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RESEARCH

Nicosulfuron Weed Control in Maize as Influenced by Adjuvants: Original vs. Generic Herbicide

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Abstract
In the absence of new herbicides on the market, adding adjuvants into the tank with herbicides is a strategy for increasing efficacy. In our research, we tested whether there are differences in weed control as influenced by the original nicosulfuron formulation and a generic counterpart. In this study, we tested the addition of two commonly used adjuvants: ammonium-sulfate (AMS) and non-ionic surfactant (NIS). In a three-year experiment, based on a percentage of biomass reduction and canopy cover, these results showed no differences in any treatments when comparing the original versus generic nicosulfuron. However, adding an NIS increased efficacy, while adding AMS decreased herbicide activity. The average percentage reduction of biomass in this study was about 80%, implying that using solely nicosulfuron as aceto-lactate synthase inhibiting herbicide is not a good solution in weed control in maize and that other methods for weed control should be considered and integrated, in order to increase weed control efficacy.

Keywords Weed biomass reduction · Canopy cover · Grain yield

Introduction

Despite many methods to reduce herbicide use in the management of weeds, herbicides are still the most common and relatively inexpensive method (comparing to other measures) for weed control (Gianessi 2013). Furthermore, herbicide patents generally expire within twenty years of being registered while dependently on registration process they might arrive at the market in upcoming 10–15 years. Furthermore, it opens the market for generic products that depress overall prices (Davis and Frisvold 2017). In general, generic herbicides are actives with an expired patent and are available from various manufacturers, competing with the original formulation (Duke 2012). Since the original herbicide (branded) and its generic counterpart have the same active ingredient, their efficacy should not differ. For example, various glyphosate products showed to have comparable weed control in Nebraska studies (Kappler et al. 2005).

It is anticipated that no new herbicide actives will appear in the coming years; therefore, better optimization of herbicide use now could prolong their overall lifespan (Sønderskov et al. 2014). One of the reasons for no new actives could be attributed to the synthesis of generic herbicides, as well as the presence of other available options to obtain satisfactory weed control. Some options to increase lifespan include adding an adjuvant into the tank and tank mixing with other active ingredients to reduce selection pressure (Polli et al. 2021). Adjuvants are agrochemicals commonly used to enhance pesticide physico-chemical properties by increasing penetration into the plant and/or lowering drift potential (Hazen 2000), ultimately increasing herbicide efficacy. Tataridas et al. (2022) reported that adjuvants could play a tremendous role in weed control, enabling usage of lower rates, even half rates of herbicides that would help in achievement of EU Green Deal goals. The adjuvant market in Serbia (and mostly over the EU) is small, with only a few types existing. New types are expected to appear in coming years in Serbian region, following the practices in the USA, where many herbicides are labeled to be applied with at least one adjuvant.

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Maize (*Zea mays* L.) is the most planted crop in Serbia (1 million hectares) (Statistical year book of the Republic of Serbia 2022), and weed control is the biggest challenge in Serbian maize systems. In the last ten years, it has been shown that weeds are likely to adapt to climate change (to modified conditions) (Krähmer et al. 2020), and in maize several weed species became dominant. Those species are: common lambsquarters (*Chenopodium album* L.), maple-leaved goosefoot (*C. hybridum* L.), pigweeds (*Amaranthus* spp.), and johnsongrass (*Sorghum halepense* [L.] Pears) (Brankov et al. 2023a). Post-emergence herbicides are popular among farmers, because they prefer to see emerged weeds, before applying herbicides (Brankov et al. 2024). The sulfonylurea herbicide nicosulfuron, is an acetolactate synthase inhibitor (ALS) that inhibits synthesis of essential branch-chained amino acids (valine, leucine, and isoleucine), stopping cell division, and causing necrosis of young plant tissue (Tranel and Wright 2002). Maize has a natural tolerance to nicosulfuron and is used for post-emergent weed control in grain production systems (Anonymous 2022). On the market today nicosulfuron can still be found in its original formulation, as well as many generic formulations. Nicosulfuron is an old chemistry on the market and many weeds have already developed herbicide resistance to ALS herbicides (Heap 2014) and it will not be of interests for farmers to lose that herbicide, while its efficacy still can be improved. Furthermore, there is a lack of data on testing the difference between the original nicosulfuron formulation and its generic equivalent, while new testing methods such as canopy cover, besides weed biomass reduction, can be used to evaluate effects of applied herbicides. Therefore, our study sought to evaluate nicosulfuron efficacy on weedy populations in maize: 1) comparing the original herbicidal formulation to a generic counterpart with the same active ingredient; 2) adding a NIS or AMS adjuvant into the tank; 3) measuring percent of weed biomass reduction, canopy cover, and impact on maize grain yield.

Material and Methods

A three-year field experiment was set at the Maize Research Institute "Zemun Polje" experimental station, Belgrade, Serbia (44°52′ N 20°20′ E) during 2020–2022. A maize hybrid ZP 707 was planted each year at a density of 60,000 plants ha^{-1}. The information about soil is presented in the supplementary material (Table S1). The whole experimental field is separated into two 10 hectare sections, where maize is planted on one section, and winter wheat (*Triticum vulgare* L.) on the other to enable rotation each successive year.

The original nicosulfuron product (Motivell Extra 6 OD, 60 g ai L^{-1}, Londerzeel, Belgium) and its generic counterpart (Talisman, Galenika Fitofarmacija, 40 g ai L^{-1}, Belgrade, Serbia) were applied at 45 g a.i. ha^{-1} (which represent a recommended field rate in Serbia) and tested in combination without and with two adjuvants: ammonium-sulfate (AMS) (AmoSulfan: ammonium-sulfate, 20% N + 24% S, WG. Elixir Group DOO; Serbia, rate: 5.00% v v^{-1}), and non-ionic surfactant (NIS) (Dash: 349 g/l oil (fatty acid esters) and 209 g/l alkoxylated alcohols-phosphate esters, EC. BASF SE, Germany, rate: 0.5% v v^{-1}). Therefore, six experimental treatments were tested in total: 3 adjuvants × 2 herbicides. Experiment also included an untreated control (treatment free) and another control that was weed free (manually kept free of weeds). The experiments were set up as a randomized complete block design with four replications. Individual plot size was 24.5 m^2 (4.9 × 5 m) and each plot contained seven maize rows spaced 0.7 m apart. Soil was prepared before maize sowing, starting the experiment at planting with a clean seedbed (pre-emergence herbicides were not applied).

Herbicide treatments were applied in May each year when maize had developed 5–6 leaves (15–16 BBCH) using a CO_2 backpack sprayer with a four-nozzle boom with a TTI (Air Induction Nozzle, TeeJet Technologies, Glendale Heights, IL, USA) nozzle (11102) calibrated to deliver

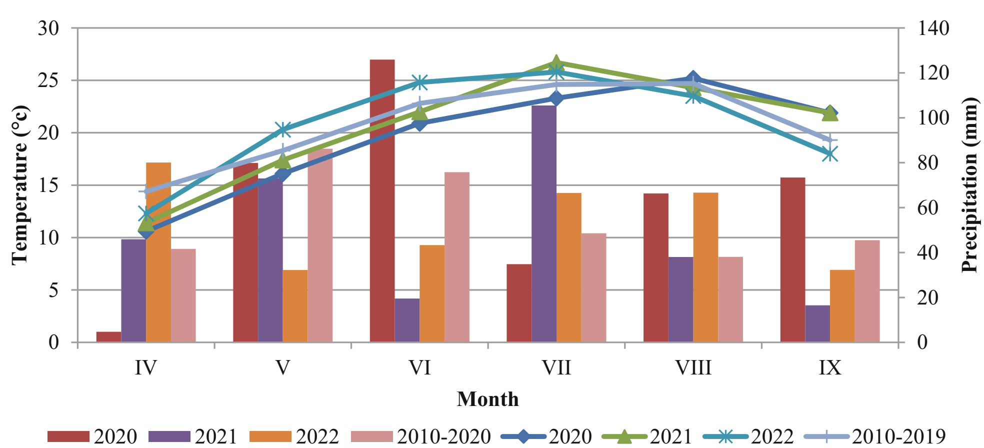

Fig. 1 Air temperatures (°C, monthly average) and precipitation (mm, total amount) during April-September of the three experimental years including the ten-year average (2010–2019) for the Zemun Polje location (Serbia). Bars indicate total precipitation; and lines are average monthly temperatures

a spray volume of 140 L ha^{-1} of solution at 275.8 kPa. During herbicide application the following wind velocity was recorded: 0 m s^{-1}, 2–3 m s^{-1}, and 2 m s^{-1}, respectively for 2020, 2021, and 2022.

Meteorological Conditions

The first experimental year (2020) was characterized by optimal precipitation for maize development, while the two consecutive years (2021 and 2022) had low precipitation after sowing (Fig. 1).

Whole plot canopy cover (%) was assessed at 21 days after treatment (DAT) using the CANOPEO mobile phone application (Division of Agricultural Sciences and Natural Resources, the OSU App Centre and Oklahoma State University). CANOPEO is an image-based application used to accurately determine the percentage of green canopy cover, by the classifying and counting pixels representing green canopy an image. Fractional green canopy cover ranges from 0: no green canopy cover to 100%: complete green canopy cover. One picture was taken per plot using a mobile phone at 1.5 m height from the plot using a tripod, at an angle of 45°. Pictures were taken during sunny days without clouds, from 12.00–14.00 h for all seasons as reported by McGlinch et al. (2021), Patrignani and Ochsner (2015).

Herbicide efficacy was evaluated 21 DAT using a 0.5 × 0.5 meter square measuring weed dry biomass. Weeds were identified, collected, and dried at 60 °C until they reached a constant mass, and the dry weight of each species per square meter was recorded. The biomass data were converted into percentage (%) of biomass reduction compared to the untreated control. The total number of weed species across treatments is presented in Supplementary material (Table 2). Five most abundance species, which total biomass was more than 90% of all weeds, are presented in the Table 2. After evaluation, all weeds were removed from the field by hoeing. At harvest, maize grain yield (t ha^{-1}) was recorded from the two center rows and calculated at 15.5% moisture content.

The data obtained were processed using the statistical package STATISTICA 8.0 for Windows (TIBCO software Inc., Palo Alto, CA 94304). Herbicide combinatios and year were included as fixed effects. The differences between the treatments were determined by two-way analysis of the variance (ANOVA), with mean separations made with $\alpha =$ 0.05 using Fisher's protected LSD test. Regression analyses were done using SPSS for Windows Version 15., in order to test dependence of weed biomass reduction and grain yield.

Table 1 Analysis of variance for effects herbicide, year, and their interaction on percentage of biomass reduction, canopy cover, and grain yield at 21 DAT

Factor	% Biomass reduction	Canopy cover	Grain yield
	p-value		
Herbicide	0.000	0.000	0.380
Year	0.000	0.431	0.000
Herbicide × Year	0.000	0.000	0.000

Results

Herbicide showed a significant effect on percentage of % biomass reduction and canopy cover, with no effect on grain yield. Effect of the year was significant for % of biomass reduction and grain yield, while interaction between herbicide and year for all measured parameters (Table 1).

Weed density varied across all years (Table S1). The most abundant species in the experimental years were: common lambsquarters (*Chenopodium album* L.), mapleleaved goosefoot (*Chenopodium hybridum* L.), jimsonweed (*Datura stramonium* L.), black nightshade (*Solanum nigrum* L.), and johnsongrass (*Sorghum halepense* [L.] Pers.).

The highest efficacy of applied herbicides was achieved adding the NIS adjuvant, especially in the 2020 and 2022 years. For *Chenopodium album* and *Ch. hybridum*, average increase of efficacy was more than 15% compared to the solo herbicide treatments. *Sorghum halepense* biomass reduction was lower only in the 2020 (up to 72%), while satisfactory biomass reduction was achieved in 2021 and 2022 years (91 and 98%, respectively). Adding the AMS adjuvant did not showed positive effects, reducing efficacy of applied herbicides (21% on average). There was no evidence of a difference in efficacy of compared the original and the generic nicosulfuron. That was the case for all years of the experiment (Table 2).

As year expressed a significant effect for the biomass reduction, treatments were further processed for each year. The percentage of biomass reduction differed across year, however, there was a similar pattern across treatments from year to year. There were no differences between solo herbicide treatments, where biomass reduction ranked between 58.2–82.9% for the original nicosulfuron, and 61.8–82.4% for the generic nicosulfuron formulation. When adding the AMS adjuvant with the herbicide, it showed a decrease in weed control of 38.9–65.7% and 42.6–68.0%, for the original and generic herbicides, respectively. The greatest impact on weed biomass reduction was observed when nonionic surfactant (NIS) was added to nicosulfuron, showing 78.8–93.8% and 77.1–94.8% for the original and generic herbicides, respectively (Table 3).

Table 2 Percentage of biomass reduction of five most abundant species as influenced by herbicides

	Treatments					
Weeds	H1	H1A1	H1A2	H2	H2A1	H2H2
2020						
Chenopodium album	69.1 c	77.6 b	87.5 a	79.8 b	80.5 b	86.5 a
Chenopodium hybridum	78.4 c	87.6 b	95.4 a	92.9 a	75.9 c	92.5 a
Datura stramonium	76.6 b	73.4 b	97.9 a	92.6 a	74.5 b	76.6 b
Solanum nigrum	35.4 c	45.6 b	55.3 a	27.1 c	17.5 d	54.9 c
Sorghum halepense	40.3 b	15.1 c	65.9 a	40.9 b	6.3 c	72.1 a
2021						
Chenopodium album	93.0 a	85.3 b	95.4 a	91.3 a	61.8 c	94.4 a
Chenopodium hybridum	98.9 a	98.9 a	100.0 a	100.0 a	94.7 a	100.0 a
Datura stramonium	100.0 a	95.9 a	100.0 a	100.0 a	100.0 a	100.0 a
Hibiscus trionum	97.2 a	90.0 b	84.6 c	93.0 b	79.0 d	95.1 a
Sorghum halepense	84.7 ab	77.9 b	91.9 a	80.4 b	62.1 c	86.6 a
2022						
Chenopodium album	70.9 b	64.0 c	97.2 a	70.5 b	57.8 c	94.8 a
Chenopodium hybridum	97.3 a	66.9 c	96.4 a	99.7 a	85.4 b	95.3 a
Datura stramonium	92.7 b	98.6 a	99.3 a	92.2 b	92.5 b	90.9 b
Solanum nigrum	100.0 a	100.0 a	100.0 a	100.0 a	100.0 a	100.0 a
Sorghum halepense	96.9 a	97.4 a	98.6 a	94.2 a	98.2 a	98.5 a

Mean values were compared within individual experimental years. Means followed by the same letter in the column within treatments, do no differ using Fisher's test at α = 0.05

H1 Motivel Extra 6 OD, *H1A1* Motivel Extra 6 OD + AMS, *H1A2* Motivel Extra 6 OD + NIS, *H2* Talisman, *H1A1* Talisman + AMS, *H1A2* Talisman + NIS

Table 3 Biomass reduction (%) influenced by herbicides. Data combined across species

Year	Treatments					
	H1	H1A1	H1A2	H2	H2A1	H2H2
2020	58.2 b	38.9 c	78.8 a	61.8 b	42.6 c	77.1 a
2021	66.5 b	50.4 c	74.5 a	61.4 b	46.8 c	72.0 a
2022	82.9 b	65.7 c	93.8 a	82.4 b	68.0 c	94.8 a
Average	69.2	51.7	82.3	68.5	53.7	81.3

Mean values were compared within individual experimental years. Means followed by the same letter indicate no difference using Fisher's test with α = 0.05

H1 Motivel Extra 6 OD, *H1A1* Motivel Extra 6 OD + AMS, *H1A2* Motivel Extra 6 OD + NIS, *H2* Talisman, *H1A1* Talisman + AMS, *H1A2* Talisman + NIS

It was observed that experimental year had an influence on maize grain yield, with the highest yields being recorded in 2020 and 2022, and a reduction in yield by greater than 80% in 2021 due to the high temperatures recorded during maize pollination (Figs. 1 and 2). No differences between solo nicosulfuron were recorded. In contrast, adding adjuvants greatly impacted maize yield (Fig. 2). Adding an NIS adjuvant to nicosulfuron resulted in the greatest positive impact on maize yield, while adding AMS resulted in yield reduction, due to a decrease in weed control. Yields as present of weed-free control are presented in the Fig. S1.

A regression analysis showed that weed biomass reduction resulted in a significant increase in maize grain yield, except for A0 (no adjuvant) and AMS treatments ($R2 = 0.064$ and $R2 = 0.057$, respectively) (Fig. 3). There were no differences between both herbicide treatments ($R2 = 0.190$ for Nic1 (original nicosulfruon) and $R2 = 0.191$ for Nic2 (generic nicosulfruon)). Nevertheless, a significant difference between adjuvants and their impact was observed. While the weed biomass reduction did not correlate significantly with a grain yield increase in the AMS treatment, there was a positive correlation between weed biomass reduction and maize grain yield in the NIS treatment ($R2 = 0.449$).

Discussion

Since no new herbicide actives have been introduced to the market for the last 30 years, optimizing their efficacy is a way to prolong their existence, combined with achieving satisfactory weed control. Since generic herbicides are

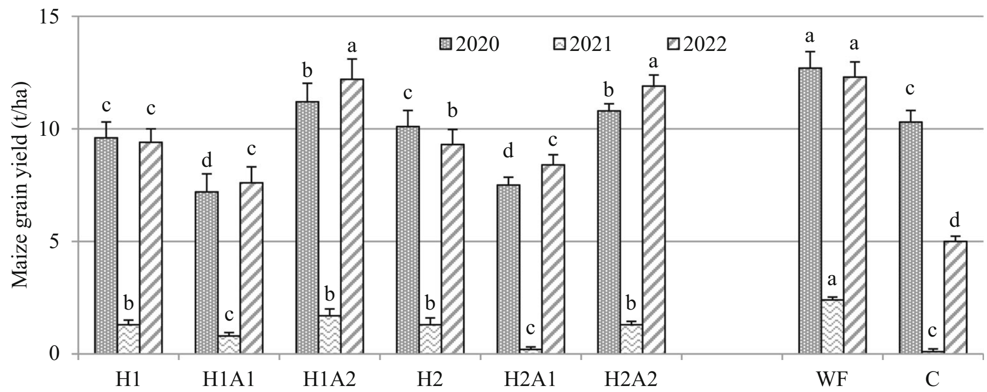

Fig. 2 Maize grain yield as influenced by herbicides in three experimental years. Means followed by the same letter across treatments within the same year do no differ using Fisher's test at α = 0.05. indicated by differing letter. *H1* Motivel Extra 6 OD, *H1A1* Motivel Extra 6 OD + AMS, *H1A2* Motivel Extra 6 OD + NIS, *H2* Talisman, *H1A1* Talisman + AMS, *H1A2* Talisman + NIS, *C* control, *WF* weed free

widely distributed on the market, it was of particular importance to compare the original formulations, with generic equivalents. Jabit et al. (2022) evaluated seven different products containing glufosinate as the active ingredient and concluded that the original formulation showed the most consistent results in weed control in oil palm plantations when compared to generic herbicides. Nicosulfuron is one of the most used herbicides in Serbia (Brankov et al. 2023a), and in this research, original and generic products have the same formulation (oil dispersion) without differences in weed control found (Table 1). The overall efficacy in our study was not satisfactory (<90% of weeds biomass reduction), even with the addition of adjuvants, especially for the most abundant species: *Sorghum halepense* and *Chenopodium album*. This could be explained by utilizing a TTI nozzle, which produces larger droplets and provides lower coverage (Brankov et al. 2023b). Weather conditions at the time of applications were favorable for sprayings, and in these conditions, applications could have been done using nozzles that produce fine droplets, enabling higher coverage. However, as meteorological conditions might not be ideal in springs due to high wind velocity in Serbia, we wanted to test nozzles that produce courser droplets, which decrease off-target drift.

Nicosulfuron is a systemic herbicide, and thus nozzle type is should not be as crucial for its efficacy against weeds as it is for contact herbicides (Ferguson et al. 2018). The greatest weed biomass reduction in this study was close to an 80% reduction, implying unsatisfactory efficacy. One of the reason might be found due to using a TTI nozzle which produces a coarser droplet. While these coarse droplet nozzles enable spraying under less favorable conditions, there is a chance of decreased efficacy. When applying systemic herbicides, Ferreira et al. (2020) observed no differences in weed control when using nozzles producing either fine or coarse droplets.

The adjuvant market in Serbia is small, and in the rare event that one is utilized, it is most often AMS or NIS. Since NIS adjuvants reduce surface tension on plant leaves, they help herbicide to penetrate to a higher degree in the plant (Sobiech et al. 2020). The results from this study are in line with the previously published results, where nicosulfuron efficacy was increased using either NIS or MSO adjuvant (Idziak et al. 2023). In our study, adding a NIS adjuvant increased nicosulfuron efficacy from 15–20%, while on the other hand, adding the AMS adjuvant decreased nicosulfuron efficacy, indicating it is not a suitable combination; as the % of weed biomass reduction was close to 40%. The reason could be found in the characteristics of AMS, since it is used as a water-conditioner to help overcome the antagonistic effect of positively charged ions bonding to the herbicide from minerals in hard water (Zollinger 2012), and is commonly added to glyphosate or glufosinate herbicides (Polli et al. 2021). Similarly to our research, (Idziak and Woznica 2013) found antagonistic effects for sulfonylurea herbicides when mixing with AMS. Nicosulfuron remains constant in neutral and alkaline solutions, and decomposes quickly in acidic environments (Bunting et al. 2004). The addition of AMS to the tank lowers the pH of the solution and could have been a contributing factor to the lower efficacy and associated decomposition of nicosulfuron while in the tank.

Furthermore, in this research, herbicide efficacy varied across years and the lowest efficacy was observed in the first year. This was attributed to the spring in the first year being favorable for the emergence of both weeds and maize. The problem was exacerbated by there being no pre-emergent herbicides applied. In fact, canopy cover was greatest in 2020, when compared to 2021 and 2022. It is shown that the efficacy of a herbicide is dependent on various factors, with weed size being one of the most important (Knoche 1994).

Under conditions of lower herbicide efficacy, the probability of survival is increased. It is reported that weeds which survive herbicide treatments might develop metabolic resistance (non-target site resistance) (Gressel 2011), making weed management challenging in years to come. In our research, *Chenopodium* sp., black night shade (*Solanum nigrum* L.), and johnsongrass, only showed partial control from the herbicide treatments (Table 2), especially when

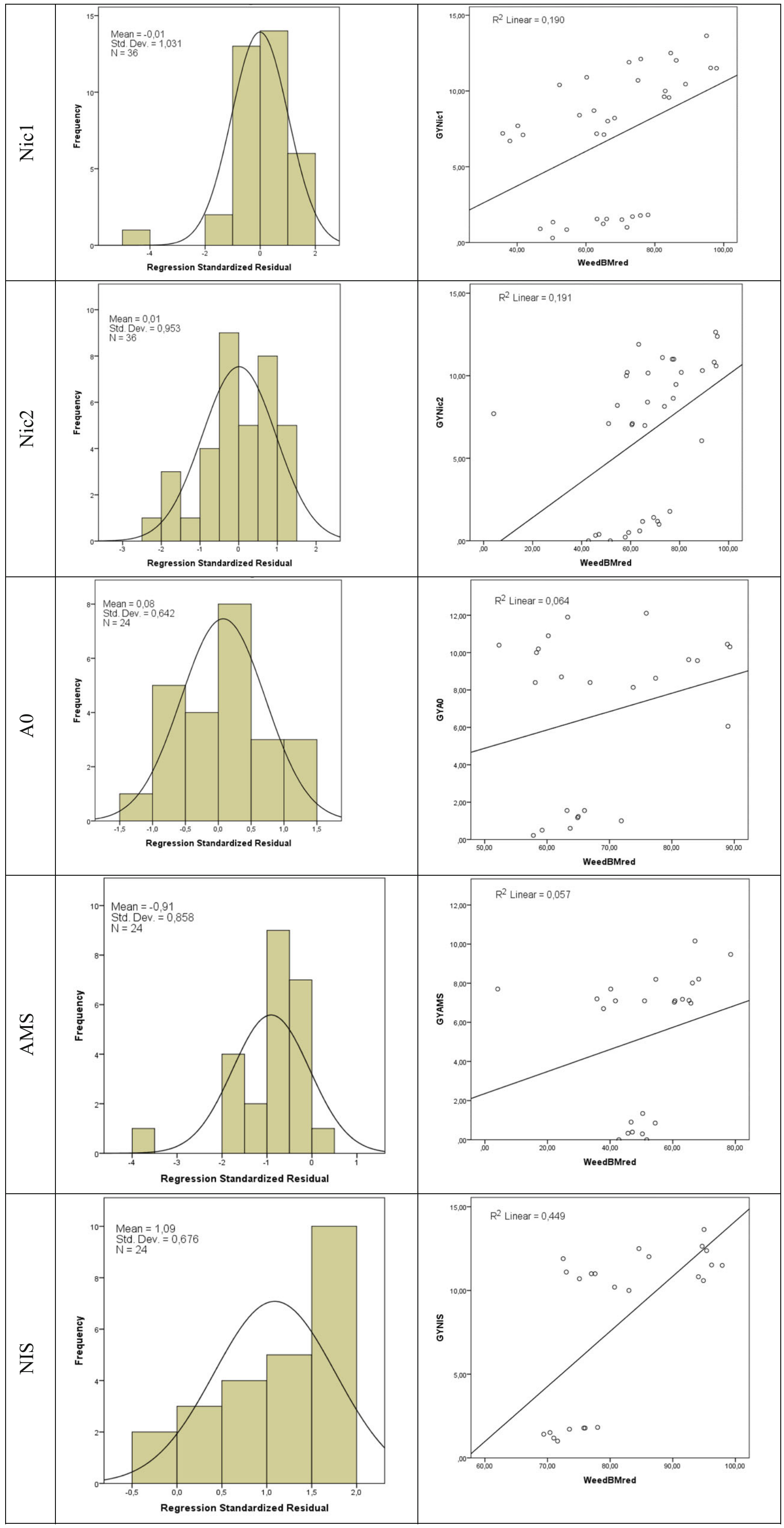

Fig. 3 Interdependence of weed biomass reduction and maize grain yield (GY) showing the influence of nicosulfuron herbicides (Nic1 and Nic2), as well as adjuvants (no adjuvant = A0, AMS and NIS). *Nic1* Motivel Extra 6 OD, *Nic2* Talisman

AMS adjuvant was added, signifying that those weeds could represent a reservoir for resistance development and spreading. Vieira et al. (2020) reported lower efficacy of glyphosate, dicamba, and 2,4 D on *Amaranth* species, following low doses exposure in the F2 progeny.

Increased herbicide efficacy aids in yield stability, and these data showed that there was no significant difference between original and generic herbicide products. The expected herbicide efficacy was not achieved, and confirmed that a single active ingredient is not recommended to provide satisfactory control. Adjuvants are a way to increase efficacy and support maize yield potential. In this case, NIS was the better candidate to support herbicide effectiveness and thus, yield stability. On the other hand, some adjuvants, like AMS, had adverse effects in combination with nicosulfuron, suggesting a necessity for more research on herbicide + adjuvants combination in specific agro-ecological conditions.

Conclusion

Our research in a three-year experiment indicated that there are no differences in efficacy for the original and generic nicosulfuron formulations on weed control. It is important to underline, however, that the addition of adjuvants can increase or reduce herbicide efficacy. In this study, adding a NIS adjuvant was highly valuable in increasing weed control efficacy, and supported increased maize yield. Although, overall efficacy was lower than acceptable, combination with other weed control tactics should be adopted into a weed control program in order to prevent weed survival from inadequate herbicide treatments, as this could promote resistance development. For future research considerations, the addition of a pre-emergence program combined with multiple herbicides to the tank for post applications could be considered.

Supplementary Information The online version of this article (https://doi.org/10.1007/s10343-024-01014-7) contains supplementary material, which is available to authorized users.

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Author Contribution Milan Brankov: Conceptualization, Writing, Editing; Milena Simić: Supervision, Statistical Analyses, Reviewing; Spencer L. Samuelson: Methodology, Writing, Supervision; Dušan Nikolić: Data citation, Editing; Zoran Čamdžija: Methodology, Writing; Violeta Mandić: Writing, Editing; Vesna Dragičević: Supervision, Reviewing

Availability of data and materials All data and materials are available on the reasonable request

Conflict of interest M. Brankov, M. Simić, S.L. Samuelson, D. Nikolić, Z. Čamdžija, V. Mandić and V. Dragičević declare that they have no competing interests.

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