species, can potentially reduce herbicide use and/or improve weed control.

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