INTERNATIONAL JOURNAL OF AGRICULTURE & BIOLOGY
ISSN Print: 1560–8530; ISSN Online: 1814–9596
25–127/2025/34:340211
http://www.fspublishers.org

Full Length Article

Combination of Pre-emergence Herbicides for Weed Control in Soybean

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Received 07 February 2025; Accepted 09 April 2025; Published online 18 May 2025

Editor: Khawar Jabran

Abstract

Pre-emergence herbicides supress weeds in soybean, enabling the crop to stablish without competition. Therefore, this study aimed to evaluate the selectivity of pre-emergence herbicide combinations in soybean and their effectiveness in controlling weed species. A field experiment was conducted using a randomized block design with four replications. Treatments included two controls (weeded and unweeded) and ten combinations of pre-emergence herbicides: sulfentrazone + diuron; sulfentrazone + clomazone; sulfentrazone + imazethapyr; flumioxazin + imazethapyr; flumioxazin + pyroxasulfone; s-metolachlor + flumioxazin; s-metolachlor + diclosulam; s-metolachlor + fomesafen; s-metolachlor + metribuzin and flumioxazin + imazethapyr + s-metolachlor. Herbicides were applied in a plant-and-spray system. The weed species evaluated were *Bidens pilosa* L., *Commelina benghalensis* L., *Digitaria insularis* L., *Euphorbia heterophylla* L. and *Ipomoea purpurea* L. *E. heterophylla* had the lowest germination flow in the sulfentrazone + clomazone treatment group, which was close to 0 plants/m², obtaining about 90% control at 14 DAE, whereas the sulfentrazone + imazethapyr mixture showed control below 20% at 21 DAE and between 20% and 40% at 28 DAE. *D. insularis* showed low germination flow in all treatments except for sulfentrazone + imazethapyr, s-metolachlor + metribuzin and flumioxazin + imazethapyr, with 1, 2 and 4 plants/m² and, respectively. The combinations of pre-emergence herbicides contributed to an increased spectrum of weed control and no herbicide combination showed significant phytotoxicity to the soybean crop. Compared with the untreated control treatment, the majority of the treatments resulted in an increase in crop yield, with most showing a yield increase of 814 kg ha^{-1} compared to the unweeded control.

Keywords: *Bidens pilosa; Commelina benghalensis; Digitaria insularis; Euphorbia heterophylla*, Pre-emergence herbicides, Soybean

Introduction

The use of pre-emergence herbicides enables the establishment of soybean crop in weed-free areas, preventing competition (Salomão *et al.* 2021) and consequently extending the period before interference (PAI). This period represents the time during which the crop can coexist with weeds without experiencing significant losses of yield (Rizzardi *et al.* 2020). Additionally, these herbicides can reduce weed germination and subsequent re-infestation by maintaining their active ingredients in the soil for an extended period, providing a residual effect (Grint *et al.* 2022). In this context, Silva *et al.* (2023) reported that the herbicides: flumioxazin + imazethapyr, sulfentrazone + diuron, diclosulan and s-metolachlor were highly effective in suppressing new weed emergence during the post-emergence phase of soybean.

However, the correct positioning of these herbicides requires consideration of their physicochemical properties, such as: octanol-water partition constant (Kow), dissociation constant (pKa), water solubility and half-life, along with climatic conditions at the time of the application (Bandeira *et al.* 2022). Therefore, the effectiveness of residual herbicides depends on an understanding of their environmental behaviour based on these physicochemical

To cite this paper: Munaro FC, PVD Silva, RPPN Borges, ESD Medeiros, BF Schedenffeldt, PA Monquero, MF Ortiz, EP Westra, PAV Salmazo, M Mauad, RDC Dias (2025). Combination of pre-emergence herbicides for weed control in Soybean. *Intl J Agric Biol* 34:340211. https://doi.org/10.17957/IJAB/15.2359

characteristics (Prado *et al.* 2022). Thus, rotating and diversifying herbicides mechanisms of action are essential, particularly, in grain production areas, to mitigate resistance development (Ofosu *et al.* 2023).

To enhance weed control effectiveness, combining pre-emergence herbicides with different mechanisms of action broadens the control spectrum and helps preventing the selection of resistant biotypes. The study by Gazola *et al.* (2021) supports this approach, reporting that s-metolachlor application in soybean effectively controlled *Eleusine indica* and *Digitaria insularis* L.*,* achieving control levels close to 100% and maintaining a residual effect for at least 30 days. For *Bidens pilosa* L., the control reached 80% after 30 days, whereas for *Amaranthus hybridus,* control remained effective up to this period, but declined over time.

These results indicate that the efficacy of a given herbicide can vary significantly among different weed species. The combination of different pre-emergence herbicides may extend their residual effect, enhance the suppression of new germination cycles and improve overall control efficacy. However, there is a relative scarcity of studies on the impact of pre-emergence herbicide combinations in controlling different weed species (Barbieri *et al.* 2023). Therefore, the presented study aimed to evaluate the selectivity of pre-emergence herbicides in soybean crop and their effectiveness in controlling different weed species.

Materials and Methods

Area characterization

A field experiment was conducted at the Experimental Farm of Agricultural Sciences (FAECA) of the Federal University of Grande Dourados – UFGD, located in Dourados/MS. The geographical location of the area is 46°51' W longitude, 21°57'S latitude and 430 m altitude above sea level. The region is situated in a tropical climate type Am, with an average annual rainfall pf 1428 mm and average temperature of 22.7°C according to the Koppen climate classification (Fietz *et al.* 2017).

The data on climatic conditions in the municipality of Dourados during the experimental period were collected through Embrapa-UFGD's rainfall station. Fig. 1 presents the daily historical series of accumulated rainfall and minimum and maximum temperatures in the municipality from October 1, 2021, to March 31, 2022 (Emprapa 2023).

Soil samples were collected, at the time of the experiment, at a depth of 0-20 cm and classified as Dystroferric Red Oxisol (Santos *et al.* 2018), which has a clayey texture whose physical-chemical properties (Table 1).

Experiment design

The experimental delineation was positioned in block design with four repetitions, which experimental units consisted of 3 x 6 m plots, totalizing an area of 18 m^2 with 5 soybean rows in a floor area of 12 m^2. The soybean's cultivar chosen for sowing was Monsoy 6410 due to its representativeness of the region. This cultivar presents a 6.4 maturation's degree and indeterminate cycle with medium size to high branching index.

The weed species were chosen based on their regional importance, correlating the frequency of germination and their difficulty to control. For this purpose, the following species were selected: *Bidens pilosa* L.*, Digitaria insularis* L.*, Commelina benghalensis* L.*, Euphorbia heterophylla* L. and *Ipomoea purpurea.* The treatments used for the experiment were based on trade associations of different pre-emergence herbicides (Table 2).

Installation and driving

Natural infestations of the species *B. pilosa* and *D. insularis* were identified at the experimental area before the beginning of the experiment through a population survey via the square inventory method. Thus, desiccation was performed prior to the beginning of the experiment through clethodim (240 g ai ha^{-1}), which was applied at a dose of 0.5 L ha^{-1} of commercial product (Select®) with a sequential application with ammonium glufosinate (400 g ai ha^{-1}) at a dose of 2 L ha^{-1} (Trunfo®) 10 days after the initial treatment. After these applications, a light harrowing operation was performed, followed by the use of a grader, totalling this preparation management of the area 20 days before soybean planting. This preparation was necessary so that only a new germination flow of *D. insularis* and *B. pilosa* remained in the experimental area and not the plants that were already perennial in the area. This management method was also adopted because there was no residual control over weeds or soybeans.

The crop was sown on October 21st, 2021 with 0.45 m spacing between rows and 14 seeds per linear m, aiming a final stand population of approximately 310,000 plants per ha. Fertilization was performed on the sowing line with formulated NPK fertilizer on 10/21/2021. Soybean seeds were treated before planting with the fungicide and insecticide standak top (25 g L^{-1} pyraclostrobin + 225 g L^{-1} thiophanate methyl + 250 g L^{-1} fipronil), using the recommended dose of 200 mL for 100 kg of soybean seeds. The crop was mechanically sown in a seeder.

Commelina benghalensis L., *B pilosa, D, insularis, E. heterophylla* and *I. purpurea* were sown immediately after soybean sowing and before herbicide application to obtain greater uniformity in terms of germination flow and evaluation. Sowing was performed superficially without the need for incorporation into the soil. The seeds of the analysed species were purchased from the commercial company Agrocosmos in sufficient quantity to obtain a population density of 10 plants/m^2.

Table 1: Chemical and physical analysis of the soil was performed at the experimental site

Ca^{2+}	Mg	H + Al	SB	CEC	Al	K	P	BS	pH
				cmol dm^{-3}				%	$CaCl_2$
4.56	2.08	7.08	6.74	13.82	0.12	18	40.73	48.8	5.08

CEC - Effective cation exchange capacity; SB - Sum of bases; pH determination using the $CaCl_2$ method; BS - Base saturation. Source: TECSOLO Laboratory

Table 2: Pre-emergent herbicide treatments and their associations, as well as treatments with controls without herbicides (weeded and absolute)

Herbicides	Herbicides	Dose	Commercial dose
Assets	Trade name	(g ai ha^{-1})	(L or kg ha^{-1})
absolute control	-	-	-
weeded witness	-	-	-
sulfentrazone + diuron	Stone	210 + 420	1.2
sulfentrazone + clomazone	Boral + Reactor	600 + 540	1.2 + 1.5
sulfentrazone + imazethapyr	Allus	360 + 96	1.2
flumioxazin + imazethapyr	Zethamaxx	50 + 106	0.5
flumioxazin + pyroxasulfone	Kyojin	60 + 90	0.3
s-metolachlor + flumioxazin	Grasp	840 + 42	0.3
s-metolachlor + diclosulam	Dual Gold + Spider	1440 + 29	1.5 + 0.035
s-metolachlor + fomesafen	Eddus	1035 + 228	2
s-metolachlor + metribuzin	Boundary	942 + 224	1.5
flumioxazin + imazethapyr + s-metolachlor	Zethamaxx + Dual Gold	50 + 106 + 1440	0.5 + 1.5

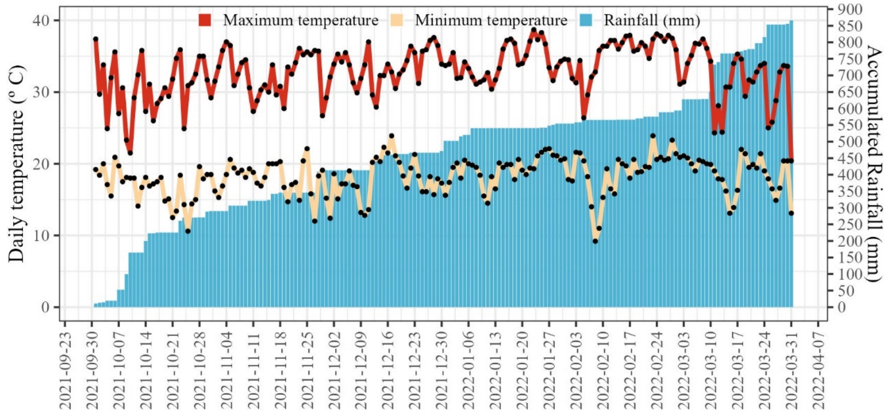

Fig. 1: Historical daily data on accumulated rainfall and minimum and maximum temperatures in Dourados, Mato Grosso do Sul, Brazil, from October 1, 2021 to March 31, 2022

The herbicide treatments were applied immediately after sowing during soybean and weed's pre-emergence through a backpack sprayer pressurized with CO_2, equipped with a spray boom containing six Teejet 110.02 fan nozzles spaced 0.5 m apart, covering a range of 3 m and an application volume of 150 L ha^{-1}. For the Dourados-MS region, the climatic conditions were 28°C, with a wind speed of 5 km h^{-1} and sunny skies.

Then, the crop was monitored and when necessary, conducted fungicides and insecticides applications. Thus, on 11/15/2021, the fungicide Prothioconazole + Bixafen + Trifloxostrobin (500 mL ha^{-1}) was applied. In the year 2022, in January, foliar fertilizer supplemented with boron and zinc (495 mL ha^{-1}), the insecticide lambdacyhalothrin + thiamethoxan (250 mL ha^{-1}), the fungicide fluxapyroxad + pyraclostrobin (350 mL ha^{-1}), the insecticide bifenthrin + carbosulfan (600 mL ha^{-1}) and the fungicide difenoconazole + cyproconazole (300 mL ha^{-1}) were applied.

Experimental evaluations

It was conducted visual evaluations of phytotoxicity, weed control and germination flow at 7, 14, 21 and 28 days after soybean emergence (DAE) through the ALAM's visual scale (Alam 1974), which assigns a score of 0% for the absence of symptoms caused by the herbicide and 100% for the death of the weed. For the phytotoxicity's evaluation, 0% was considered to indicate the absence of damage and 80–100% indicated total plant destruction (plant death). Finally, for the quantification of germination flow, the weeds of each species were counted at each evaluation point. For all three variables, the inventory square method was used.

To determine yield, the 3 central rows of each plot were manually harvested, discounting the front and 2 m border and 2 m front and final lines, totalizing an area of 12 m^2. This operation was performed when the grains had 15% moisture.

Immediately after harvest, the plots were threshed and the weights of the grains corrected to 13% moisture were measured. Finally, the data were extrapolated to kg ha^{-1}.

Statistical analysis

The use of Generalized Additive Models for Location, Scale and Shape (GAMLSS) with a Beta distribution inflated at 0 or 1 is justified by the nature of the response variable analysed—weed control rates expressed as proportions (values within the closed interval [0,1]). In such cases, traditional analysis of variance (ANOVA) may not be appropriate, as it assumes normality of residuals and homogeneity of variances, assumptions that are often violated when modelling proportional data, especially when values are concentrated at the extremes (0% or 100%) (Rigby *et al.* 2019; Palharani *et al.* 2023).

The Beta Inflated distribution of 0's or 1's is adequate to address percentage variables restricted to the [0.100%] interval, such as the weed control indices used in this study. This distribution is particularly useful when there is a high incidence of extreme values, as observed in cases where some treatments have 100% controls.

For all the statistical analyses, R *software* (R Core Team 2021) was used and for the construction of the GAMLSS models, GAMLSS libraries were used (Rigby *et al.* 2019). The estimates of the means and the respective standard errors were obtained using the *multcomp* and *emmeans* libraries (Lenth 2021). The results are presented in graphs generated using the *ggplot2* library (Wickham 2016).

Results

There was a significant effect ($P \leq 0.05$) of the herbicide × DAE for *D. insularis* (F = 57.158), *E. heterophylla* (F = 2.191), *C. benghalensis* (F = 0.923), *B. pilosa* (F = 0.748) and phytotoxicity (F = 0.580).

The statistical analysis of the *Caterpillar plots* resulted in the ranking of treatments, starting with the herbicides that resulted in the highest estimates and ending with the lowest. In addition, the corresponding standard errors are incorporated into this estimate.

For *D. insularis*, regardless of the evaluation period, all treatments resulted in more than 92% control (Fig. 2a), while for *E. heterophylla,* at 7 DAE, most treatments showed values lower than 80% (Fig. 2b).

At 14 DAE and 21 DAE, sulfentrazone + clomazone showed control greater than 80%. In the last evaluation period, sulfentrazone + clomazone, flumioxazin + imazethapyr, flumioxazin + pyroxasulfone, flumioxazin + imazethapyr + s-metallochlor and sulfentrazone + diuron resulted in superior control. at 80% (Fig. 2b).

In the final evaluation, several treatments demonstrated more than 80% effective control. These included sulfentrazone + clomazone, flumioxazin + imazethapyr, flumioxazin + pyroxasulfone, flumioxazin + imazethapyr + s-metolachlor and sulfentrazone + diuron.

Regarding the species *C. benghalensis* (Fig. 2c), all treatments provided effective control, with rates above 85% at all evaluation times. However, there was a slight reduction in the efficacy of sulfentrazone + imazethapyr at 28 DAE, with a mean control close to 90%.

For the species *B. pilosa* (Fig. 2d), almost all treatments achieved control above 90%, except for sulfentrazone + imazethapyr, which consistently showed efficacy below this mark.

The weed control means, shown in Fig. 3a, revealed that treatments sulfentrazone + clomazone, flumioxazin + imazethapyr, flumioxazin + pyroxasulfone, s-metolachlor + diclosulam and flumioxazin + imazethapyr + s-metolachlor maintained efficacies above 80%. Although the initial results showed a control of 95%, there was a reduction to 80% by the end of the evaluation for some treatments, as indicated in Fig. 3b. This suggests that although the initial control was high, the efficacy decreased with time. At 28 DAE, treatments sulfentrazone + diuron, s-metolachlor + fomesafen, s-metolachlor + metribuzin, sulfentrazone + imazethapyr and s-metolachlor + flumioxazin presented control lower than 80%.

For soybean phytotoxicity (Fig. 2e), initial symptoms were observed with scores of 6 to 9% at 7 DAE in all treatments. This index decreased gradually over time, reaching values below 2% at 28 DAE, which indicates a significant recovery of the culture. Regarding the species *I. purpurea*, the analysis did not reveal significant differences that could be attributed to the interactions between the herbicides and the evaluation periods (Table 3). As a result, it was not feasible to present a detailed graph of the percentage control for this species over time. Fig. 3 shows the *caterpillar plot* for the study between the levels of the treatments (Fig. 3a) and the regression fit for the control of the species *I. purpurea* as a function of the DAEs (Fig. 3b).

Regarding the number of plants that emerged in the field after herbicide application, there was a significant effect ($P \leq 0.05$) on the herbicide × DAE interaction, with the following effects on *D. insularis* (F = 3.019), *E. heterophylla* (F = 1.786), *C. benghalensis* (F = 3.106), *B. pilosa* (F = 2.425) and *I. purpurea* (F = 3.524) (Table 4).

Data on the emergence of *D. insularis* are detailed in Fig. 4a. At 7 DAE, unweeded control presented the greatest emergence, with approximately 2 plants/m², while the other treatments presented less than 0.5 plants/m². At 14 DAE, flumioxazin + imazethapyr and sulfentrazone + imazethapyr exhibited infestation densities above 1 plant/m², meanwhile control showed more than 2 plants/m². At 21 DAE, the infestation rate remained close to zero for most treatments, except for flumioxazin + imazethapyr, which had less than 1 plant/m². At 28 DAE, there was an increase in germination flow: flumioxazin + imazethapyr had more than 0.5 plants/m², s-metolachlor + metribuzin had 1 plant/m², unweeded control reached 2 plants/m² and sulfentrazone + imazethapyr reached 4 plants/m². Sulfentrazone + diuron and the other treatments maintained insignificant germination flows.

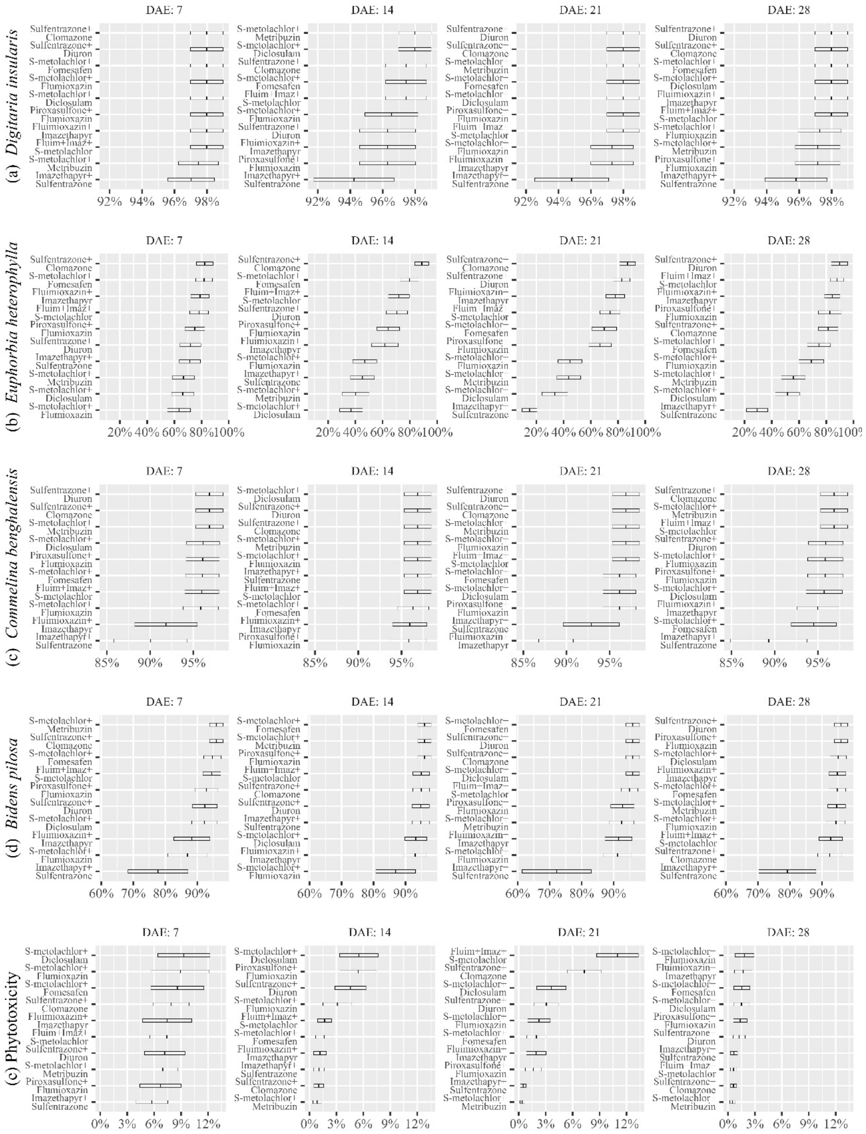

Fig. 2: *Caterpillar plots* of herbicide interactions across different days after emergence (DAE). Each point represents the estimated mean with its confidence interval, ranked in ascending order. Figures (**a**) – (**d**) show the percentage control of different weed species; (**e**) displays herbicide phytotoxicity on soybean

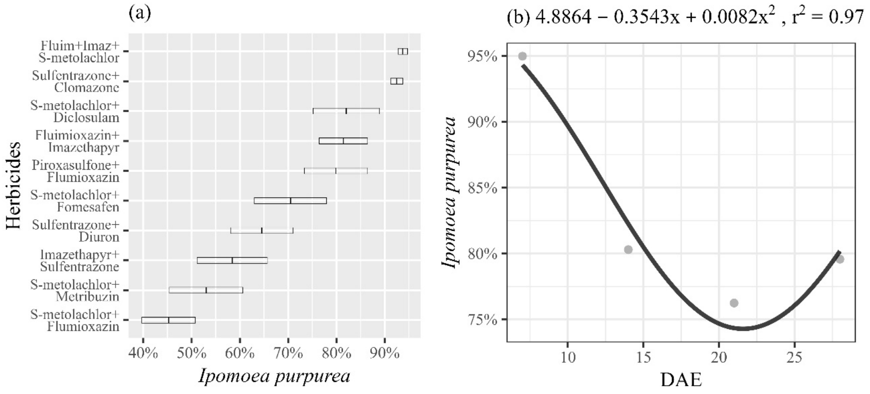

Fig. 3: *Caterpillar plot* showing the estimated means and confidence intervals for *I. purpurea*, based on the fitted beta regression model with a logit link function. Means are ranked in ascending order

Table 3: Results of the F statistic in the deviance analysis following the Shapiro–Wilk normality (SH) test and of the coefficient of variation (%) when the variables were evaluated

Variables	F statistic				SH	CV (%)
	Block	Herbicide (H)	DAE	H x DAE	Value p	
Digitaria insularis L.	1.922	7.667**	1477.546**	57.158**	0.140	16.25%
Euphorbia heterophylla L.	4.627**	7.099**	10.763**	2.191**	0.688	24.45%
Commelina benghalensis L.	3.235**	2.964**	37.212**	8.005**	0.923	17.43%
Bidens pilosa L.	1.747	1.619**	54.700**	8.782**	0.748	20.77%
Ipomoea purpurea L.	8.073**	4.721**	2.811**	1.339	0.006	27.67%
Phytotoxicity	2.323	1.545	371.354**	7.203**	0.580	96.07%

SH – Shapiro-Wilk normality; DAE – Days After Emergence; CV – Coefficient of Variation; **, significant according to the F test ($P < 0.05$)

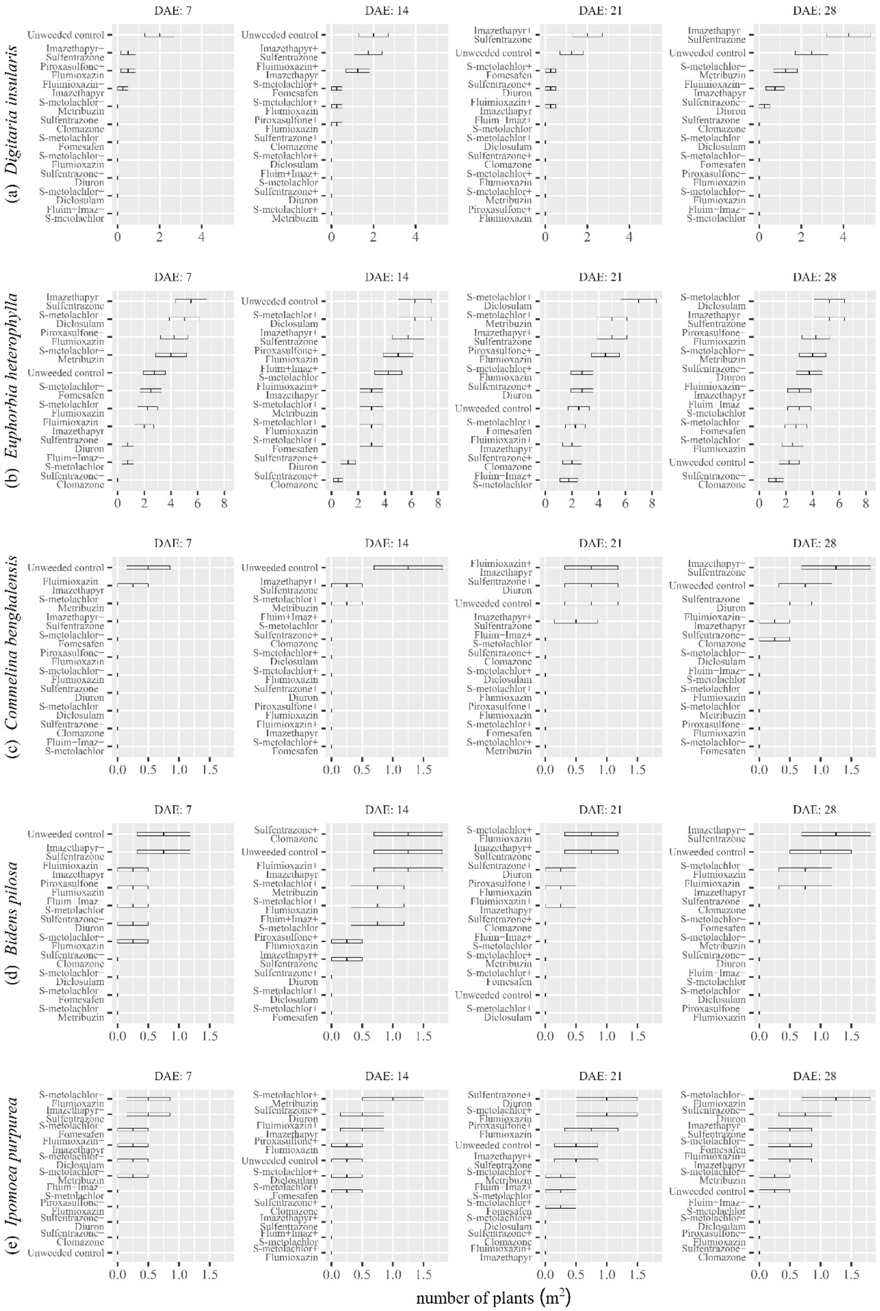

Fig. 4: *Caterpillar plots* illustrating herbicide interactions at each day after emergence (DAE) for the number of plants per m². Points indicate estimated means with confidence intervals, ranked in ascending order

Table 4: Results of the F statistic in the deviance analysis following the Shapiro–Wilk normality test (SH) and the coefficient of variation (%) when the number of plants (N m^{-2}) was evaluated

Variables	F statistic				SH	CV (%)
	Block	Herbicide (H)	DAE	H x DAE	Value *P*	
Digitaria insularis L.	1.574	3.158**	3.579**	3.109**	0.600	165.20%
Euphorbia heterophylla L.	3.049**	1.681	4.452**	1.786**	0.054	48.63%
Commelina benghalensis L.	3.754**	3.861**	5.644**	3.106**	0.931	197.62%
Bidens pilosa L.	0.995	2.736**	5.497**	2.425**	0.176	189.79%
Ipomoea purpurea L.	2.930**	2.911	3.346**	3.524**	0.168	143.31%
Total	2.289	3.219**	11.309**	2.320**	0.028	39.28%

**, significant according to the F test ($P < 0.05$)

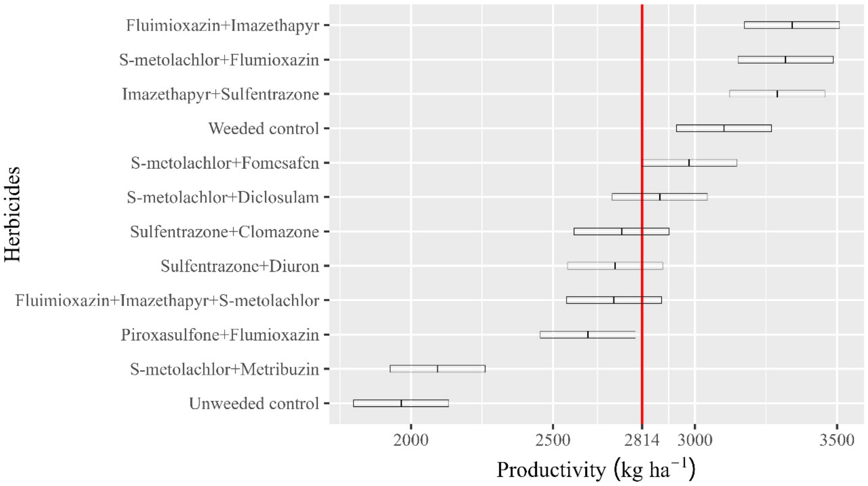

Fig. 5: *Caterpillar plot* of treatment effects on soybean yield. Points represent estimated means with standard errors, ordered from lowest to highest. The red line indicates the overall mean productivity

At 7 DAE, the emergence of *E. heterophylla* (Fig. 4b) was observed with flumioxazin + pyroxasulfone, s-metolachlor + diclosulam, s-metolachlor + metribuzin and sulfentrazone + imazethapyr, which presented more than 4 plants per m². Flumioxazin + imazethapyr, s-metolachlor + fomesafen, s-metolachlor + flumioxazin and unweeded control had more than 2 plants per m², meanwhile the emergence of sulfentrazone + clomazone was close to zero. The last evaluation at 28 DAE showed that s-metolachlor + diclosulam and sulfentrazone + imazethapyr had more than 5 plants/m², flumioxazin + pyroxasulfone and s-metolachlor + metribuzin had less than 4 plants/m², sulfentrazone + clomazone had more than 1 plant/m² and all other treatments had more than 2 plants/m².

For *C. benghalensis* (Fig. 4c), the germination flow was not very high throughout the study period. Initially, only flumioxazin + imazethapyr and unweeded control showed densities lower than 0.5 plants/m². This trend continued at 14 DAE, with s-metolachlor + metribuzin and sulfentrazone + imazethapyr registering less than 0.5 plants/m² and T1 (absolute control) exhibiting a slight increase to 1.25 plants/m². At 21 DAE, plant emergence was observed at flumioxazin + imazethapyr, sulfentrazone + diuron, sulfentrazone + imazethapyr and absolute control, all exceeding 0.5 plants/m². In the last evaluation at 28 DAE, sulfentrazone + imazethapyr and absolute control showed densities greater than 1 and 0.75 plants/m², respectively. Sulfentrazone + clomazone and flumioxazin + imazethapyr maintained values lower than 0.5 plants/m², while plant emergence did not occur in any of the other treatments.

For *B. pilosa* (Fig. 4d), plant emergence throughout the evaluated periods showed varied patterns among treatments. At 7 DAE, sulfentrazone + imazethapyr and absolute control presented 0.75 plants/m², while flumioxazin + pyroxasulfone, flumioxazin + imazethapyr + s-metolachlor, sulfentrazone + diuron and s-metolachlor + flumioxazin recorded less than 0.5 plants/m². At 14 DAE, sulfentrazone + clomazone, flumioxazin + imazethapyr and absolute control had an infestation of 1.25 plants/m², while flumioxazin + imazethapyr + s-metolachlor, s-metolachlor + metribuzin and s-metolachlor + flumioxazin had an infestation of 0.75 plants/m². Flumioxazin + pyroxasulfone and sulfentrazone + imazethapyr maintained infestations lower than 0.5 plants m^{-2}. At 21 DAE, sulfentrazone + imazethapyr and s-metolachlor + flumioxazin had a density of 0.75 plants/m² and flumioxazin + imazethapyr, flumioxazin + pyroxasulfone and sulfentrazone + diuron had a density less than 0.5 plants/m². At 28 DAE, imazethapyr + sulfentrazone and absolute control culminated in 1.25 and 1.00 plants/m², respectively, while flumioxazin + imazethapyr and s-metolachlor + flumioxazin presented 0.50 plants/m².

For *I. purpurea* (Fig. 4e), at 7 DAE, imazethapyr + sulfentrazone and s-metolachlor + flumioxazin presented 0.50 plants/m², while the majority of the other treatments had no germination flow. At 14 DAE, s-metolachlor + metribuzin reached 1 plant/m², meanwhile unweeded control reached 0.25 plants/m². The other treatments did not affect the germination flow. At the end, 0.75 and 1.25 plants/m² were recorded for sulfentrazone+diuron and s-metolachlor+ flumioxazin, respectively. Flumioxazin + imazethapyr, s-metolachlor + fomesafen and imazethapyr + sulfentrazone had 0.50 plants/m², while s-metolachlor + metribuzin had 0.25 plants/m². The other treatments had no germinal flow (Fig. 4e).

For the analysis of productivity, a normal distribution with logarithmic linkage was used. Deviance analysis revealed a significant effect of treatment on yield (F = 4.901; $P < 0.001$). The Shapiro–Wilk test with $P = 0.471$ confirmed the adequacy of the normal distribution to the model residuals. The coefficient of variation was 12.99%.

As shown in Fig. 5, treatments flumioxazin + imazethapyr, sulfentrazone + imazethapyr and s-metolachlor + flumioxazin achieved the highest yields, with values above 3250 kg ha^{-1}. The weeded control, s-metolachlor + fomesafen and s-metolachlor + diclosulam treatments resulted in yields above 2,600 kg ha^{-1}. Flumioxazin + pyroxasulfone, flumioxazin + imazethapyr + s-metolachlor, sulfentrazone + diuron and sulfentrazone + clomazone produced more than 2,500 kg ha^{-1}. Finally, s-metolachlor + metribuzin and weeded control presented yields close to 2,000 kg ha^{-1}.

Discussion

None of the pre-emergence herbicide treatments caused significant phytotoxic effects on soybean plants, confirming the selectivity of the products used. This result emphasizes the importance of applying pre-emergence herbicides, which facilitate crop establishment without weed interference and exhibit lower phytotoxicity compared to certain post-emergence herbicides. This behaviour is anticipated, as some of the herbicides used in this study, such as flumioxazin and diuron, demonstrate selectivity due to their position in the soil (toponomic), where the herbicide-treated zone does not come into contact with the soybean root system because of the herbicide's spatial and/or temporal placement relative to the plant, leading to high selectivity (Silva *et al.* 2018).

The availability of herbicides in the soil was influenced by their interaction with the percentage of organic matter and the cation exchange capacity (CEC) of 13.82 cmol dm^{-3}, since soils with higher organic matter content and high CEC tend to increase herbicide adsorption reducing their concentration in the soil solution. Therefore, this phenomenon can decrease their availability in the soil solution, thereby, limiting absorption by the target plants and limiting the herbicides efficiency. According to Copaja and Sepúlveda (2022), variations in clay and organic matter levels directly influence herbicide adsorption in the soil.

Additionally, herbicides solubility played a crucial role in their movement within the soil profile. Following application, heavy rainfall (27.4 mm shortly after sowing) promoted greater leaching of more soluble compounds, such as sulfentrazone + clomazone and sulfentrazone + imazethapyr, which have solubilities of 110 mg L^{-1} (pH 6.0) + 1100 mg L^{-1} (25°C) and 1400 mg L^{-1} (25°C) + 110 mg L^{-1} (pH 6.0), respectively (PPDB 2024). This displacement may reduce herbicide concentration in the upper soil layer, where soybean seeds germinate, resulting in the lowest phytotoxicities observed in all evaluation periods.

In weed management, an herbicide is deemed effective if it achieves a control rate of over 80%, as stated by Oliveira, Freitas and Vieira (Oliveira *et al.* 2009). At 28 DAE, treatments flumioxazin + imazethapyr + s-metolachlor, flumioxazin + imazethapyr, flumioxazin + pyroxasulfone and sulfentrazone + clomazone demonstrated acceptable control for all weed species assessed, including *B. pilosa*, *D. insularis*, *C. benghalensis, E. heterophylla and I. purpurea*, with efficacy rates exceeding 80%. These results highlight these treatments as viable options for managing complex and difficult-to-control species.

The observed efficacy can be linked to the inclusion of protoporphyrinogen-inhibiting herbicides, such as flumioxazin and sulfentrazone and the combination of herbicides with complementary control spectra. These combinations include herbicides targeting both monocots and eudicots, broadening the spectrum of control. Noteworthy examples are the combinations of flumioxazin + s-metolachlor and flumioxazin + pyroxasulfone for eudicots and sulfentrazone + clomazone for monocots, demonstrating the effectiveness of this integrated weed management strategy.

Furthermore, irrespective of the evaluation period or treatment applied, *D. insularis* control rates in the study exceeded 90%. This outcome reflects the effectiveness of herbicides that include products with a broad spectrum of action on monocots, such as clomazone, imazethapyr and s-metolachlor (Agrofit 2024). Research conducted by Drehmer *et al.* (2015), Bauer *et al.* (2021) and Gazola *et al.* (2021) confirmed this effectiveness, with exceptional control rates reported from using s-metolachlor and the combination of imazethapyr + flumioxazin, with control rates nearing 100%. Additionally, Bottcher *et al.* (2022) noted that optimal control of *D. insularis* is achieved through herbicide combinations, particularly imazethapyr + saflufenacil.

Considering the germination patterns of *D. insularis*, both the untreated control and the sulfentrazone + imazethapyr treatments gradually increased the presence of this weed in the field. This finding underscores the critical role of pre-emergence herbicides in limiting *D. insularis* growth after soybean emergence, especially in scenarios where resistance to EPSPs and ACCase herbicides is widespread, reducing post-emergence control options (Palharani *et al.* 2023; HRAC Global 2024).

In the case of *E. heterophylla* management, treatments with PPO-inhibiting herbicides such as sulfentrazone and flumioxazin exhibited high effectiveness, as these compounds are known for controlling a wide range of weed species. Although the efficacy of flumioxazin showed a slight decrease after 30 days, its persistence highlights the durability of these herbicides in field conditions (Gazola *et al*. 2021). Similarly, sulfentrazone maintained its effectiveness over a longer period, reinforcing the importance of selecting herbicides with long residual activity for integrated weed management programs (Shaner 2014).

While PPO inhibitors proved highly effective, combinations of ALS-inhibiting herbicides, such as imazethapyr + imazapic, encountered challenges in controlling resistant *E. heterophylla* biotypes. This reflects the growing issue of herbicide resistance and emphasizes the need for alternative strategies (Adegas *et al*. 2023). Effective *E. heterophylla* control requires herbicides with strong residual activity, particularly PPO inhibitors. However, a reduction in residual efficacy over time is important factor in weed management, as noted by Khalil *et al*. (2019), who highlighted that soil herbicide degradation can impact long-term performance, reinforcing the need for continuous monitoring and adjustment of management strategies.

In addition, *E. heterophylla* showed notable germination patterns in treatments involving s-metolachlor + diclosulam and sulfentrazone + imazethapyr, likely due to the presence of ALS-inhibiting herbicides. This species was the first to develop herbicide resistance in Brazil, particularly in Mato Grosso do Sul (HRAC Global 2024) and resistance is now widespread. The necessity of combining different herbicides and adjusting dosages has been demonstrated, as Castro *et al*. (2023) successfully controlled *E. heterophylla* with a mixture of sulfentrazone and diclosulam combined with ametryn and mesotrione. However, Pereira *et al*. (2022) found that the sole use of ALS inhibitors, such as imazethapyr, was ineffective in controlling this weed.

The consistent control of *C. benghalensis* across all treatments highlights the importance of pre-emergence management, addressing both seed and vegetative propagation. The use of herbicide combinations has been vital in broadening the control spectrum and enhancing overall management success. Bottcher *et al.* (2022) emphasized the significance of herbicide mixtures for managing *C. benghalensis* Additionally, Silva *et al*. (2019) indicated that pre-emergence herbicides like indaziflam are more effective in areas with lower vegetative cover, where reduced ground cover of *C. benghalensis* allows better herbicide-soil contact.

In the case of *B. pilosa*, the exception to effective control by T5 (sulfentrazone + imazethapyr) suggests specific difficulties. This finding is significant, particularly given the well-established efficacy of sulfentrazone. The reduced effectiveness could be attributed to the resistance of *B. pilosa* to ALS-inhibiting herbicides, such as imazethapyr, which was evident in the naturally infested area. Pereira *et al.* (2022) observed that for *B. pilosa* biotypes resistant to ALS inhibitors, imazethapyr alone was insufficient and combining it with herbicides like glyphosate was necessary for achieving adequate control.

Regarding soybean yield, Soltani *et al.* (2022) emphasized the potential economic losses linked to delayed weed control, showing that weed presence for 30 days can reduce productivity by 5%, even under low infestation levels. Gazola *et al*. (2021) reinforced these observations, highlighting the effectiveness of pre-emergence residual herbicides in reducing resource competition and preventing yield losses. Furthermore, these herbicides extend the period before interference (PAI), the timeframe during which weeds can coexist with soybean without negatively affecting yields (Rizzardi *et al*. 2020).

Therefore, the use of herbicides before soybean sowing plays a vital role in minimizing weed germination cycles. The appropriate selection of pre-emergence herbicides should balance effective weed control with selectivity for the soybean crop. As demonstrated in this study, pre-emergence herbicide applications reduced competition between weeds and soybeans, leading to increased yields and decreasing the need for post-emergence interventions, significantly reducing selection pressure for resistant biotypes. Additionally, herbicide selection should take into account the area's history and promote the combination of products with different modes of action, aiming to avoid the repetitive application of the same herbicide and consequently, increasing the risk of resistance incidence in the area. This approach not only mitigates the risk of resistance but also broadens the control spectrum, addressing both monocot and eudicot weeds.

Conclusion

Pre-emergence combinations such as flumioxazin + imazethapyr + s-metolachlor, flumioxazin + imazethapyr, pyroxasulfone + flumioxazin and sulfentrazone + clomazone effectively controlled *E. heterophylla*, *D. insularis*, *C. benghalensis* and *B. pilosa* without causing phytotoxicity in soybean. These combinations also reduced weed emergence, except for *D. insularis*, where imazethapyr + sulfentrazone, s-metolachlor + metribuzin and flumioxazin + imazethapyr were more effective. Mixtures such as flumioxazin + imazethapyr, sulfentrazone + imazethapyr and s-metolachlor + flumioxazin favoured yield without significant phytotoxic effects.

Acknowledgements

Nothing.

Author Contribution

FCM, RPPNB and PAVS worked on performing the experiments and collecting data; PVS, ESM, BFS, PAM, MM, RDCD, MFO, EPW and TEM worked on performing the data, analysis, implementation of the computational models, preparing the first version of the manuscript, conducting a literature review, making corrections to the manuscript.

Conflicts of Interest

The authors declare that they have no conflicts of interest related to this manuscript.

Data Availability

The data that support the findings of this study are available from the corresponding author, Bruna Ferrari Schedenffeldt, upon reasonable request.

Ethics Approval

Not applicable to this study.

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