

## Research Article

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# Biologically effective dose of flumioxazin and pyroxasulfone for control of multiple herbicide-resistant waterhemp (*Amaranthus tuberculatus*) in soybean

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**Abstract**

Two studies were conducted to ascertain the biologically effective dose (BED) of flumioxazin and pyroxasulfone for multiple herbicide-resistant (MHR) waterhemp [*Amaranthus tuberculatus* (Moq.) Sauer] control in soybean [*Glycine max* (L.) Merr.] in southwestern Ontario, Canada, during 2016 and 2017. In the flumioxazin study, the predicted flumioxazin doses for 50%, 80%, and 90% MHR *A. tuberculatus* control were 19, 37, and 59 g ai ha<sup>-1</sup> at 2 wk after application (WAA) and 31, 83, and 151 g ai ha<sup>-1</sup>, respectively, at 12 WAA. The predicted flumioxazin doses to cause 5% and 10% soybean injury were 129 and 404 g ai ha<sup>-1</sup>, respectively, at 2 wk after emergence (WAE), and the predicted flumioxazin doses to obtain 50%, 80%, and 95% of the weed-free control plot's yield were determined to be 3, 14, and 65 g ai ha<sup>-1</sup>, respectively. In the pyroxasulfone study, the predicted pyroxasulfone doses that provided 50%, 80%, and 90% MHR *A. tuberculatus* visible control were 25, 50, and 88 g ai ha<sup>-1</sup> at 2 WAA and 41, 109, and 274 g ai ha<sup>-1</sup> at 12 WAA, respectively. The dose of pyroxasulfone predicted for 80% reduction in MHR *A. tuberculatus* density was 117 g ai ha<sup>-1</sup>, and the doses of pyroxasulfone predicted for 80% and 90% reduction in *A. tuberculatus* biomass were 204 and 382 g ai ha<sup>-1</sup>, respectively. The predicted doses of pyroxasulfone that caused 5% and 10% injury in soybean at 2 WAE were 585 and 698 g ai ha<sup>-1</sup>, respectively. The predicted doses of pyroxasulfone required to obtain 50%, 80%, and 95% yield relative to the weed-free plots were 6, 24, and 112 g ai ha<sup>-1</sup>, respectively. Flumioxazin and pyroxasulfone applied preemergence at the appropriate doses provided early-season MHR *A. tuberculatus* control in soybean.

**Introduction**

Waterhemp [*Amaranthus tuberculatus* (Moq.) Sauer] is an economically important weed throughout much North America, particularly in southern Ontario (Benoit et al. 2020; Costea et al. 2005). *Amaranthus tuberculatus* is a troublesome weed in food and fiber crops due to the increasing incidence of the weed itself, increasing complexity of herbicide resistance, *A. tuberculatus*'s ability to reduce crop yield through competition, and increased input costs; growers are forced to employ integrated weed management strategies (Livingston et al. 2016; Orson 1999). To date, *A. tuberculatus* in Ontario has evolved resistance to the acetolactate synthase inhibitor (Group 2), photosystem II inhibitor (Group 5), 5-enolpyruvylshikimate-3-phosphate synthase inhibitor (Group 9), and protoporphyrinogen oxidase (PPO) inhibitor (Group 14) herbicide sites of action, with some populations characterized with multiple herbicide-resistant (MHR) biotypes to all four groups (Heap 2021).

The increasing geographic presence of *A. tuberculatus* and the evolution of MHR *A. tuberculatus* make control more difficult and expensive and may substantially impact yields of crops such as soybean [*Glycine max* (L.) Merr.]. Vyn et al. (2007) documented up to 73% soybean yield loss in fields with high weed density (<1,000 plants m<sup>-2</sup>) due to *A. tuberculatus* interference, which equates to a loss of 2,200 kg ha<sup>-1</sup> based on Ontario's average soybean yield of 3,020 kg ha<sup>-1</sup> (OMAFRA 2019). In dollar value, a soybean grower with an uncontrolled *A. tuberculatus* population could lose up to CAN\$1,008 ha<sup>-1</sup> based on the average Ontario weighted price of CAN\$0.46 kg<sup>-1</sup> in 2018 (GFO 2019).

Agronomic practices greatly influence the competitiveness and economic impact of *A. tuberculatus* on crops. *Amaranthus tuberculatus* control early in the season is vital for soybean growers to minimize yield loss from *A. tuberculatus* interference and maximize net economic returns. Utilizing a two-pass weed control program including an effective

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**Table 1.** Location and application information for flumioxazin and pyroxasulfone biologically effective dose (BED) studies on multiple herbicide-resistant *Amaranthus tuberculatus* during 2016 and 2017 in Ontario, Canada.

Studies	Location	Year	Soil parameters			Planting date	Spray date	Emergence date
			Soil type	Percent OM <sup>a</sup>	pH			
BED of flumioxazin and BED of pyroxasulfone	Walpole I	2016	Sandy loam	6.4	7.6	May 30	June 2	June 7
	Cottam	2016	Sandy loam	2.9	6.5	May 23	May 24	May 30
	Walpole II	2016	Sandy loam	4.3	7.8	May 30	June 2	June 7
	Walpole I	2017	Sandy loam	2.1	8.0	June 8	June 9	June 14
	Cottam	2017	Sandy loam	2.2	6.4	May 19	May 23	May 29
	Walpole II	2017	Loamy sand	2.3	8.3	June 2	June 7	June 9

<sup>a</sup>Abbreviation: OM, organic matter.

preemergence residual herbicide, followed by an effective post-emergence herbicide to control cohorts of *A. tuberculatus* that emerge later, provides the most consistent MHR *A. tuberculatus* control in soybean (Schryver 2017; Vyn et al. 2007). Soil-applied herbicides are a valuable component of an overall MHR *A. tuberculatus* control strategy in soybean.

Flumioxazin, an *N*-phenylphthalimide herbicide with soil-residual and contact activity, inhibits PPO (Dayan and Duke 2010; Price et al. 2004). Flumioxazin provides control of *A. tuberculatus* and other troublesome weeds in Ontario, including redroot pigweed (*Amaranthus retroflexus* L.), common lambs-quarters (*Chenopodium album* L.), common ragweed (*Ambrosia artemisiifolia* L.), eastern black nightshade (*Solanum ptycanthum* Dunal), smartweed (*Polygonum pensylvanicum* L.), and velvetleaf (*Abutilon theophrasti* Medik.) (Niekamp 1998; Nordby et al. 2007; Taylor-Lovell et al. 2002; Valent 1998). Flumioxazin is absorbed mainly through plant roots and is minimally translocated within the phloem (Ferrell and Vencill 2003; OMAFRA 2009; Shaner 2014). Susceptible plants fail to emerge after the application of flumioxazin preplant or preemergence. Emerged weeds at the time of flumioxazin application show necrosis within hours of treatment and desiccate in days (Shaner 2014). Flumioxazin breaks down quickly in soil and water, primarily through microbial activity and hydrolysis (Ferrell and Vencill 2003), with half-lives of less than 1 d in water (USEPA 2003) and less than 20 d in soil (Ferrell and Vencill 2003); this is much shorter than the half-lives of other PPO herbicides like sulfentrazone (Gehrke et al. 2020). Flumioxazin provides residual weed control up to 14 wk (OMAFRA 2009).

Pyroxasulfone is an isoxazoline herbicide that inhibits very-long-chain fatty-acid (VLCFA) synthesis in susceptible weeds (Anonymous 2019a). Pyroxasulfone controls *A. tuberculatus* and other *Amaranthus* species and other dicots such as *C. album*, kochia [*Bassia scoparia* (L.) A.J. Scott], and *S. ptycanthum* (Anonymous 2019a). It also controls annual monocot weeds, including barnyardgrass [*Echinochloa crus-galli* (P.) Beauv.], large crabgrass [*Digitaria sanguinalis* (L.) Scop.], and *Setaria* species (Anonymous 2019a). Pyroxasulfone is mainly absorbed from the soil through the roots of susceptible plants (Anonymous 2019a). It has a half-life of 8.2 to 70 d, which is longer than most other VLCFA elongases-inhibiting herbicides (Mueller and Steckel 2011; Stephenson et al. 2017).

In Ontario, flumioxazin and pyroxasulfone are currently labeled for use in soybean at the rates of 71 to 107 g ai ha<sup>-1</sup> and

125 to 247 g ai ha<sup>-1</sup>, respectively, dependent upon soil texture and organic matter levels. Literature supports that flumioxazin and pyroxasulfone are highly active preemergence herbicides against *Amaranthus* species and MHR *A. tuberculatus* (Nakatani et al. 2016; Strom et al. 2019). To our knowledge, the biologically effective dose (BED) of flumioxazin and pyroxasulfone for MHR *A. tuberculatus* control has not been determined. Proper herbicide dosing is vital background information for growers, agronomists, and ag-retailers that helps them plan their weed management programs to control this problematic weed. Therefore, the objective of these two studies was to establish the BED of flumioxazin and pyroxasulfone applied preemergence for MHR *A. tuberculatus* control in soybean.

## Materials and Methods

### Experimental Methods

Two studies were established: the first study investigated flumioxazin, and the second study investigated pyroxasulfone. These studies each consisted of six field experiments during 2016 and 2017 (three in each year) in commercial soybean fields across southern Ontario with MHR *A. tuberculatus*, previously confirmed with Group 2 (imazethapyr), Group 5 (atrazine), and Group 9 (glyphosate) resistance (Benoit 2019; Heap 2021; Schryver 2017). One field experiment was established near Cottam, ON, Canada (42.149076°N, 82.683687°W). The other two field experiments were established on Walpole Island, ON, Canada (42.561492°N, 82.501487°W and 42.554334°N, 82.515518°W), at two separate field sites. Location, year, soil parameters, and planting, spray, and emergence dates are presented in Table 1.

For both studies, the experiments were established as a randomized complete block design with four replications. Experimental locations were prepared for planting with two passes at right angles to each other using a S-tine cultivator with an attached basket harrow. Plots consisted of 3 soybean rows 75 cm apart for a total plot size of 2.25-m wide and 8-m long. Glyphosate/dicamba-resistant (GDR) soybean cultivars ‘DKB30-61’ (2016) and ‘DKB 10-01’ (2017) were seeded approximately 4-cm deep with a commercial planter calibrated to deposit ~400,000 seeds ha<sup>-1</sup> on the dates presented in Table 1. Treatments in the flumioxazin experiment included a nontreated plot, a weed-free plot that was hand weeded and hoed as necessary, and flumioxazin applications preemergence at 4.5, 8.9, 17.9, 35.8, 71.5, 107.3, 214.6, 429.2,

**Table 2.** Regression parameters ( $\pm$ SE) and predicted flumioxazin rates from an exponential regression model of percent soybean injury at 2, 4, and 8 wk after herbicide application (WAA) and ascending rectangular hyperbola model of grain yield at maturity adjusted to dry weight across six experiments conducted during 2016 and 2017 in Ontario, Canada.

Variable	Parameter estimates ( $\pm$ SE) <sup>a</sup>		Predicted flumioxazin rate <sup>b</sup>		
	<i>a</i>	<i>b</i>	<i>R</i> <sub>5</sub>	<i>R</i> <sub>10</sub>	
Exponential regression			g ai ha <sup>-1</sup>		
Injury at 2 WAA	3.61 (0.70)	0 (0)	129		404
Injury at 4 WAA	1.68 (0.51)	0 (0)	362		592
Injury at 8 WAA	0.09 (0.13)	0 (0)	740		869
Ascending rectangular hyperbola	<i>a</i>	<i>i</i>	<i>R</i> <sub>50</sub>	<i>R</i> <sub>80</sub>	<i>R</i> <sub>95</sub>
Yield	1.59 (0.07)	0.46 (0.13)	3	14	65

<sup>a</sup>Exponential regression parameters (Equation 4): *a*, magnitude constant; *b*, rate constant. Ascending rectangular hyperbola parameters (Equation 1): *a*, upper asymptote; *i*, initial slope (at *X* = 0).

<sup>b</sup>*R*<sub>5</sub> and *R*<sub>10</sub> are the flumioxazin rates required to give 5% and 10% soybean injury, respectively. *R*<sub>50</sub>, *R*<sub>80</sub>, and *R*<sub>95</sub> are the rates of flumioxazin required to obtain 50%, 80%, and 95% yield of weed-free plots, respectively.

and 858.4 g ai ha<sup>-1</sup>. Treatments in the pyroxasulfone experiment included a nontreated plot, a weed-free plot, and pyroxasulfone applications preemergence at 5.6, 11.2, 22.3, 44.6, 89.3, 133.9, 267.8, and 535.5 g ai ha<sup>-1</sup>. Herbicide rates used were a modified titration of the commercially labeled field rates. Treatment applications took place within 5 d after planting using a compressed CO<sub>2</sub>-powered backpack sprayer and with a 1.5-m hand boom equipped with four Hypro ULD 120-02 spray nozzles (Pentair, 375 5th Ave NW, New Brighton, MN) with nozzle spacing at 50 cm producing a 2.0-m-wide spray area, calibrated to apply 200 L ha<sup>-1</sup> of water.

For both studies, evaluation of soybean injury occurred at 2, 4, and 8 wk after soybean emergence (WAE), and visible *A. tuberculatus* control assessments were conducted at 2, 4, 8, and 12 wk after application (WAA). The assessments used a range of 0% to 100% with 0% being no visible soybean injury or no *A. tuberculatus* control, and 100 being total necrosis/death of soybean or *A. tuberculatus*. To obtain *A. tuberculatus* density (plants m<sup>-2</sup>) and above-ground biomass (g m<sup>-2</sup>), two 0.25-m<sup>-2</sup> frames were placed at random in each plot, and *A. tuberculatus* within the quadrats was counted, cut as close as possible to the soil surface, and dried in a kiln at 60 C ambient air temperature until no further mass/moisture reductions were observed. A small-plot combine was used to combine two rows of each plot at harvest maturity; yield (kg ha<sup>-1</sup>) and moisture content were recorded. Harvested yields were adjusted to standard moisture (13%) for statistical analysis.

### Statistical Analysis

Data variance was analyzed via PROC GLIMMIX in SAS v. 9.4 (SAS Institute, 100 SAS Campus Drive, Cary, NC). Treatment was the only fixed effect, while site, replication by site, and site by treatment were identified as random effects. Because the interactions of site by treatment were determined to be nonsignificant, the data were averaged together across all sites.

### Regression Analysis

For each study, the dose responses of flumioxazin and pyroxasulfone were determined for soybean injury at 2, 4, and 8 WAA, MHR *A. tuberculatus* control at 2, 4, 8, and 12 WAA, the population density of *A. tuberculatus* and dry biomass at 8 WAA, and soybean seed yield by fitting one of four regression equations, depending on the dependent variable, to the results via PROC NLIN in SAS v. 9.4

(SAS Institute). The regression analysis used the following equations.

Rectangular hyperbolic equation (fit to yield results) (Cousens 1985):

$$y = (i * \text{dose}) / (1 + [(i * \text{dose}) / a]) \quad [1]$$

where *a* = upper asymptote, and *i* = initial slope.

Exponential decay equation (fit to density and biomass results):

$$y = a + b * e^{(-c * \text{dose})} \quad [2]$$

where *a* = lower asymptote, *b* = change in *y* from intercept to *a*, and *c* = slope from intercept to *a*.

Ascending dose-response equation (fit to control results):

$$y = c + (d - c) / (1 + e\{b * [\ln(\text{dose}) - \ln(i_{50})]\}) \quad [3]$$

where *c* = lower asymptote, *d* = upper asymptote, *b* = slope of the line, and *i*<sub>50</sub> = rate to observe 50% response.

Exponential regression equation (fit to injury results):

$$Y = a * e(b * \text{dose}) \quad [4]$$

where *a* = upper asymptote, and *b* = slope.

For each study, the expected doses of flumioxazin or pyroxasulfone were calculated using the values generated via regression analysis for 50%, 80%, and 90% control of *A. tuberculatus*; 5% and 10% soybean injury; 80%, 90%, and 95% *A. tuberculatus* biomass and density reduction; and 50%, 80%, and 95% of the soybean seed yield relative to the weed-free control. Where the regression model could not calculate the required dose a dash (—) was used in place of numerical values in tables.

## Results and Discussion

### BED of Flumioxazin for Soybean Injury and MHR *Amaranthus tuberculatus* Control

The predicted doses of flumioxazin that caused 5% visible injury to soybean at 2, 4, and 8 WAA were 129, 362, and 740 g ha<sup>-1</sup>, and the predicted flumioxazin doses that caused 10% injury to soybean at 2, 4, and 8 WAA were 404, 592, and 869 g ha<sup>-1</sup>, respectively (Table 2). The predicted flumioxazin doses that were needed for 50%, 80%, and 95% soybean yield relative to the weed-free control

**Table 3.** Regression parameters ( $\pm$ SE) and predicted flumioxazin rates from a dose–response model of percent *Amaranthus tuberculatus* control and inverse exponential model of *A. tuberculatus* density (plants  $m^{-2}$ ) and aboveground biomass (g  $m^{-2}$ ) at 8 wk after herbicide application (WAA) across six experiments conducted during 2016 and 2017 in Ontario, Canada.

Variable	Parameter estimates ( $\pm$ SE) <sup>a</sup>			Predicted flumioxazin rate <sup>b</sup>			
	<i>d</i>	<i>c</i>	<i>b</i>	<i>i</i> <sub>50</sub>	R <sub>50</sub>	R <sub>80</sub>	R <sub>90</sub>
Dose response					g ai ha <sup>-1</sup>		
Control at 2 WAA	97 (1.9)	0 (0)	2.24 (0.26)	18 (1.1)	19	37	59
Control at 4 WAA	99 (2.4)	0 (0)	1.58 (0.16)	22 (1.7)	23	57	100
Control at 8 WAA	100 (2.7)	0 (0)	1.35 (0.13)	28 (2.4)	28	80	147
Control at 12 WAA	99 (2.6)	0 (0)	1.45 (0.14)	31 (2.4)	31	83	151
Inverse exponential	<i>a</i>	<i>b</i>	<i>c</i>		R <sub>80eq</sub>	R <sub>90eq</sub>	R <sub>95eq</sub>
Density	1.88 (22.79)	207.3 (37.79)	0.03 (0.02)		50	73	97
Biomass	2.73 (10.03)	106 (12.25)	0.01 (0)		141	210	301

<sup>a</sup>Dose–response parameters (Equation 3): *d*, upper asymptote; *c*, lower asymptote; *b*, slope; *i*<sub>50</sub>, Rate required for 50% control. Inverse exponential parameters (Equation 2): *a*, lower asymptote; *b*, reduction in *y* from intercept to asymptote; *c*, slope.

<sup>b</sup>R<sub>50</sub>, R<sub>80</sub>, and R<sub>90</sub> are the flumioxazin rates required to give 50%, 80%, and 90% control of *A. tuberculatus*, respectively. R<sub>80eq</sub>, R<sub>90eq</sub>, and R<sub>95eq</sub> are the rates of flumioxazin required to reduce *A. tuberculatus* density and biomass by 80%, 90%, and 95%, respectively.

plots were 3, 14, and 65 g ha<sup>-1</sup>, respectively (Table 2). Low levels of transient injury were observed early in the season, with visible injury no longer apparent at soybean maturity. In past publications, Priess et al. (2020) computed that a preemergence flumioxazin application of 105 g ha<sup>-1</sup> caused 4% to 30% injury in soybean, depending on cultivar. Steppig et al. (2018) reported an application of flumioxazin (preemergence) at 107 g ha<sup>-1</sup> caused an average of 13% soybean injury at 2 WAA, while Mahoney et al. (2014) reported an average of 8% and 3% soybean injury at 2 and 4 WAA, respectively with preemergence-applied flumioxazin at 142 g ha<sup>-1</sup>.

Results of this study are consistent with others documenting that flumioxazin applied preemergence at 71 to 105 g ha<sup>-1</sup> resulted in minimal yield losses in soybean (McNaughton et al. 2014; Taylor-Lovell et al. 2001). Niekamp et al. (1999) found a flumioxazin preemergence application at 90 g ha<sup>-1</sup> caused no significant loss of yield, even in the presence of significant soybean injury. The authors suggested that the injury was due to reduced metabolism of flumioxazin in abnormally cold, wet conditions and decreased microbial degradation (Niekamp et al. 1999). The label for Valtera™ herbicide (51.1 % flumioxazin) does not recommend applications to poorly drained soils and/or applications made when weather conditions are abnormally cold or wet (Anonymous 2019b). Interestingly, even in studies with severe injury, yield losses were relatively low or nonsignificant (Mahoney et al. 2014). The aggressive growth habit of soybean and the ability to branch and compensate for reduced stands have been suggested as reasons for soybean recovery from herbicide injury and subsequent minimal yield loss (Taylor-Lovell et al. 2001).

Flumioxazin doses predicted for 50%, 80%, and 90% MHR *A. tuberculatus* control were 19, 37, and 59 g ha<sup>-1</sup> at 2 WAA; 23, 57, and 100 g ha<sup>-1</sup> at 4 WAA; 28, 80, and 147 g ha<sup>-1</sup> at 8 WAA; and 31, 83, and 151 g ha<sup>-1</sup> at 12 WAA, respectively (Table 3). The doses of flumioxazin required for 50%, 80%, and 90% visible control of MHR *A. tuberculatus* increased at each assessment timing. At 8 and 12 WAA, flumioxazin's predicted dose for 90% MHR *A. tuberculatus* control was above the maximum label dose of 107 g ha<sup>-1</sup> in Canada. The predicted flumioxazin doses for 80%, 90%, and 95% decline in *A. tuberculatus* density were 50, 73, and 97 g ha<sup>-1</sup>, respectively (Table 3). Higher doses were predicted for the same levels of biomass reduction (Table 3). The predicted doses of flumioxazin for 80%, 90%, and 95% decline of *A. tuberculatus* biomass were 141, 210, and 301 g ha<sup>-1</sup>, respectively (Table 3).

These above results are similar to those of Schryver et al. (2017), who reported 85% and 77% MHR *A. tuberculatus* control at

8 and 12 WAA, respectively, with an application of flumioxazin preemergence at 107 g ha<sup>-1</sup> in soybean. The same study found 75% density and 82% MHR *A. tuberculatus* biomass reduction when flumioxazin was applied preemergence (Schryver et al. 2017). In an Illinois study, flumioxazin (70 g ha<sup>-1</sup> preemergence) controlled 4-hydroxyphenylpyruvate dioxygenase-resistant *A. tuberculatus* 90% at 60 DAA (Hausman et al. 2013). Hay et al. (2019) reported 84% MHR *A. tuberculatus* control at 8 WAA with a flumioxazin application (preemergence) at 107 g ha<sup>-1</sup> in soybean. In contrast, Legleiter et al. (2009) found only 48% to 53% *A. tuberculatus* control at 12 WAA with a flumioxazin preemergence application at 90 g ha<sup>-1</sup> in soybean. Conflicting findings may be because of differences in *A. tuberculatus* emergence pattern, *A. tuberculatus* density, soybean emergence timing and canopy development, environmental conditions, or soil characteristics.

#### BED of Pyroxasulfone for Soybean Injury and MHR *Amaranthus tuberculatus* Control

The predicted doses of pyroxasulfone that caused 5% and 10% injury in soybean were 585 and 698 g ha<sup>-1</sup> at 2 WAA and 625 and 730 g ha<sup>-1</sup> at 4 WAA, respectively, which is much higher than the maximum labeled rate in Canada (246.5 g ha<sup>-1</sup>) (Table 4). This indicates excellent soybean tolerance to pyroxasulfone applied preemergence. Soybean injury at 8 WAA could not be predicted via regression equation, because the dose that caused 5% or 10% soybean injury was beyond the dose range evaluated. The predicted doses of pyroxasulfone needed to obtain 50%, 80%, and 95% yield of the weed-free control were 6, 24, and 112 g ha<sup>-1</sup>, respectively (Table 4). Pyroxasulfone doses required for severe soybean injury were beyond the dose range evaluated in this study. In the current study, there was a wide margin of soybean safety, with pyroxasulfone showing low levels of transient soybean injury (>6%) that rapidly disappeared even at doses that exceeded two times the highest labeled dose of 246.5 g ha<sup>-1</sup> (Anonymous 2019a). In other studies, McNaughton et al. (2014) found 178 g ha<sup>-1</sup> of pyroxasulfone applied preemergence caused 3% soybean injury with no yield loss detected. Belfry et al. (2015) also observed minor transient soybean injury with a preemergence treatment of 100 to 150 g ai ha<sup>-1</sup> pyroxasulfone. Others have found pyroxasulfone applications (preemergence) up to 500 g ha<sup>-1</sup> caused negligible (<10%) soybean injury at 2 WAA, but there was up to 25% biomass reduction at 5 WAA (Stephenson et al. 2017; Williams et al. 2017; Yamaji et al. 2014). Soybean tends to recover from early injury without

**Table 4.** Regression parameters ( $\pm$ SE) and predicted pyroxasulfone rates from an exponential regression model of percent crop injury at 2, 4, and 8 wk after herbicide application (WAA) and ascending rectangular hyperbola model of grain yield at maturity adjusted to dry weight across six experiments conducted during 2016 and 2017 in Ontario, Canada.

Variable	Parameter estimates ( $\pm$ SE) <sup>a</sup>		Predicted pyroxasulfone rate <sup>b</sup>		
	<i>a</i>	<i>b</i>	<i>R</i> <sub>5</sub>	<i>R</i> <sub>10</sub>	
Exponential regression			g ai ha <sup>-1</sup>		
Injury at 2 WAA	0.14 (0.05)	0 (0)	585	698	
Injury at 4 WAA	0.08 (0.07)	0 (0)	625	730	
Injury at 8 WAA	0 (0.17)	0 (0)	—	—	
Ascending rectangular hyperbola	<i>a</i>	<i>i</i>	<i>R</i> <sub>50</sub>	<i>R</i> <sub>80</sub>	<i>R</i> <sub>95</sub>
Yield	1.67 (0.07)	0.28 (0.06)	6	24	112

<sup>a</sup>Exponential regression parameters (Equation 4): *a*, magnitude constant; *b*, rate constant. Ascending rectangular hyperbola parameters (Equation 1): *a*, upper asymptote; *i*, initial slope (at *X* = 0).

<sup>b</sup>*R*<sub>5</sub> and *R*<sub>10</sub> are the pyroxasulfone rates required to give 5% and 10% injury of soybean, respectively. *R*<sub>50</sub>, *R*<sub>80</sub>, and *R*<sub>95</sub> are the rates of pyroxasulfone required to obtain 50%, 80%, and 95% yield of weed-free plots, respectively. A dash (—) represents a value that could not be calculated by the regression equation.

**Table 5.** Regression parameters ( $\pm$ SE) and predicted pyroxasulfone rates from a dose–response model of percent *Amaranthus tuberculatus* control and inverse exponential model of *A. tuberculatus* density (plants m<sup>-2</sup>) and aboveground biomass (g m<sup>-2</sup>) at 8 wk after herbicide application (WAA) across six experiments conducted during 2016 and 2017 in Ontario, Canada.

Variable	Parameter estimates ( $\pm$ SE) <sup>a</sup>			Predicted pyroxasulfone rate <sup>b</sup>			
	<i>d</i>	<i>c</i>	<i>b</i>	<i>i</i> <sub>50</sub>	<i>R</i> <sub>50</sub>	<i>R</i> <sub>80</sub>	<i>R</i> <sub>90</sub>
Dose response					g ai ha <sup>-1</sup>		
Control at 2 WAA	94 (2.2)	0 (0)	2.41 (0.31)	24 (1.5)	25	50	88
Control at 4 WAA	94 (2.6)	0 (0)	2.05 (0.25)	30 (2.1)	32	72	145
Control at 8 WAA	95 (3.5)	0 (0)	1.58 (0.19)	37 (3.5)	40	110	247
Control at 12 WAA	93 (3.3)	0 (0)	1.69 (0.21)	38 (3.4)	41	109	274
Inverse exponential	<i>a</i>	<i>b</i>	<i>c</i>		<i>R</i> <sub>80eq</sub>	<i>R</i> <sub>90eq</sub>	<i>R</i> <sub>95eq</sub>
Density	32.12 (35.83)	210.2 (44.27)	0.02 (0.01)		117	—	—
Biomass	9.99 (12.9)	110.7 (14.13)	0.01 (0)		204	382	—

<sup>a</sup>Dose–response parameters (Equation 3): *d*, upper asymptote; *c*, lower asymptote; *b*, slope; *i*<sub>50</sub>, rate required for 50% control. Inverse exponential parameters (Equation 2): *a*, lower asymptote; *b* reduction in *y* from intercept to asymptote; *c*, slope.

<sup>b</sup>*R*<sub>50</sub>, *R*<sub>80</sub>, and *R*<sub>90</sub> are the pyroxasulfone rates required to give 50%, 80%, and 90% control of *A. tuberculatus*, respectively. *R*<sub>80eq</sub>, *R*<sub>90eq</sub>, and *R*<sub>95eq</sub> are the rates of pyroxasulfone required to reduce *A. tuberculatus* density and biomass by 80%, 90%, and 95%, respectively. A dash (—) represents a value that could not be calculated via the regression equation.

any impact on yield (Stephenson et al. 2017; Williams et al. 2017; Yamaji et al. 2014).

The pyroxasulfone doses for 50%, 80%, and 90% MHR *A. tuberculatus* control were 25, 50, and 88 g ha<sup>-1</sup> at 2 WAA; 32, 72, and 145 g ha<sup>-1</sup> at 4 WAA; 40, 110, and 247 g ha<sup>-1</sup> at 8 WAA; and 41, 109, and 274 g ha<sup>-1</sup> at 12 WAA, respectively (Table 5). The doses of pyroxasulfone required for 50%, 80%, and 90% MHR *A. tuberculatus* control increased at each assessment timing. At 12 WAA, the pyroxasulfone dose predicted to achieve 90% MHR *A. tuberculatus* control was above the maximum label dose of 246.5 g ha<sup>-1</sup> in Canada. The predicted dose of pyroxasulfone to reduce *A. tuberculatus* density by 80% was 117 g ha<sup>-1</sup> (Table 5). Additionally, 204 and 382 g ai ha<sup>-1</sup> pyroxasulfone were predicted for 80% and 90% decreases in *A. tuberculatus* biomass, respectively (Table 5). Pyroxasulfone doses required for 90% and 95% *A. tuberculatus* density reduction and 95% *A. tuberculatus* biomass reduction could not be calculated, because those doses were beyond the doses applied in this study. Results are similar to those of Oliveira et al. (2017), who found that pyroxasulfone applied preemergence at 90, 180, and 270 g ha<sup>-1</sup> controlled MHR *A. tuberculatus* 51%, 93%, and 94%, respectively, at 8 WAA. Additionally, Oliveira et al. (2017) showed that pyroxasulfone applied preemergence at 270 g ha<sup>-1</sup> decreased MHR *A. tuberculatus* density 95% in soybean. Other researchers have found preemergence applications of pyroxasulfone (89 to 210 g ha<sup>-1</sup>) to control

*A. tuberculatus* 78% to 87% at 8 WAA (Hausman et al. 2013; Hay et al. 2018; Schryver et al. 2017). Meyer et al. (2016) reported an 81% reduction in *A. tuberculatus* density at 5 WAA with preemergence applications of pyroxasulfone (179 g ai ha<sup>-1</sup>).

This study demonstrates acceptable levels of soybean safety at currently labeled rates of flumioxazin and pyroxasulfone in Ontario, Canada. Flumioxazin and pyroxasulfone applied preemergence provide commercially acceptable early-season MHR *A. tuberculatus* control in soybean at currently labeled rates up to 4 WAA. At 12 WAA, the calculated doses to achieve 90% control of *A. tuberculatus* with flumioxazin or pyroxasulfone were greater than the current Canadian label rates. Higher doses were required for control at each increasing assessment interval, with 70% and 89% more pyroxasulfone and 47% and 51% more flumioxazin required to provide 90% control at 8 WAA and 12 WAA, respectively, as opposed to 4 WAA. The authors suggest that the implementation of a two-pass herbicide program consisting of an effective preemergence residual herbicide such as pyroxasulfone plus flumioxazin and an effective postemergence herbicide could enhance full-season MHR *A. tuberculatus* control in GDR soybean.

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## References

- Anonymous (2019a) Specimen label, Pyroxasulfone 85 WG English label-2019-6220. Durham, NC: K-I Chemical USA Inc. 24 p
- Anonymous (2019b) Specimen label, Valtera™ herbicide English label-2019-3771. Guelph, ON, Canada: Valent Canada Inc. 37 p
- Belfry KD, McNaughton KE, Sikkema PH (2015) Weed control in soybean using pyroxasulfone and sulfentrazone. *Can J Plant Sci* 95:1199–1204
- Benoit L (2019) The Distribution and Control of Herbicide-Resistant Waterhemp (*Amaranthus tuberculatus*) in Ontario. MS thesis. Guelph, ON, Canada: Department of Plant Agriculture, University of Guelph. 191 p
- Benoit L, Hedges B, Schryver MG, Soltani N, Hooker DC, Robinson DE, Laforest M, Soufiane B, Tranel PJ, Giacomini D, Sikkema PH (2020) The first record of protoporphyrinogen oxidase and four-way herbicide resistance in eastern Canada. *Can J Plant Sci* 100:327–331
- Costea M, Weaver S, Tardif F (2005) The biology of invasive alien plants in Canada. 3. *Amaranthus tuberculatus* (Moq.) Sauer var. *rudis* (Sauer) Costea and Tardif. *Can J Plant Sci* 85:507–522
- Cousens (1985) A simple model relating yield loss to weed density. *Ann Appl Biol* 107:239–252
- Dayan FE, Duke SO (2010) Protoporphyrinogen oxidase-inhibiting herbicides. Pages 1733–1751 in Hayes' Handbook of Pesticide Toxicology. Academic Press
- Ferrell JA, Vencill WK (2003) Flumioxazin soil persistence and mineralization in laboratory experiments. *J Agric Food Chem* 51:4719–4721
- Gehrke V, Camargo E, Avila L (2020) Sulfentrazone: environmental dynamics and selectivity. *Planta Daninha* 38, 10.1590/s0100-83582020380100032
- [GFO] Grain Farmers of Ontario (2019) Historical Soybean Prices. <http://gfo.ca/marketing/average-commodity-prices/historical-soybean-prices>. Accessed: September 20, 2021
- Hausman NE, Tranel PJ, Riechers DE, Maxwell DJ, Gonzini LC, Hager AG (2013) Responses of an HPPD inhibitor-resistant waterhemp (*Amaranthus tuberculatus*) population to soil-residual herbicides. *Weed Technol* 27:704–711
- Hay M, Shoup D, Peterson D (2019) Herbicide options for control of Palmer amaranth (*Amaranthus palmeri*) and common waterhemp (*Amaranthus rudis*) in double-crop soybean. *Weed Technol* 33:106–114
- Hay MM, Shoup DE, Peterson DE (2018) Palmer Amaranth (*Amaranthus palmeri*) and common waterhemp (*Amaranthus rudis*) control with very-long-chain fatty acid inhibiting herbicides. *Crop Forage Turfgrass Manag* 4:1–9
- Heap I (2021) The International Survey of Herbicide-Resistant Weeds. [www.weedscience.com](http://www.weedscience.com). Accessed: September 21, 2021
- Legleiter TR, Bradley KW, Massey RE (2009) Glyphosate-resistant waterhemp (*Amaranthus rudis*) control and economic returns with herbicide programs in soybean. *Weed Technol* 23:54–61
- Livingston M, Fernandez-Cornejo J, Frisvold G (2016) Economic returns to herbicide resistance management in the short and long run: the role of neighbor effects. *Weed Sci* 64(S1):595–608
- Mahoney K, Tardif F, Robinson D, Nurse R, Sikkema P (2014) Tolerance of soybean (*Glycine max* L.) to protoporphyrinogen oxidase inhibitors and very long chain fatty acid synthesis inhibitors applied preemergence. *Am J Plant Sci* 5:1117–1124
- McNaughton K, Shropshire C, Robinson D, Sikkema P (2014) Soybean (*Glycine max*) tolerance to timing applications of pyroxasulfone, flumioxazin, and flumioxazin. *Weed Technol* 28:494–500
- Meyer C, Norsworthy J, Young B, Steckel L, Bradley K, Johnson W, Butts T (2016) Early-season Palmer amaranth and waterhemp control from preemergence programs utilizing 4-hydroxyphenylpyruvate dioxygenase-inhibiting and auxinic herbicides in soybean. *Weed Technology* 30:67–75
- Mueller TC, Steckel LE (2011) Efficacy and dissipation of pyroxasulfone and three chloroacetamides in Tennessee field soil. *Weed Sci* 59:574–579
- Nakatani M, Yamaji Y, Honda H, Uchida Y (2016) Development of the novel pre-emergence herbicide pyroxasulfone. *J Pest Sci* 41:107–112
- Niekamp JW (1998) Weed Management with Sulfentrazone and Flumioxazin in No-Tillage Soybean. MS thesis. Columbia: University of Missouri. x p
- Niekamp JW, Johnson WG, Smeda RJ (1999) Broadleaf weed control with sulfentrazone and flumioxazin in no-tillage soybean (*Glycine max*). *Weed Technol* 13:233–238
- Nordby D, Hartzler B, Bradley K (2007) Biology and management of waterhemp. USDA/Perdue Extension—Knowledge to Go. [www.glyphosateweeds crops.org](http://www.glyphosateweeds crops.org)
- Oliveira M, Jhala A, Gaines T, Irmak S, Amundsen K, Scott J, Knezevic S (2017) Confirmation and control of HPPD-inhibiting herbicide-resistant waterhemp (*Amaranthus tuberculatus*) in Nebraska. *Weed Technol* 31:67–79
- [OMAFRA] Ontario Ministry of Agriculture, Food and Rural Affairs (2009) Flumioxazin—Ontario Crop IPM. <http://www.omafra.gov.on.ca/IPM/english/weeds-herbicides/herbicides/flumioxazin.html>. Accessed: September 10, 2021
- [OMAFRA] Ontario Ministry of Agriculture, Food and Rural Affairs (2019) Map shows Record High Soybean Yields. <https://www.agricorp.com/en-ca/News/2019/Pages/PI-MapShowsRecordHighSoybeanYields.aspx>. Accessed: September 21, 2021
- Orson JH (1999) The cost to the farmer of herbicide resistance. *Weed Technol* 13:607–611
- Price AJ, Pline WA, Wilcut JW, Cranmer JR, Danehower D (2004) Physiological basis for cotton tolerance to flumioxazin applied postemergence directed. *Weed Sci* 52:1–7
- Priess G, Norsworthy J, Roberts T, Spurlock T (2020) Flumioxazin effects on soybean canopy formation and soil-borne pathogen presence. *Weed Technol* 34:711–717
- Schryver M (2017) The Distribution and Control of Glyphosate-Resistant Waterhemp (*Amaranthus tuberculatus* var. *rudis*) in Soybean (*Glycine max*) in Ontario. MS thesis. Guelph, ON, Canada: Department of Plant Agriculture, University of Guelph. x p
- Schryver MG, Soltani N, Hooker DC, Robinson DE, Tranel PJ, Sikkema PH (2017) Control of glyphosate-resistant common waterhemp (*Amaranthus tuberculatus* var. *rudis*) in soybean in Ontario. *Weed Technol* 31:811–812
- Shaner DL (2014) Herbicide Handbook. 10th ed. Champaign, IL: Weed Science Society of America. 513 pp
- Stephenson D, Blouin D, Griffin J, Landry R, Woolam B, Hardwick J (2017) Effect of pyroxasulfone application timing and rate on soybean. *Weed Technol* 31:202–206
- Steppig NR, Norsworthy JK, Scott RC, Lorenz GM (2018) Insecticide seed treatments reduced crop injury from flumioxazin, chlorsulfuron, saflufenacil, pyroxasulfone, and flumioxazin + pyroxasulfone + chlorimuron in soybean. *Int J Agron* article ID 9107549
- Strom SA, Gonzini LC, Mitsdarfer C, Davis AS, Riechers DE, Hager AG (2019) Characterization of multiple herbicide-resistant waterhemp (*Amaranthus tuberculatus*) populations from Illinois to VLCFA-inhibiting herbicides. *Weed Sci* 67:369–379
- Taylor-Lovell S, Wax LM, Bollero, G. (2002) Preemergence flumioxazin and pendimethalin and postemergence herbicide systems for soybean (*Glycine max*). *Weed Technol* 16:502–511
- Taylor-Lovell S, Wax LM, Nelson R (2001) Phytotoxic response and yield of soybean (*Glycine max*) varieties treated with sulfentrazone or flumioxazin. *Weed Technol* 15:95–102
- [USEPA] U.S. Environmental Protection Agency (2003) Flumioxazin: Environmental Fate and Ecological Risk Assessment. PC-129034. [https://www3.epa.gov/pesticides/chem\\_search/cleared\\_reviews/csr\\_PC-129034\\_14-Aug-03\\_a.pdf](https://www3.epa.gov/pesticides/chem_search/cleared_reviews/csr_PC-129034_14-Aug-03_a.pdf)
- Valent (1998) Valor Herbicide Technical Information Bulletin. Walnut Creek, CA: Valor U.S.A. Corp.
- Vyn JD, Swanton CJ, Weaver SE, Sikkema PH (2007) Control of herbicide-resistant common waterhemp (*Amaranthus tuberculatus* var. *rudis*) with pre- and post-emergence herbicides in soybean. *Can J Plant Sci* 87:175–182
- Williams M, Hausman N, Moody J (2017) Vegetable soybean tolerance to pyroxasulfone. *Weed Technol* 31:416–420
- Yamaji Y, Honda H, Kobayashi M, Hanai R, Inoue J (2014) Weed control efficacy of a novel herbicide, pyroxasulfone. *J Pestic Sci* 39:165–169