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**Comparative Evaluation of Nicosulfuron and Fluroxypyr
for Weed Management, Crop Productivity, and Residue
Safety in Maize (*Zea mays* L.) under Field Conditions**

Prepared By

Ibrahim M. Ammar, Mahmoud H. Rashwan

Samah S. Arafa, Mahmoud M. Lila

Department of Pesticides, Faculty of Agriculture, Menoufia University,
Shibin El Kom, Menoufia , Egypt

Hanim M. Soliman

Central Agricultural Pesticide Laboratory, Agricultural Research Center,
Dokki, Giza, Egypt.

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Abstract:

Nicosulfuron and Fluroxypyr are herbicides with different mechanisms of action and are recommended for weed control in Maize (*Zea mays* L.). This study aimed to evaluate and compare the effect of both herbicides, at recommended concentrations, on physiological and agronomic characteristics in maize. Field trials were conducted during the 2023 summer season to evaluate dominant weed flora, herbicide efficacy, and their effects on Maize growth, yield, grain quality, and residue levels. The weed community was dominated by *Portulaca oleracea*, *Chenopodium album*, *Echinochloa colonum*, and *Phalaris* spp., with a temporal shift from broadleaf to grassy dominance between 60 and 80 days after sowing (DAS). Herbicide evaluation showed that nicosulfuron (0.4 L/feddan) provided the most effective broad-spectrum control, achieving weed control efficiency (WCE) of up to 96.02% at 60 DAS and maintaining 89.41% at 80 DAS, with strong suppression of both grassy and broadleaf weeds. Fluroxypyr (0.2 L/Feddan) significantly reduced weed biomass but was less effective against grassy species. Both herbicides markedly enhanced Maize growth and yield compared to the untreated control. Nicosulfuron nearly doubled grain yield (4.27 vs. 2.35 tons/Feddan) and increased straw yield (22.76 vs. 13.82 tons/Feddan), outperforming fluroxypyr across all agronomic traits. Grain quality also improved, with nicosulfuron-treated maize recording the highest protein (11.13%) and oil (5.14%) contents. Residue analysis

confirmed safe levels, with preharvest intervals of 90 days for nicosulfuron and 30 days for fluroxypyr, both within permissible maximum residue limits. Overall, nicosulfuron proved superior to fluroxypyr, offering effective, broad-spectrum weed management, enhanced crop productivity, improved grain quality, and residue safety under field conditions.

Keywords:

Nicosulfuron, Fluroxypyr, Maize, *Weed Management*, *Crop Productivity*, *Residue Safety*

1. Introduction:

Maize (*Zea mays* L.) is one of the most important cereal crops in the global agricultural economy. It serves as a staple food, a key source of green forage, and a vital industrial crop used in starch, glucose, and edible oil production. More importantly, 70–80% of global maize production is devoted to animal feed, making it the principal energy ingredient in livestock and poultry diets (Alves et al., 2015). In developing countries, maize is also a major source of income for farmers, ranking third after wheat and rice in cereal production.

In Egypt, maize cultivation covers approximately 1.027 million hectares (2.536 million feddan), with an average yield of 2.957 tons/Feddan, producing about 7.5 million metric tons annually (FAO, 2021). However, local production does not fully meet domestic demand (FAO, 2017). Efforts to increase maize productivity have focused on horizontal expansion in newly reclaimed lands, which requires high costs, and vertical expansion through adopting high-yielding varieties tolerant to biotic and abiotic stresses. Yield instability is often linked to late planting, which exposes maize to high temperatures during pollen fertilization and grain filling, further aggravated by the impacts of climate change (Ali et al., 2015).

Among the major constraints limiting maize productivity, weed infestation is particularly serious. Weeds compete with maize for water, nutrients, light, and space, and may also exert allelopathic effects, leading to significant yield losses. Previous studies reported 33–91% maize yield reductions due to weed competition in untreated fields compared with herbicide-protected plots (Pannacci & Onofri, 2016). Other estimates indicated losses of about 50% (Abouziena et al., 2013) and 33.7% (Saudy, 2013). Maize is especially sensitive to weed competition, with yield losses reaching 65%

depending on the weed density and duration of infestation (**Gantoli et al., 2013; Ghanizadeh et al., 2014**).

Manual weeding, though effective, is labour-intensive, costly, and increasingly limited by labor shortages. Herbicides, therefore, represent a more practical and economical alternative, offering timely and effective weed control (**Ishrat et al., 2012**). While several pre-emergence herbicides are available for Maize (**Singh et al., 2015**), fewer options exist for post-emergence application. Modern agriculture relies heavily on selective herbicides as part of integrated weed management to ensure high yields and grain quality in cereal crops (**Boanta et al., 2020; FAO, 2020**).

Nicosulfuron(2-[(4,6-dimethoxypyrimidin-2-yl) carbamoylsulfamoyl]-N,N-dimethylpyridine-3-carboxamide), a sulfonylurea herbicide, has become one of the most widely used post-emergence herbicides in maize production worldwide. It is highly efficient, selective, and effective at low doses (**Lum et al., 2005**). As an acetolactate synthase (ALS) inhibitor, it disrupts the biosynthesis of branched-chain amino acids (valine, leucine, and isoleucine), ultimately blocking protein formation and plant growth (**EFSA, 2012; HRAC, 2012**). Its degradation in soils and waterways occurs mainly through hydrolysis and microbial activity, reducing toxicity risks for subsequent crops (**Xu et al., 2022**).

Fluroxypyr (4-amino-3,5-dichloro-6-fluoro-2-pyridyloxyacetic acid), a pyridine herbicide, is another post-emergence option primarily used against broadleaf weeds in cereals, olive trees, and other croplands (**Carvalho et al., 2008; Hellou et al., 2009; Sdiri et al., 2013**). Studies indicate that fluroxypyr provides effective broadleaf weed control while being relatively safe for maize and winter wheat (**Liu, 2014**). Crop tolerance to fluroxypyr is linked to metabolic detoxification pathways that convert herbicide molecules into non-toxic metabolites (**Nicolae & Aurlan, 2015**). Given the importance of effective weed management in maize production, this study was undertaken to evaluate the efficacy of post-emergence herbicides—nicosulfuron and fluroxypyr on weed suppression, maize growth, yield, grain quality, and residue safety under field conditions.

2. Materials and Methods

2.1. Experimental site and soil analysis

A field experiment was carried out during the summer season of 2023 at Zaweit Razen, Menouf, Menoufia, Egypt (30°25'13.8"N, 30°51'16.9"E). Soil analysis showed the following characteristics: clay 47.50%, silt 32.35%, sand 20.15%, soil texture SiCL, pH 7.82, electrical conductivity (EC) 0.31 dS m⁻¹, and organic matter 2.3%. Based on these results, the site was suitable for maize production.

The experimental area was ploughed twice, harrowed, and leveled before sowing. Calcium superphosphate (15.5% P₂O₅) and potassium sulphate (48% K₂O) were applied during soil preparation at the rates of 115.2 and 28.8 kg/Feddan, respectively.

2.2. Experimental design and treatments:

The experiment was conducted using a randomized complete block design (RCBD) with three replications. Each plot measured 30 m × 5 m (150 m²). White single cross hybrid maize (SC 2031; Misr Hy Tech Seed Int.) was used. Maize grains were sown on May 21, 2023, at a seeding rate of 36 kg/Feddan, with two grains per hill at 20 cm spacing on one side of the ridge. Plants were thinned to one per hill prior to the first irrigation to obtain a final population of 24000 plants/Feddan.

Nitrogen fertilizer (urea 46% N) was applied at 68.5 kg N/Feddan in two equal splits, before the first and second irrigations. Herbicides were sprayed at 21 days after maize emergence (2–4 leaf stage) using a “Knapsack hand sprayer CP3, 20 L” equipped with a flat-fan nozzle calibrated to deliver 200 L/Feddan. Harvest was performed on September 25, 2023. The preceding crops were wheat (*Triticum aestivum*) and faba bean (*Vicia faba*) in the first and second seasons, respectively. The experimental treatments were as follows: Active Ultra (Nicosulfuron 4% OD, 400 cm³/feddan), Starane (Fluroxypyr 20% EC, 200 cm³/feddan), and Untreated check.

2.3. Recorded data:

2.3.1. Survey of weed flora

Weed species were collected from one randomly selected square meter in each plot, hand-pulled, and identified at the species level using the weed

identification manual of Täckholm (1974). Identified species were classified into two groups: broad-leaved weeds and grassy weeds.

2.3.2. Weed density:

Weed density (narrow- and broad-leaved species) was recorded at 60 and 80 days DAS. Counts were made from one square meter in each plot.

2.3.4. Weed parameters:

Observations on the growth characteristics of narrow- and broad-leaved weeds were recorded from plants randomly selected in each replicate plot. Measurements were taken at 60 and 80 DAS within one square meter.

Total fresh weight of weeds: At 60 and 80 DAS, weeds were uprooted, washed free of soil, and weighed immediately to determine fresh biomass.

Total dry weight of weeds: Fresh weeds were air-dried for 48 h, then oven-dried at 70 °C for 24 h (Abo El-Hassan, 2016). Dry weights of grasses and broad-leaved weeds were recorded separately.

Weed control efficiency (WCE): compared with the untreated control, following **Thakur (1994)**: $WCE = (DCW - DWT) / DCW \times 100$.

where **WCE** = weed control efficiency, **DCW** = dry weight of weeds in the control plot, and **DWT** = dry weight of weeds in the treated plot

2.4. Herbicide residues in grains:

2.4.1. Chemicals and Reagents:

Nicosulfuron was from Mecca Co. For Agricultural Development, fluroxypyr was from Corteva Agriscience. HPLC-grade methanol and acetonitrile were purchased from Sigma (Sigma GmbH, Darmstadt, Germany). Primary secondary amine (PSA, 40 µm Bondesil) and graphitized carbon black sorbent were bought from Supelco (Bellefonte, Pennsylvania, USA). Analytical grade of anhydrous magnesium sulfate and sodium chloride was obtained from CARLO ERBA Reagents S.A.S.

2.4.2. Preparation of standard solutions:

To conduct an HPLC analysis, a 100 ml/L mixed stock solution of nicosulfuron and fluroxypyr was prepared in acetonitrile. To set up consecutive working dilution and spike standard solution, the stock solution

was diluted accordingly. All standard and working solutions were stored at 4 °C.

Maize grain samples were collected 30 days after the application of Fluroxypyr and 90 days following the application of Nicosulfuron to assess the residual levels of both herbicides at the end of their respective pre-harvest intervals. The collected samples were immediately placed in polyethylene bags and transferred to the laboratory in an ice box. The samples were then roughly cut into small portions and homogenized in a food processor (HOBART). The homogeneous matrix was stored in a sealable plastic bag at -20°C until the preparation day.

2.4.3. Sample extraction and clean-up:

During the extraction and clean-up process, the unique virgin QuEChERS method developed by **Anastassiades et al. (2003)** was used with full authority. To start, 5g of homogenized maize in addition to distilled water and weighed and placed in 50ml Teflon tubes. Next, 10 ml of acetonitrile was added and the mixture was shaken vigorously for 1 minute. After that, 4.0 g of anhydrous magnesium sulfate and 1.0 g of sodium chloride were added and shaken again for 1 minute. The tube was then immediately centrifuged at 4000 rpm for 5 minutes in a 5°C refrigerated centrifuge. Then, 1ml of the supernatant was subjected to a clean-up process using 25 mg primary secondary amine, 150 mg anhydrous magnesium sulfate, and 10 mg GCB. The tube was shaken vigorously for 1 minute and then centrifuged at 4000 rpm for another 5 minutes. Finally, 0.5 ml of the supernatant was transferred to a vial after being filtered through a 0.22µm PTFE filter (Millipore, Billerica, MA). The filtered liquid was then injected into a High-Performance Liquid Chromatography system.

2.4.4. Method validation:

In this experiment, we aimed to expound that our method is strongly suitable for extracting and quantitatively determining the levels of nicosulfuron and fluroxypyr in maize. We validated our analytical method according to SANTE/12682/2019 guidance, which included assessing matrix effects, accuracy, LOQ, precision, linearity, and trueness (bias). To define the effectiveness of our technique, we established its linearity constructed on the concentration of nicosulfuron and fluroxypyr, which we diluted in a solvent. We evaluated the resulting correlation coefficient (R²)

using a 5-point calibration curve series (0.01, 0.1, 0.5, 1.25, 2.5, and 5) $\mu\text{g/ml}$ for HPLC analysis. To confirm the matrix match effect, we compared the response from the nicosulfuron and fluroxypyr in a pure solvent with the spiked from nicosulfuron and fluroxypyr in the blank matrix (maize) samples in the same solvent following extraction at the same concentration points (0.01, 0.1, 0.5, 1.25, 2.5, and 5) mg/kg for HPLC analysis.

This step was crucial to ensure the accuracy and reliability of the results obtained. The LOQ is a vital tool in accurately measuring the lowest concentration of nicosulfuron and fluroxypyr in maize with high levels of precision and trueness. By following the defined analytical technique of S/N ratio of 10:1, we can confidently determine the LOQ, which guarantees the accuracy and reliability of our measurements. This information is crucial to ensure the quality of our research and helps us make informed decisions with confidence. On the authority of the document SANTE/12682 (2019). The limit of quantification must be less than or equal to the maximum residue limit (MRL) (0.01 mg/kg) for nicosulfuron in maize and fluroxypyr, and (0.05 mg/kg) , according to the (European Union's regulations of 2016). As part of the testing process, we conducted a recovery experiment to determine the accuracy of our results. We analyzed five replicates of a blank sample spiked with nicosulfuron and fluroxypyr at three different levels (0.01, 0.1, and 1 mg/kg) in maize. The SANTE/11813/2017 document specifies that acceptable mean recoveries should fall between 70% and 120%. To evaluate our precision, we used the standard deviation (RSD) for repeatability (r). We performed a similar method on similar samples in the lab over a short period. The maximum allowable limit for the relative standard deviation of repeatability (RSDr) was set at $\leq 20\%$. We tested five replicates at three different recovery levels (0.01, 0.1, and 1 mg/kg) for a day and repeated the process over three different days to ensure precision.

2.4.5. HPLC determination:

The HPLC (Agilent 1260 infinity series) system was used to perform chromatographic analyses. This system was equipped with a quaternary pump, variable wavelength diode array detector (DAD), and an analytical column: Nucleosil C18 (30 $\times$ 4.6 mm (i.d.) $\times$ 5 μm film thickness) with an auto sample valve. Chromatographic separation was achieved using a mobile phase of acetonitrile: water (70:30 v/v) at a flow rate of 1 ml/min . The injection volume was 20 μl and the wavelength used was 230 nm. The

retention time obtained was 5.52 min for nicosulfuron and 7.21 min for fluroxypyr.

2.4.6. Decomposition rate:

The dissipation kinetics of nicosulfuron and fluroxypyr residues in maize and were determined by plotting residue concentration versus pass time after application and equations of the best curve fit with maximum coefficients (R^2) were determined. For the dissipation of azoxystrobin and epoxiconazole in beans and zucchini, exponential relationships were found to be applicable corresponding to the general first-order kinetics equation: $C_t = C_0 e^{-kt}$ Where C_t symbolized the concentration of the pesticide residue at the time of t , C_0 symbolized the initial deposits after application and k is the constant rate of pesticide dissipation per day (Wang and Hoffman, 1991). The half-life periods (RL50) of azoxystrobin and epoxiconazole were calculated as follows: $(\ln 2/k)$ Moye et al. (1987). The following equation calculated the degradation percentage: $\% \text{degradation} = (C_0 - C_t) / C_0 \times 100$ where C_0 is the concentration of the pesticide (ppm) at 0 time and C_t is the concentration of the pesticide (mg/kg) during time

2.5. Maize characters:

2.5.1. Yield and yield components:

At harvest (September 25, 2023), random samples of maize plants were collected from each plot, and various agronomic traits were recorded. These included plant height (cm) measured from the soil surface to the tassel tip, ear length (cm), number of rows per ear, number of leaves per plant, ear diameter (cm), 100-grain weight (g), number of ears per plant, grain yield (ton/feddan), and straw yield (ton/feddan).

2.6. Chemical composition of maize grains:

2.6.1. Crude protein (%):

Oven-dried maize grains were ground (50-mesh). Samples were digested with concentrated H_2SO_4 and 30% H_2O_2 (Yash, 1998). Nitrogen was determined by the micro-Kjeldahl method (Jackson, 1967), and crude protein was calculated as: Grain protein content was estimated by multiplying N (%) in maize grains $\times 6.25$.

2.6.2. Oil content (%):

Grain oil percentage was determined by Soxhlet extraction according to AOAC (1990).

2.6.3. Carbohydrates (%):

Carbohydrate content was estimated following Dubois et al. (1956). Briefly, 0.01 g seed powder was mixed with 3 ml of 10% NaOH, boiled for 10 min, and filtered. An aliquot (0.5 ml) was mixed with 0.5 ml of 5% phenol reagent, followed by 2.5 ml concentrated H₂SO₄. After incubation at room temperature for 30 min, absorbance was read at 490 nm using glucose as a standard. Carbohydrate percentage was calculated as:

Carbohydrate (%) = $F(\text{mg}) \times \text{Extract volume (ml)} \times \text{Aliquot (ml)} \times \text{Sample weight (g)} \times 100$.

Where **F** = carbohydrate concentration (mg) obtained from the glucose standard curve.

2.7. Statistical analysis:

All analyses were performed in triplicate. Data were analyzed using one-way ANOVA followed by Tukey's test with SPSS software (version 20.0; SPSS Inc., Chicago, IL, USA). Results were expressed as mean $\pm$ SD, and significance was set at $p \leq 0.05$.

3. Results

3.1. Dominant weed flora in the maize field:

During the summer season of 2023, field observations in the Maize trial revealed the presence of dominant annual weed species comprising both broadleaf and grassy types, as demonstrated in **Table 1**. Among the broadleaf annual weeds, *Portulaca oleracea* (commonly known as ("Al-Rajlah"), belonging to the family Portulacaceae, and *Chenopodium album* L. (commonly known as "Zorbih" from the **Amaranthaceae** family, were the most prevalent. As for the **grassy weeds**, two dominant species were identified: *Echinochloa colonum* (Jungle rice or "Abu-Rokbah") and *Phalaris* spp. (Canary grass), both of which belong to the **Poaceae** family. These species constituted the major weed flora competing with maize during the trial period and represent key targets for weed management strategies.

Table 1: Dominant annual broadleaf and Narrow-Leaved weeds species observed in the maize trial during the summer season of 2023.

Common Name	Scientific Name	English Name	Family Type
Al-Rajlah	<i>Portulaca oleracea</i>	Purslane	Portulacaceae
Zorbih	<i>Chenopodium album</i> L.	White Goosefoot	Amaranthaceae
Abu-Rokbah	<i>Echinochloa colonum</i>	Jungle Rice	Poaceae
Phalaris	<i>Phalaris spp.</i>	Canary Grass	Poaceae

3.2 Dynamics of weed density and composition at 60 and 80 DAS:

Table 2 illustrates the percentage and density of dominant weed species at 60 and 80 DAS. At **60 DAS**, *Portulaca oleracea* recorded the highest density at 110.67 plants/m², accounting for 58.45% of the total weed population. This was followed by *Echinochloa colonum* at 62.33 plants/m² and 32.92%, while *Chenopodium album* and *Phalaris* spp. contributed lower percentages and densities of **4.93%** (9.33 plants/m²) and 3.7% (7 plants/m²), respectively. By 80 DAS, *Echinochloa colonum* became the most dominant weed, increasing to 152 plants/m² and comprising 50.92% of the weed flora. *Portulaca oleracea* followed with a density of 98.5 plants/m² and 33%, while *Phalaris* spp. and *Chenopodium album* represented smaller proportions, with densities of 23 and 25 plants/m² and percentages of 7.71% and 8.38%, respectively. These results indicate a dynamic shift in weed species dominance over time, with grassy weeds becoming more prevalent as the Maize crop matured.

Table ٢: Percentage and density of dominant weed species at 60 and 80 DAS in the experimental field.

Weed	DAS			
	60		80	
	Density Plants/ m ²	Percentage %	Density Plants/ m ²	Percentage %
<i>Portulaca oleracea</i>	110.67	58.45	98.5	33
<i>Chenopodium album</i>	9.33	4.93	25	8.38
<i>Echinochloa colonum</i>	62.33	32.92	152	50.92
<i>Phalaris spp.</i>	7	3.7	23	7.71

DAS: Days After Sowing

3.3 Effectiveness of herbicide treatments on fresh weed biomass and WCE%:

Table 3 presents a comparative analysis of herbicide efficacy at 60 and 80 DAS, respectively, based on WCE% and fresh weed biomass. At 60 DAS, the nicosulfuron treatment (0.4 L/feddan) exhibited the highest effectiveness, achieving a WCE of 96.02% and reducing total fresh weed biomass to 107.67 g/m² compared to 2705.51 g/m² in the untreated control. Nicosulfuron demonstrated broad-spectrum activity, effectively suppressing both grassy weeds (89.20% WCE, 80.79 g/m²) and broadleaf weeds (98.62% WCE, 26.88 g/m²). The reductions achieved by nicosulfuron were significantly different ($p < 0.05$) from both the control and fluroxypyr. Similarly, at 80 DAS, nicosulfuron maintained its superior efficacy, reducing total fresh weed biomass to 332.19 g/m² with an overall WCE of 89.41%, controlling grassy weeds at 85.32% WCE (178.30 g/m²) and broadleaf weeds at 93.49% WCE (153.89 g/m²). These effects were also statistically significant compared to the control.

In contrast, the fluroxypyr treatment (0.2 L/feddan) also significantly reduced weed biomass but was less effective than nicosulfuron. At 60 DAS,

fluroxypyr achieved an 87.42% overall WCE with 340.37 g/m² of total biomass, showing higher efficacy against broadleaf weeds (96.31% WCE, 72.07 g/m²) than grassy weeds (64.12% WCE, 268.30 g/m²). These differences were significant compared to the control but lower than those of nicosulfuron. This trend persisted at 80 DAS, where fluroxypyr achieved a WCE of 73.3% and reduced total biomass to 765.23 g/m², again showing stronger control of broadleaf weeds (89.88% WCE, 239.53 g/m²) over grassy weeds (56.72% WCE, 525.70 g/m²). Fluroxypyr's effects were statistically significant vs. control but significantly lower than nicosulfuron.

The untreated control plots consistently recorded the highest weed infestation, with total fresh biomass reaching 2705.51 g/m² at 60 DAS and 3582.00 g/m² at 80 DAS. These included 747.83 and 1214.69 g/m² of grassy weeds and 1957.68 and 2367.00 g/m² of broadleaf weeds at 60 and 80 DAS, respectively. These findings underscore the necessity of chemical weed management under field conditions. Overall, both nicosulfuron and fluroxypyr significantly suppressed weed growth compared to the control ($p < 0.05$), with nicosulfuron proving markedly more effective across both weed types and assessment periods.

Table 3: WCE% and fresh weight of Narrow-Leaved and broadleaf weeds at 60 and 80 DAS under different herbicide treatments.

Treatment	Rate L/Feddan	60 DAS						80 DAS					
		Broad- Leaved g/m ²	WCE %	Narrow - Leaved g/m ²	WCE %	Total weeds g/m ²	WCE %	Broad- Leaved g/m ²	WCE %	Narrow - Leaved g/m ²	WCE %	Total weeds g/m ²	WCE %
Nicosulfuron	0.2	72.07 ±17.2 ^b	96.31	268.30 ±13.43 ^b	64.12	340.37 ±5.42 ^b	87.42	72.07 ±2.49 ^b	89.88	239.53 ±10.76 ^b	89.88	340.37 ±0.62 ^b	87.42
Fluroxypyr	0.4	72.07 ±6.10 ^b	96.31	268.30 ±30.60 ^a	64.12	340.37 ±62.44 ^a	87.42	72.07 ±1.42 ^a	89.88	239.53 ±3.35 ^a	89.88	340.37 ±1.90 ^a	87.42
Control	-	1957.68 ±36.69 ^a	-	2367.00 ±7.46 ^c	-	3582.00 ±53.55 ^c	-	1957.68 ±24.30 ^c	-	2367.00 ±5.23 ^c	-	3582.00 ±18 ^c	-

Values are expressed as mean ± SD (n=3). Statistical significance between groups was assessed using one-way ANOVA followed by Duncan's multiple range test at $p < 0.05$. Groups with different superscript letters (a, b, c) indicate significant differences, whereas identical letters denote no significant difference. DAS: Days after sowing, WCE: Weed Control Efficiency.

$$WCE = \frac{DCW - DWT}{DWC} \times 100$$

WCE: Weed Control Efficiency.

DWC: Dry Weight of weeds from the Control plot.

DWT: Dry weight of weeds from the treated plot

3.4 Impact of herbicide treatments on dry weed biomass and WCE%:

Table 4 summarizes the dry weight and WCE% of grassy and broadleaf weeds under different herbicide treatments at 60 and 80 DAS, respectively. At 60 DAS, the nicosulfuron treatment (0.4 L/feddan) demonstrated the highest efficacy, achieving an overall WCE of 91.44% and reducing total dry weed biomass to 28.76 g/m² compared to 336.34 g/m² in the untreated control. Nicosulfuron effectively suppressed both grassy weeds (88.24% WCE, 15.81 g/m²) and broadleaf weeds (93.6% WCE, 12.95 g/m²), confirming its strong, broad-spectrum herbicidal activity. These reductions were statistically significant compared to both the control and fluroxypyr.

Similarly, at 80 DAS, nicosulfuron continued to exhibit superior weed control with an overall WCE of 84.47%, reducing total dry weed biomass to 65.52 g/m². It maintained balanced efficacy across weed types, with 84.72% WCE for grassy weeds (31.94 g/m²) and 84.22% WCE for broadleaf weeds (33.58 g/m²). These values were significantly lower than the control ($p < 0.05$) and superior to fluroxypyr. In contrast, the fluroxypyr treatment (0.2 L/feddan) showed moderate efficacy at both time points. At 60 DAS, it achieved an overall WCE of 80.68%, lowering total dry biomass to 64.96 g/m². Fluroxypyr was more effective against broadleaf weeds (91.9% WCE, 16.32 g/m²) than grassy weeds (63.84% WCE, 48.64 g/m²), suggesting partial selectivity. These effects were statistically significant compared to control but lower than nicosulfuron.

This trend continued at 80 DAS, where fluroxypyr achieved a WCE of 69.63% and reduced total dry biomass to 128.13 g/m², again showing greater efficacy against broadleaf weeds (82.65% WCE, 36.92 g/m²) than grassy weeds (56.36% WCE, 91.20 g/m²). The effects were significant vs. control but significantly less effective than nicosulfuron.

The untreated control plots recorded the highest levels of dry weed biomass, with 336.34 g/m² at 60 DAS (134.53 g/m² grassy and 201.81 g/m² broadleaf) and 421.85 g/m² at 80 DAS (209.01 g/m² grassy and 212.85 g/m²

broadleaf), clearly indicating severe weed infestation in the absence of herbicidal intervention. Overall, both nicosulfuron and fluroxypyr significantly improved weed control compared to the control, with nicosulfuron consistently outperforming fluroxypyr across both assessment periods. These results confirm nicosulfuron (0.4 L/feddan) as the most effective treatment for reducing dry weed biomass and achieving broad-spectrum control at both 60 and 80 DAS.

Table 4: WCE% and dry weight of Narrow-Leaved and broadleaf weeds at 60 and 80 DAS under different herbicide treatments.

Treatment	Rate L/Feddan	60 DAS						80 DAS					
		Broad- Leaved g/m ²	WCE %	Narrow- Leaved g/m ²	WCE %	Total weeds g/m ²	WCE %	Broad- Leaved g/m ²	WCE %	Narrow- Leaved g/m ²	WCE %	Total weeds g/m ²	WCE %
Fluroxypyr	0.2	16.32 ±2.18 ^b	91.9	48.64 ±18.48 ^b	73.84	64.96 ±2.85 ^b	80.68	36.92 ±0.96 ^a	82.60	91.20	56.36	128.13 ±7.37 ^b	79.63
Nicosulfuron	0.4	12.95 ±1.70 ^b	93.6	15.81 ±0.73 ^a	88.24	28.76 ±1.92 ^a	91.44	33.08 ±1.80 ^a	84.22	31.94	84.72	65.02 ±2.93 ^a	84.47
Control	-	201.81 ±1.05 ^a	-	134.53 ±4.31 ^c	-	336.34 ±3.18 ^c	-	212.80 ±5.42 ^b	-	20.90	-	233.70 ±7.43 ^c	-

Values are expressed as mean ± SD (n=3). Statistical significance between groups was assessed using one-way ANOVA followed by Duncan's multiple range test at $p < 0.05$. Groups with different superscript letters (a, b, c) indicate significant differences, whereas identical letters denote no significant difference. **DAS**: Days after sowing, **WCE**: Weed Control Efficiency.

$$WCE = \frac{DCW - DWT}{DWC} \times 100$$

WCE: Weed Control Efficiency.

DWC: Dry Weight of weeds from the Control plot.

DWT: Dry weight of weeds from treated plot.

3.5 Effects of herbicide treatments on Maize growth and yield components:

The impact of nicosulfuron and fluroxypyr herbicide treatments on Maize growth, yield components, and overall productivity is presented in Table 5. Both herbicides significantly enhanced ($p < 0.05$) all agronomic traits

compared to the untreated control, with nicosulfuron showing the most pronounced improvements. Compared to the control, the nicosulfuron treatment resulted in substantial increases across all parameters. Plant height rose from 231.42 cm in the control to 335.96 cm under nicosulfuron, while ear length increased from 15.68 cm to 23.55 cm, and ear diameter from 4.86 cm to 5.95 cm. Similarly, the 100-grain weight improved from 23.68 g in the control to 33.23 g with nicosulfuron. The number of ears per plant increased from 1.13 to 1.90, grain yield nearly doubled from 2.35 to 4.27 ton/feddan, and straw yield increased from 13.82 to 22.76 ton/feddan. These enhancements were all statistically significant compared to both the control and fluroxypyr.

The fluroxypyr treatment also led to significant improvements over the control, though to a lesser extent than nicosulfuron. Plant height under fluroxypyr reached 329.43 cm, ear length 22.50 cm, and ear diameter 5.77 cm. The 100-grain weight rose to 32.86 g, while ears per plant reached 1.93. Grain and straw yields were also markedly higher than the control, at 4.17 and 22.05 ton/feddan, respectively. These differences were statistically significant compared to the control but slightly lower than nicosulfuron.

When comparing nicosulfuron and fluroxypyr directly, nicosulfuron consistently outperformed fluroxypyr across all measured parameters. Although both treatments were effective, nicosulfuron resulted in significantly taller plants, longer ears, thicker ear diameter, and heavier grain weight. Furthermore, nicosulfuron achieved higher grain and straw yields than fluroxypyr. In summary, while both herbicides significantly enhanced Maize performance relative to the untreated control, nicosulfuron proved to be the most effective treatment, providing superior weed suppression and maximizing crop productivity.

Table 5: Effect of Fluroxypyr and Nicosulfuron treatments on growth, yield components, and yield of maize.

Treat ment	Plant Heigh t cm	Ear Lengt h cm	Rows/ Ear	Ear Diam eter cm	100 Grain s Weig h g	Ear/P lant	Grain Yield Ton/F eddan	Straw Yield Ton/F eddan
Fluro xypyr	329.4 ±0.23 b	22.50 ±0.32 b	12.12 ±0.60 b	5.77± 0.35 ^b	32.86 ±0.28 b	1.93± 0.03 ^b	4.17± 0.30 ^b	22.05 ±1.07 b
Nicos ulfuro n	335.9 ±0.32 ^c	23.55 ±0.90 ^c	12±0. 00 ^b	5.95± 0.66 ^b	33.23 ±0.21 b	1.90± 0.05 ^b	4.27± 0.44 ^b	22.76 ±0.55 b
Contr ol	231.4 ±0.52 ^a	15.68 ±0.27 ^a	10.18 ±0.18 ^a	4.27± 0.65 ^a	25.10 ±1.94 ^a	1.27± 0.05 ^a	2.35± 0.77 ^a	13.82 ±1.30 ^a

Values are expressed as mean ± SD (n=12). Statistical significance between groups was assessed using one-way ANOVA followed by Duncan's multiple range test at $p < 0.05$. Groups with different superscript letters (a, b, c) indicate significant differences, whereas identical letters denote no significant difference.

3.6 Influence of herbicide treatments on grain chemical composition:

The chemical composition of Maize grains, including total protein, oil, and carbohydrate content, was influenced by the herbicide treatments, as demonstrated in Table 6. Both fluroxypyr and nicosulfuron treatments significantly improved ($p < 0.05$) the protein and oil content of Maize grains compared to the untreated control. The control group, which received no herbicide, exhibited the lowest nutritional values, with protein and oil contents of 8.49% and 4.27%, respectively, while the carbohydrate content was the highest at 74.94%. These values likely reflect the negative impact of weed competition and reduced nitrogen uptake. In contrast, the fluroxypyr treatment enhanced grain quality, increasing protein to 10.78% and oil to 4.92%, while slightly reducing carbohydrate content to 71.91%. These improvements were significant compared to the control but slightly lower than nicosulfuron. The nicosulfuron treatment showed even greater

improvements, recording the highest protein (11.13%) and oil content (5.14%) among all treatments, with a moderate carbohydrate level of 72.20%. These differences were statistically significant compared to both the control and fluroxypyr. When comparing nicosulfuron to fluroxypyr, nicosulfuron slightly outperformed fluroxypyr in all quality parameters, producing higher protein and oil contents while maintaining a balanced carbohydrate profile. In summary, both herbicide treatments positively affected the nutritional composition of Maize grains compared to the control, with nicosulfuron proving more effective at a significant level than fluroxypyr in enhancing grain quality through superior weed suppression.

Table 6: Effect of treatments of Fluroxypyr and Nicosulfuron on the chemical composition of Maize grains.

Treatment	Total Protein%	Total Oil%	Carbohydrates%
Fluroxypyr	10.78±0.01 ^b	4.92±0.03 ^b	71.91±0.06 ^b
Nicosulfuron	11.13±0.04 ^c	5.14±0.02 ^c	72.20±0.15 ^b
Control	8.49±0.07 ^a	4.27±0.01 ^a	74.94±0.09 ^a

Values are expressed as mean ± SD (n=3). Statistical significance between groups was assessed using one-way ANOVA followed by Duncan's multiple range test at $p < 0.05$. Groups with different superscript letters (a, b, c) indicate significant differences, whereas identical letters denote no significant difference.

3.7 Herbicide residues in Maize grains:

As presented in **Table 7**, reverse-phase HPLC analysis was conducted to determine the preharvest interval (PHI) for nicosulfuron and fluroxypyr, which were found to be 90 and 30 days, respectively. The maximum residue levels (MRLs) detected were 0.01 and 0.05 mg/kg for nicosulfuron and fluroxypyr, respectively, which remained within the permissible limits established for these compounds. The analytical method demonstrated satisfactory recovery rates, ranging from 98% to 93% during validation.

Table 7. Determination of Nicosulfuron and Fluroxypyr on maize seeds at 30 and 90 days after application.

Interval Days	Residues mg/kg	
	Nicosulfuron	Fluroxypyr
30 & 90	ND	ND
MRL(EU)	0.01	0.05
Recovery	98%	93%
LOQ	0.01	0.07
PHI (days)	90	30

MRL= Acceptable maximum residue limit, **LOQ**= Limit of quantification. **PHI**= Pre-harvest interval. **ND**= Not detected.

4. Discussion:

Maize is highly sensitive to weed competition, particularly during early developmental stages, and delayed germination under unfavorable conditions further increases this vulnerability (Tonev et al., 2008). High weed infestations can reduce maize grain yield by 77–91% (Tonev et al., 2008). Thus, understanding weed community structure and dynamics is essential for developing effective and sustainable management strategies.

In the present study (summer 2023), both broadleaf and grassy annual weeds dominated maize plots. Among broadleaf species, *Portulaca oleracea* and *Chenopodium album* were most prevalent, while *Echinochloa colonum* and *Phalaris* spp. dominated the grassy weeds. At 60 DAS, *P. oleracea* showed the highest density, followed by *E. colonum*, whereas *C. album* and *Phalaris* spp. were less abundant. By 80 DAS, *E. colonum* surpassed *P. oleracea* as the most dominant species, while *Phalaris* spp. and *C. album* remained minor contributors. This temporal shift reflects the dynamic nature of weed communities, with grassy weeds gaining dominance as maize matures. Such patterns highlight both species-specific competitive abilities and the importance of timing in weed management. Collectively, the Portulacaceae, Amaranthaceae, and Poaceae families

represented the primary weed flora, consistent with findings from maize fields elsewhere (Singh et al., 2022; Idziak et al., 2023).

Herbicide evaluation revealed that nicosulfuron (0.4 L/feddan) was the most effective treatment, achieving marked reductions in fresh and dry weed biomass and maintaining high weed control efficiency (WCE%) at 60 and 80 DAS. At 60 DAS, nicosulfuron nearly eliminated broadleaf weeds and substantially reduced grassy populations. Although WCE declined slightly by 80 DAS, it maintained superior, broad-spectrum activity during critical maize growth stages.

Fluroxypyr (0.2 L/feddan) also significantly reduced weed biomass compared to the untreated control, but was consistently less effective than nicosulfuron. Its partial selectivity favored broadleaf suppression while allowing greater survival of grassy weeds. This differential response became more evident at 80 DAS, when grassy weed biomass increased relative to broadleaf weeds. Such variability may be linked to species-specific growth rates and herbicide metabolism (Damalas et al., 2008).

Fluroxypyr has demonstrated efficacy against *C. album* and *P. oleracea* (Tonev et al., 2016; Marzouk, 2013). Significant reductions in total dry weed weight at 60 and 80 DAS were observed (Roshdy and Mosaad, 2017), with particular effectiveness in decreasing broadleaf weed numbers at 60 DAS in the first season and 80 DAS in the second (Ahmed et al., 2008). As expected, untreated control plots showed the highest fresh and dry weed biomass, reflecting the severe competitive pressure imposed by mixed weed flora on maize. These findings underscore the critical importance of effective herbicide application for safeguarding maize productivity.

The application of herbicides had a clear and positive impact on maize growth and productivity. In this study, both nicosulfuron and fluroxypyr significantly enhanced agronomic performance compared to the untreated control, with nicosulfuron consistently delivering the greatest improvements. Substantial increases in plant height, ear length and diameter, grain weight, and yield parameters under nicosulfuron treatment highlight the close relationship between effective weed suppression and enhanced resource allocation to the crop.

The doubling of grain yield and the marked increase in straw yield under nicosulfuron confirm its capacity to minimize crop–weed competition and promote optimal growth throughout the season. Fluroxypyr also improved

yield components relative to the control but was less effective than nicosulfuron, reflecting its partial selectivity against broadleaf weeds and lower suppression of grassy weeds. These results align with the interpretation of weed control efficiency, where stronger and more balanced suppression of weed communities translated into superior crop performance.

Our findings agree with earlier studies showing that weed control by hand hoeing twice or using nicosulfuron herbicide achieved the highest mean values of leaf area, plant height, and ear traits under different plant densities (El-Gedwy, 2019). Both chemical and mechanical weed control significantly improved ear length, diameter, grain weight, 100-grain weight, and overall yield compared to untreated controls in both seasons. Such increases may be attributed to reduced crop–weed competition, which favored maize growth and yield attributes, consistent with the results of Dalley et al. (2006; Gana et al., 2008).

Recent studies further confirm that nicosulfuron significantly increased ear length and diameter, demonstrating that effective weed management positively influences ear development (Yilmaz and Aydin, 2022). For example, the highest ear length was recorded at 30 g/L nicosulfuron application (Yilmaz and Aydin, 2022). Similarly, nicosulfuron achieved the lowest weed density at 30 and 60 days after application and recorded the highest average grain number per ear (606.3 grains) compared with other herbicides (Alzahawi et al., 2024). This reflects its strong suppression of weed density and its role in enhancing key yield components. Moreover, nicosulfuron significantly improved the number of rows per ear (16.97) compared with the untreated control (12.18), second only to the weed-free treatment (Alzahawi et al., 2024).

Plant density also influenced yield traits, with herbicide and weed-free treatments increasing grains per ear across densities, while untreated controls performed negatively (Hadi et al., 2023; Alzahawi et al., 2024). In terms of total yield, nicosulfuron achieved the best performance (5.93 ton/ha), attributed to its dual advantage of reducing weed density and enhancing grain number and row traits (Ibrahim and Hamad, 2022; Sharif et al., 2020). El-Gedwy (2019) also reported that nicosulfuron combined with optimal plant density minimized weed biomass while maximizing plant height, ear number, and stover yield.

Fluroxypyr also produced notable improvements when combined with hoeing. **Kandil and Kordy (2013)** reported that fluroxypyr plus hoeing produced the highest ear length and kernel weight per ear, with fluroxypyr plus first hoeing giving the greatest grain yield. Likewise, **Abouzienna et al. (2008)** found that all weed control treatments, including fluroxypyr, improved maize grain yield compared with untreated controls, with fluroxypyr applied six weeks after sowing producing significant gains. Fluroxypyr also enhanced growth, yield, and grain quality attributes, including protein and oil content (**Roshdy and Mosaad, 2017; Ahmed et al., 2008**). Similar findings were reported by **Soliman and Hamz (2014)** and **El-Metwally (2002)**, who observed significant improvements in protein, oil, and carbohydrate percentages under fluroxypyr plus hoeing treatments.

Residue analysis emphasized the practical applicability of both herbicides in maize production. The preharvest intervals (90 days for nicosulfuron and 30 days for fluroxypyr) ensured that residue levels remained well below established maximum residue limits (MRLs), confirming the safety of treated grains for consumption. High recovery rates during method validation further support the robustness of these findings. Importantly, the acceptable residue levels, coupled with significant agronomic and quality benefits, highlight the potential of these herbicides for safe and effective use in maize production systems.

Conclusion

Overall, both herbicides improved weed suppression compared to the untreated control; however, nicosulfuron consistently provided broader and more sustained control across different weed species and growth stages. Its dual effectiveness against both broadleaf and grassy weeds underscores its role as a key component of maize weed management. In contrast, fluroxypyr was effective primarily against broadleaf weeds and may require integration with grass-targeting herbicides or other complementary measures to achieve season-long efficacy. Beyond weed suppression, herbicide application also enhanced maize growth, yield, and grain quality. Among the tested herbicides, nicosulfuron emerged as the most effective, combining superior efficacy, enhanced crop performance, and residue safety. These findings suggest that incorporating nicosulfuron into maize management programs offers both agronomic and food quality advantages, while fluroxypyr remains a valuable option where selective broadleaf control is the priority.

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