



EFFICACY AND SELECTIVITY OF PRE-EM HERBICIDE ON DEPENDENCE OF SOIL TYPES AND PRECIPITATION IN SUNFLOWER CROP

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ABSTRACT. During the growing seasons in 2018 and 2019, two field trials were conducted to estimate how precipitation affects the efficacy of PRE-em herbicides in sunflower crop grown on different soil types. Both regions were naturally infested with a high population of *Polygonum aviculare* L., *Solanum nigrum* L., *Chenopodium album* L., *Amaranthus retroflexus* L., *Portulaca olearacea* L. and *Echinochloa crus-galli* (L.) P. Beauv. Efficacy of PRE-em herbicides varied among weed species, treatments, periods of efficacy estimation, regions and years, respectively. Overall performances of the PRE-em herbicides were correlated with the weather and soil properties. Humid April in Bitola region in 2018, particularly the first week after application (34 mm) before weed emergence caused herbicide leaching from the soil surface, which probably was the most likely reason for the lower efficacy of PRE-em herbicides in 2018, compared to their application in 2019. In 2018 precipitation above 30 years average were recorded in the Titov Veles region as well, but due to their equal occurrence particularly during the first and second week after application, as well as soil type properties (higher content of clay and organic matter) leaching did not occur and efficacy was good to excellent. Contrary, the limited precipitation after PRE-em application (five, nine, and eight mm during the first week before application, first and second week after application) may have contributed to the poor performance of PRE-em herbicides in the Titov Veles region in 2019 compared with 2018. Heavy precipitation directly following PRE-em application caused sunflower injury in the Bitola region in 2018, which ranged from 9–28% across PRE-em treatments seven days after application. Injuries of oxyfluorfen and dimethenamid were more serious (24 and 28%, respectively). Sunflower yields for each treatment in both regions generally reflected overall weed control and crop injury.

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Introduction

Sunflower (*Helianthus annuus* L.) is one of the four most important annual crops in the world grown primarily for edible oil and is cultivated on all continents (De la Vega, Hall, 2002). It is gaining importance for oil production due to its photo insensitivity, short duration, low water requirement, drought tolerance and wide range of adaptability to various agro-climatic conditions (Reddy, 2005). Sunflower is an important oil crop in North Macedonia and is mainly grown following winter wheat or barley in non-irrigated cropping systems (Egumenovski *et al.*, 2003). Despite

the adoption of good management practices, the productivity of sunflower in North Macedonia has been low, with average productivity of 1 440 kg ha⁻¹ (Anonymous, 2019a), which is very much lower than the EU average of 2 210 kg ha⁻¹ (Anonymous, 2019b), indicating wider scope for improving the yield potential. Weed competition has long been known to decrease sunflower yield (Johnson, 1971). Sunflower is usually planted in rows spaced 76 cm apart at lower densities than some other crops. Consequently, weeds that emerge during this time thrive in the wide interrow spaces. The season-long weedy conditions caused a 25.7% reduction in seed yield of sunflower (Wanjari *et*



al., 2000). The uncontrolled weed growth during the entire crop growth season caused an 83% reduction in seed yield of sunflower (Khan *et al.*, 1988; Legha *et al.*, 1992). Lewis, Gulden (2014) showed that sunflower yield was reduced by up to 76% when *Kochia scoparia* emerged at about the same time as the sunflower crop. Sunflower yield loss ranged from 35–54% under competition with *Avena fatua*, (Chubb, Friesen, 1985), *Sesbania herbacea* (Woon, 1987), *Orobancha cumana* (Grenz *et al.*, 2008), and mixed weed species (Reddy *et al.*, 2008). When *Artemisia biennis* emerged at about the same time as the sunflowers, the yield was reduced by up to 46% (Lewis *et al.*, 2016), whereas Johnson (1971) found that a combination of *Digitaria sanguinalis*, *Eleusine indica*, *Cassia obtusifolia*, *Ipomea purpurea*, *Ipomea hederacea*, and *Amaranthus retroflexus* decreased sunflower yield by 62% when the weeds competed with sunflower for the entire growing season. Therefore, weed control during the first 50–60 days after sunflower sowing is essential for high yield (Wanjari *et al.*, 2000). The outcome of crop-weed competition should be practised as early as possible to allow time for weed control measures (Knežević, 2000). Concerning weed control, due to its sowing period (mid-March to mid-April), this crop is very often characterized by a complex specific weed flora, composed of grass and broad-leaved weeds (Fried *et al.*, 2006). This weed flora has been traditionally controlled with PRE-em herbicide applications, due to the scarce availability of POST-em herbicides (di Rapparini, 1996). The use and norms of PRE-em herbicides on the sunflower vary depending on the type of herbicides and their combination (Jursik *et al.*, 2015; Simić *et al.*, 2011).

PRE-em herbicides are intended to be applied to the soil, and many require activation by rainfall and irrigation (Rainbow, Derpsch, 2011; Haskins, 2012). The activity of PRE-em herbicides applied to soil surface depends not only on the physicochemical properties of the herbicides, but the soil organic matter and clay content, and the period before the first rainfall event after application and the duration of following rainfall events (Lamoreaux *et al.*, 1993; Rodrigues, 1993; Watts, Hall, 1996). For most PRE-em herbicides precipitation is required within 7–14 days after application to dissolve the herbicide in soil water solution so that it can be taken up by the emerging weeds after germination (Buhler, Werling, 1989; Buhler, 1991; Novosel *et al.*, 1998; Chomas, Kells, 2004). It is widely known that PRE-em herbicides, such as *S*-metolachlor and dimethenamid-P require precipitation within 7–10 days after the application for proper movement into the active zone of weed seed germination (Steckel *et al.*, 2002; Anonymous, 2008). Inadequate or delayed precipitation can reduce herbicide effectiveness and decrease weed control (Armel *et al.*, 2003; Lyon, Wilson, 2005; Loux *et al.*, 2008). In addition, it is reported that different meteorological conditions influenced the activity of the soil-applied herbicides in sunflower (Simić *et al.*,

2011). Depending on soil type, high amounts of precipitation (*i.e.* greater than 25 mm), especially immediately after application, can cause herbicides to leach through the soil profile and consequently reduce efficacy (Reddy, Locke, 1996; Ferrell *et al.*, 2004; Boerboom *et al.*, 2006). Pendimethalin is an example of an herbicide that is more persistent in the soil under dry conditions and can affect rotational crops but is easily leached when soil conditions are wet (Savage, 1978; Lee *et al.*, 2000). Furthermore, pendimethalin's weed spectrum is reduced, especially the control of annual grasses, when soil conditions are dry up to 3 weeks after application (Bond, Griffin, 2005). It is well known that PRE-em herbicide sorption is highly dependent on soil organic matter, organic manure and soil pH value (Rouchaud *et al.*, 1998; Mitra *et al.*, 1999).

Taking into consideration that PRE-em herbicides can decrease and delay susceptible annual weed emergence and establishment, reduce subsequent growth, and minimize weed/crop interactions (Adcock, Banks, 1991; Black, Dyson, 1993), the main objectives were (i) to estimate the efficacy of PRE-em herbicides in sunflower depends on precipitations and soil types, and (ii) to evaluate their injury effect and influence on the sunflower yield. This research will help many farmers to use PRE-em herbicides at the right time depending on climatic conditions with special emphasis on rainfall and soil type.

Material and methods

The field trials were carried out during two sunflower growing seasons in 2018 and 2019 on commercial sunflower fields in the Bitola 41° 34' 52" N, 21° 39' 54" E and Titov Veles 41° 12' 23" N, 21° 21' 32" E sunflower growing regions in south-western and central Macedonia on Molic-vertic gleysol and Vertisol, respectively (Filipovski, 2006) (Table 1).

Table 1. Soil characteristics in the sunflower-growing regions

Region	Soil	Coarse sand %	Fine sand %	Clay + Silt %	Organic matter %	pH-water
Bitola	Molic-vertic gleysol	31.10	50.30	18.60	1.56	6.10
Titov Veles	Vertisol	3.50	34.20	60.30	2.40	7.20

The sunflower (*Helianthus annuus* L.) was grown following conventional tillage practices. The soil was tilled with a field cultivator before sowing. Nitrogen, phosphorus and potassium were applied as per soil test-based recommendation. The field trials were carried out with "Surimi CL" and "Driver CL" sunflower hybrids sowed in a well-prepared soil at a seeding rate of 60 000 seeds ha⁻¹ and 58 000 seeds ha⁻¹ on 17th April 2018 and 11th April 2019 in the Bitola region, and on 8th April 2018 and 3rd April 2019 in the Titov Veles region respectively. The trials were conducted in two different regions of the same commercial sunflower fields. The area of the main plots was 21 m² (5 m long and 4.2 m wide, *i.e.*, seven sunflower rows). At

harvesting time, the sunflower grain yield (adjusted to 9% of moisture content) was determined by hand-harvesting the central part of each plot 3.5 m² (1.4 m × 2.5 m). The weedy control plots were left untreated during the entire experimental period. Weed-free control was maintained by hand weeding. Hand-weeding was initiated at weeds emergence and continued as needed to maintain weed-free plots.

The experimental design was a randomized complete block with four replicates. Treatments included Stomp Aqua (pendimethalin 455 g l⁻¹, BASF Agro B.V Arnhem (NL), Zweigniederlassung Zürich, Switzerland) at 3.0 l ha⁻¹; Proman (metobromuron 500 g l⁻¹, Belchim Crop Protection N.V./S.A. Londerzeel, Belgium) at 3.0 l ha⁻¹; Goal (oxyfluorfen 240 g l⁻¹, Dow Agro-Science LLC, Indianapolis, IN) at 1.25 l ha⁻¹; Challenge 600 EC (aclonifen 600 g l⁻¹, Bayer Crop Science AG51368 Leverkusen, Germany) at 4.0 l ha⁻¹; Dual Gold 960 (S-metolachlor 960 g l⁻¹, Syngenta International, Basel, Switzerland) at 1.5 l ha⁻¹ and Frontier 900 EC (dimethenamid-P 900 g l⁻¹ BASF, Ludwigshafen am Rhein, Germany) at 1.7 l ha⁻¹. Untreated and weed-free controls were included in the studies, as well. All tested herbicides are registered for weed control in sunflower in the Republic of North Macedonia.

Herbicides were applied with a CO₂-pressurized backpack sprayer calibrated to deliver 300 l ha⁻¹ aqueous solution at 220 kPa. Herbicides were applied at the dry seed – beginning of seed imbibitions sunflower growing stage (BBCH 00–01). Weeds at the time of treatment were in the same growth stages as sunflower (BBCH 00–01). Weed control efficacy was estimated 28 days after applications (DAA) after weed emergence (four true sunflower leaves, BBCH 14; the first assessment) and 56 DAA shortly before canopy closure (BBCH 30–32; the second assessment) by the weed plants counting from 1 m² area within each plot, and herbicide efficacy was calculated by equation (Chinnusamy *et al.*, 2013):

$$W_{CE} = \frac{W_{up} - W_{tp}}{W_{up}} \times 100 \quad (1)$$

where:

W_{CE} – weed control efficiency

W_{up} – number of weeds in the untreated plots

W_{tp} – number of weeds in the treated plots

Sunflower injury was visually evaluated based on a 0–100% rating scale, where 0 is no injury to sunflower plants and 100 is complete death of sunflower plants (Frans *et al.*, 1986). Visual estimates of per cent sunflower injury were estimated 7 and 21 days after emergence (DAE), based on chlorosis and necrosis for each plot at both localities during the two-year experimental period. The yield was determined after harvest based on the weights of the grain containing 9% moisture.

The data were tested for homogeneity of variance and normality of distribution (Ramsey, Schafer, 1997) and were log-transformed as needed to obtain roughly equal

variances and better symmetry before ANOVA was performed. Data were transformed back to their original scale for presentation. Means were separated by using the LSD test at 5% of probability.

Results and discussion

The general assessment of weed control

The efficacy of PRE-em herbicides varied among weed species, treatments and periods of efficacy estimation, regions and years, respectively. Overall performances of the PRE-em herbicides were correlated with the weather and soil conditions. Inconsistent weather patterns between the two years of the study likely influenced the weed control. Humid April in 2018 (Table 2), particularly 1st WAA (34 mm) before weed emergence, caused herbicide leaching from soil surface which probably was the most likely reason for lower efficacy of PRE-em herbicides 2018 compared to their application in 2019 in the Bitola region (Table 4). Precipitations 1st WBA and 2nd WAA in 2018 were in line with the average for Bitola region, but 1st WAA was unusually wet, particularly the 2nd, 3rd, and 6th day of the week, as well as 1st day of the 2nd WAA.

Table 2. Mean weekly temperatures (T) and total weekly rainfall (P) 1 week before (WBA) and 4 weeks after PRE-em applications, respectively at Bitola and Titov Veles region in 2018 and 2019

Weeks	Bitola region				Titov Veles region			
	2018		2019		2018		2019	
	P, mm	T, °C	P, mm	T, °C	P, mm	T, °C	P, mm	T, °C
1 st WBA	16	12	9	16	14	14	5	17
1 st WAA	34	9	18	14	22	11	9	15
2 nd WAA	13	14	8	18	16	16	8	18
3 rd WAA	7	17	17	13	5	18	15	15
4 th WAA	4	15	9	17	1	18	13	18
Sum of P	74	–	61	–	77	–	50	–
Average of T	13.4		15.6		15.4		16.6	

Abbreviations: WBA – a week before application; WAA – a week after application; P – precipitations, T – temperature.

PRE-em treatments in both years were applied at times when herbicide applications typically occur in North Macedonia sunflower production and are thus representative of producer practices and label recommendations.

In the Titov Veles region for the same year precipitations occurred in the 1st WBA, 1st WAA and 2nd WAA were 45% above the 30 years average for this region (38 mm). In 2019, precipitation occurred in the 1st WBA, and 2nd WAA were scarce for the Bitola region, while rainfall in the 1st WAA and 3rd WAA was in the line with the average for this region (41 mm). In the Titov Veles region, in the same year, the period during the 1st WBA, 1st WAA and 2nd WAA was very dry (5, 9 and 8 mm). It rained on the third and fourth days at intervals throughout the 3rd WAA and 4th WAA, respectively (Table 2). Also, one week before and four weeks after PRE-em applications temperatures particularly in 2019 for both regions were a little bit above the average and that was attributed to favourable environmental conditions associated with non-frost night time during the estimated 1st WBA and 4 week

period after PRE-em applications, respectively. Usually, higher amounts of precipitation and heavy rains immediately after PRE-em application, particularly on sandy soils with low organic matter (Inoue *et al.*, 2010; Shaner, 2014) may cause leaching of herbicides through the soil profile below the weed seed-germinating zone and consequently decrease weed control efficacy (Heatherly, Hodges, 1998 Ferrell *et al.*, 2004). In 2018 precipitation above 30 years average were recorded in Titov Veles, as well, but due to their equal occurrence particularly during the 1st WAA and 2nd WAA, as well as soil type characteristics (higher content of clay and organic matter) leaching did not occur and efficacy was good to excellent. It is reported that higher soil organic matter content results in a higher herbicide efficacy (Xing, 2001). Opposite, the limited precipitation after PRE-em application (five, nine, and eight mm, during the 1st WBA, 1st and 2nd WAA) may have contributed to the poor performance of PRE-em herbicides in the Titov Veles region in 2019 compared with 2018 (Table 4). Since many of the PRE-em herbicides can volatilize and photodegrade on the soil surface over time, rainfall is needed to move these herbicides into the zone where weed seeds germinate (Wilcut *et al.*, 1994; Janak, Grichar, 2016) which explains the inconsistent control of predominant weeds noted with PRE-em herbicides under the drought conditions observed at Titov Veles region in early spring 2019. However, in both regions, regardless of year and herbicide treatments, the efficacy of PRE-em herbicides was insignificantly lowered by 56 DAA, due to new weed emergence occur between two estimation periods (Table 4 and 5).

Pendimethalin

PRE-em treatment with pendimethalin resulted in two distinct control years in both regions, but it did not significantly differ among periods of efficacy estimation by year. In 2018, in the Bitola region, 28 DAA weed control efficacy was ranged from 65% *Solanum nigrum* L. (SOLNI) to 77% *Chenopodium album* L. (CHEAL). Further decreasing in pendimethalin efficacy was recorded 56 DAA (between 54% SOLNI and *Echinochloa crus-galli* L. P. Beauv. (ECHCG), and 72% CHEAL. Pendimethalin efficacy was significantly improved in 2019. However, 28 DAA *Polygonum aviculare* L. (POLAV) was fully controlled (100%). Except for SOLNI (85%), the rest of the weeds were controlled between 96 and 97%. Negligible weed control decreasing occurred 56 DAA (Table 4). Unlike the Bitola region, in 2018, in the Titov Veles region efficacy of pendimethalin was substantially higher.

28 DAA weed control efficacy was ranged from 93% *Portulaca oleraceae* L. (POROL) to 96% *Amaranthus retroflexus* L. (AMARE). Only SOLNI was controlled <90%. Insignificantly lower efficacy (between 87% CHEAL and 91% AMARE and POROL pendimethalin provided 56 DAA. Significantly lower efficacy of this herbicide (78%) was recorded in control of SOLNI. In 2019 pendimethalin provided no more than 74% and 57% weed control 28 DAA and 56 DAA, respectively (Table 5). In the investigation of Pannacci *et al.*, (2007) pendimethalin applied at 921 g a.i. ha⁻¹ (grams of active ingredient per hectare) in sunflower controlled AMARE between 88% and 100%, ECHCG between 94 and 100%, and CHEAL 100%. Similar, in the sunflower crop pendimethalin effectively controlled CHEAL more than 95%, while the efficacy on ECHCG ranged between 85 and 98% (Jursik *et al.*, 2015). In the same study, the efficacy of pendimethalin on *Solanum physalifolium* was significantly lower on plots without irrigation ($\geq 67\%$ compared to irrigated plots ($\geq 85\%$)).

Metobromuron

A significant treatment by year interaction resulted in two distinct years for metobromuron weed control in both regions, but metobromuron weed control did not significantly differ among periods of efficacy estimation by year. In the Bitola region, in 2018 metobromuron provided poor control of investigated weeds. 28 DAA efficacy was ranged between 55% ECHCG and 68% SOLNI. Lower efficacy from 46% CHEAL to 63% SOLNI was recorded 56 DAA. Metobromuron efficacy was significantly improved in 2019. The 28 DAA, metobromuron controlled CHEAL, POLAV and AMARE >95%, while significantly lower efficacy was recorded in control of SOLNI and ECHCG (78 and 82%, respectively). Decreasing of metobromuron efficacy for a few per cent in control of all weeds was noticed 56 DAA (Table 4). Metobromuron provided effective weed control in the Titov Veles region in 2018. AMARE and CHEAL were controlled 96%, POROL 91%, while SOLNI was controlled only 71%, 28 DAA. Metobromuron efficacy was slightly reduced by 56 DAA. Opposite, in 2019, due to dry soil conditions, control of weeds was less than 66% and 63%, 28 and 56 DAA, respectively (Table 5). Similar to these results of Bergmann (2016), in field trials conducted from 2009 till 2012 concluded that Proman (metobromuron) applied at 3 l ha⁻¹ provided 90% control of POLAV, 93% of CHEAL, 89% of AMARE, but only 70% on SOLNI and 63% on ECHCG.

Table 3. Weed population (species and number of weeds) in sunflower at Bitola and Titov Veles region in 2018 and 2019

Weed species	Bitola region		Titov Veles region	
	2018	2019	2018	2019
<i>Polygonum aviculare</i> L.	33	14	–	–
<i>Chenopodium album</i> L.	24	21	54	27
<i>Solanum nigrum</i> L.	27	13	38	16
<i>Amaranthus retroflexus</i> L.	18	17	66	40
<i>Echinochloa crus-galli</i> (L.) P. Beauv.	16	14	–	–
<i>Galinsoga parviflora</i> Cav.	4	2	–	–
<i>Abutilon theophrasti</i> Medic.	2	3	2	1
<i>Diploaxis muralis</i> (L.) D.C.	2	–	3	3
<i>Portulaca olearacea</i> L.	1	4	48	29
<i>Digitaria sanguinalis</i> (L.) Scop.	–	–	–	9
Total weed species	9	8	6	7
Total weeds (No. m ⁻²)	127	88	212	125

Table 4. Efficacy of PRE-em herbicides (%), 28 and 56 DAA in sunflower in 2018 and 2019 in Bitola regions^{a-d}

Treatments	Bitola region																							
	Pendimethalin 3.0 l ha ⁻¹		Metobromuron 3.0 l ha ⁻¹		Oxyfluorfen 1.25 l ha ⁻¹		Aclonifen 4.0 l ha ⁻¹		S-metolachlor 1.5 l ha ⁻¹		Dimethenamid 1.7 l ha ⁻¹													
	2018	2019	2018	2019	2018	2019	2018	2019	2018	2019	2018	2019												
Weed species	28 DAA	56 DAA	28 DAA	56 DAA	28 DAA	56 DAA	28 DAA	56 DAA	28 DAA	56 DAA	28 DAA	56 DAA												
POLAV	74 ^a	69 ^a	100 ^a	98 ^a	63 ^{ab}	61 ^a	98 ^a	95 ^a	71 ^{ab}	68 ^a	98 ^{ab}	95 ^a	68 ^a	66 ^a	100 ^a	97 ^a	65 ^{ab}	61 ^{ab}	99 ^a	97 ^a	60 ^{bc}	57 ^b	100 ^a	94 ^a
SOLNI	65 ^b	54 ^b	85 ^c	81 ^c	68 ^a	63 ^a	78 ^c	66 ^c	75 ^a	75 ^a	98 ^{ab}	92 ^{abc}	64 ^a	53 ^c	76 ^c	63 ^c	71 ^a	65 ^a	95 ^b	89 ^c	75 ^a	68 ^a	96 ^{abc}	87 ^b
CHEAL	77 ^a	72 ^a	96 ^b	89 ^b	59 ^{bc}	46 ^b	95 ^{ba}	90 ^a	67 ^b	55 ^{bc}	100 ^a	90 ^{bc}	64 ^a	56 ^{bc}	95 ^b	91 ^b	61 ^b	50 ^c	97 ^{ab}	92 ^b	56 ^c	42 ^c	95 ^{bc}	88 ^b
AMARE	73 ^a	61 ^a	97 ^a	90 ^b	65 ^{ab}	58 ^a	100 ^a	92 ^a	70 ^{ab}	61 ^b	97 ^b	93 ^{ab}	68 ^a	62 ^{ab}	98 ^{ab}	91 ^b	63 ^b	56 ^{bc}	95 ^b	87 ^c	60 ^{bc}	58 ^b	94 ^c	89 ^{ab}
ECHCG	69 ^{ab}	54 ^b	96 ^b	89 ^b	55 ^c	49 ^b	82 ^c	75 ^b	69 ^b	52 ^c	97 ^b	88 ^c	70 ^a	57 ^{bc}	98 ^{ab}	90 ^b	67 ^{ab}	62 ^{ab}	100 ^a	93 ^b	64 ^b	54 ^b	100 ^a	92 ^{ab}
LSD 0.05	7.17	6.74	3.26	5.22	6.25	5.81	4.23	7.52	5.08	7.43	2.54	4.73	6.65	6.48	4.03	5.26	6.36	7.86	3.68	2.51	6.90	6.08	4.19	5.03
Random effect interactions																								
PRE-em herbicides	*		*		*		*		*		*													
treatment × year																								
PRE-em herbicides	NS		NS		NS		NS		NS		NS													
treatment × PEE																								

^aAbbreviation: PRE-em; DAA – days after application; POLAV – *Polygonum aviculare*; SOLNI – *Solanum nigrum*; CHEAL – *Chenopodium album*; AMARE – *Amaranthus retroflexus*; ECHCG – *Echinochloa crus-galli*; PEE – periods of efficacy estimation; NS – not significant; * Significant at the 5% level according to a Fisher's protected LSD test at P < 0.05

^bPRE treatments were applied in the same growth stages as sunflower (at the dry seed – beginning of seed imbibitions of sunflower growing stage – (BBCH 00-01).

^cWeed control efficacy was estimated at 28 DAA and 56 DAA

^dMeans followed by the same letter within a column are not significantly different according to Fisher's Protected LSD at P < 0.05

Table 5. Efficacy of PRE-em herbicides (%), 28 and 56 DAA in sunflower in 2018 and 2019 in Titov Veles region^{a-d}

Treatments	Titov Veles region																							
	Pendimethalin 3.0 l ha ⁻¹		Metobromuron 3.0 l ha ⁻¹		Oxyfluorfen 1.25 l ha ⁻¹		Aclonifen 4.0 l ha ⁻¹		S-metolachlor 1.5 l ha ⁻¹		Dimethenamid 1.7 l ha ⁻¹													
	2018	2019	2018	2019	2018	2019	2018	2019	2018	2019	2018	2019												
Weed species	28 DAA	56 DAA	28 DAA	56 DAA	28 DAA	56 DAA	28 DAA	56 DAA	28 DAA	56 DAA	28 DAA	56 DAA												
AMARE	96 ^a	91 ^a	68 ^b	57 ^a	96 ^a	89 ^a	60 ^{ab}	51 ^b	94 ^{ab}	90 ^a	66 ^a	54 ^b	96 ^a	87 ^a	66 ^a	60 ^a	93 ^a	87 ^a	63 ^b	53 ^b	93 ^{ab}	85 ^b	58 ^b	49 ^b
CHEAL	94 ^a	87 ^b	64 ^{bc}	55 ^{ab}	96 ^a	87 ^a	53 ^b	48 ^b	94 ^{ab}	85 ^{ab}	59 ^b	52 ^b	94 ^a	87 ^a	57 ^b	52 ^b	90 ^{ab}	81 ^{ab}	55 ^c	46 ^{bc}	91 ^{ab}	83 ^b	53 ^b	44 ^c
POROL	93 ^a	91 ^a	74 ^a	51 ^{bc}	91 ^b	82 ^b	66 ^a	53 ^b	90 ^b	80 ^b	58 ^b	49 ^b	93 ^a	83 ^a	68 ^a	55 ^{ab}	85 ^b	78 ^b	53 ^c	42 ^c	88 ^b	79 ^c	57 ^b	46 ^{bc}
SOLNI	84 ^b	78 ^c	61 ^c	49 ^c	78 ^c	71 ^c	66 ^a	63 ^a	96 ^a	90 ^a	70 ^a	68 ^a	62 ^b	58 ^b	55 ^b	52 ^b	93 ^a	88 ^a	71 ^a	68 ^a	94 ^a	90 ^a	73 ^a	70 ^a
LSD 0.05	3.30	1.87	5.60	5.59	3.42	4.36	7.35	7.08	4.30	7.01	4.56	6.40	6.01	5.00	8.20	7.51	6.77	8.03	6.94	6.75	8.2	3.92	6.83	4.53
Random effect interactions																								
PRE-em herbicides	*		*		*		*		*		*													
treatment × year																								
PRE-em herbicides	NS		NS		NS		NS		NS		NS													
treatment × PEE																								

^aAbbreviation: PRE-em; DAA – days after application; AMARE – *Amaranthus retroflexus*; CHEAL – *Chenopodium album*; POROL – *Portulaca olearacea*; SOLNI – *Solanum nigrum*; PEE – periods of efficacy estimation; NS – not significant; * Significant at the 5% level according to a Fisher's protected LSD test at P < 0.05

^bPRE treatments were applied in the same growth stages as sunflower (at the dry seed – beginning of seed imbibition of sunflower growing stage – (BBCH 00-01)

^cWeed control efficacy was estimated at 28 DAA and 56 DAA

^dMeans followed by the same letter within a column are not significantly different according to Fisher's Protected LSD at P < 0.05

Oxyfluorfen

PRE-em application of oxyfluorfen resulted in two distinct years for its efficacy in both regions. However, weed control did not significantly differ among periods of efficacy estimation by year. In the Bitola region, in 2018, oxyfluorfen controlled weeds between 67% CHEAL and 75% SOLNI 28 DAA, and 52% ECHCG and 75% SOLNI 56 DAA. Significantly increasing in weeds control oxyfluorfen provided in 2019. 28 DAA, the CHEAL was fully controlled (100%), and the rest of the weeds were controlled between 97 and 98%. Except for ECHCG which was controlled 88%, the rest of the weeds were controlled between 90 and 95%, 56 DAA (Table 4). In the Titov Veles region, in the first experimental year (2018), oxyfluorfen effectively controls all predominant weeds. Efficacy was ranged from 90% POROL to 96% SOLNI, and 80% POROL to 90% SOLNI, 28 and 56 DAA, respectively. In the second experimental year (2019), oxyfluorfen efficacy substantially decreased. Efficacy of oxyfluorfen gave only marginal control (<70% and <68%, 28 and 56 DAA, respectively) of predominant broadleaf weeds (Table 5). Oxyfluorfen applied at 240 a.i. ha⁻¹ in sunflower controlled AMARE, ECHCG and CHEAL 100% (Pannacci *et al.*, 2007). In the study of Jursík *et al.* (2015) efficacy of oxyfluorfen in sunflower, the crop was very good on AMARE (control greater than 95%) and was not affected by soil moisture conditions in any trial year, but oxyfluorfen was not as effective on CHEAL under non-irrigated conditions. In leek, oxyfluorfen at 360 g a.i. ha⁻¹ controlled SOLNI 96% (Karkanis *et al.*, 2012).

Aclonifen

A significant treatment by year interaction resulted in two distinct years for aclonifen weed control in both regions, but aclonifen weed control did not significantly differ among periods of efficacy estimation by year. In 2018, in the Bitola region, 28 DAA weed control efficacy was ranged from 64% SOLNI and CHEAL to 70% ECHCG. Decreasing of aclonifen efficacy was recorded 56 DAA. The herbicide provided control between 56% SOLNI, and 66% POLAV. Next 2019 efficacy of aclonifen was significantly improved. 28 DAA, unless SOLNI which was poorly controlled (76%), the rest of the weeds were nearly fully controlled (>98%). Decreasing aclonifen weed control for few percents occurred 56 DAA (Table 4). Unlike the Bitola region, in 2018, in the Titov Veles region efficacy of aclonifen was substantially higher. Weed control efficacy was ranged from 93% POROL to 96% AMARE, 28 DAA, and 83% POROL to 87% AMARE and CHEAL 56 DAA. Poor aclonifen efficacy (only 62 and 58%) was noted in control of SOLNI during both estimation periods. In 2019 aclonifen provided no more than 68% and 60% weed control 28 DAA and 56 DAA, respectively (Table 5). In banded herbicide application in a conventional sunflower production system aclonifen applied at 0.75 kg a.i. ha⁻¹ controlled CHEAL between 84 and 89% (Serim *et al.*, 2018). In the study of Jursík *et al.* (2015) aclonifen controlled AMARE

and CHEAL with efficacy greater than 97%, regardless of irrigation. In the same study, aclonifen controlled ECHCG (efficacy over 80%), but only when irrigation was applied or natural precipitation at the beginning of the growing season was sufficient. On the other side, regardless of irrigation *Solanum physalifolium* was controlled between 52 and 56%. Also, in the investigation of Pannacci *et al.* (2007) aclonifen applied at 900 g a.i. ha provided poor control of SOLNI (33%–67%).

S-metolachlor

A significant treatment by year interaction resulted in two distinct years for S-metolachlor weed control in both regions, but S-metolachlor weed control did not significantly differ among periods of efficacy estimation by year. In the Bitola region, in 2018 S-metolachlor provided inadequate control of investigated weeds. 28 DAA efficacy was ranged between 61% CHEAL and 71% SOLNI. Further decreasing in efficacy from 50% CHEAL to 62% ECHCG was recorded 56 DAA. S-metolachlor efficacy was significantly increased in 2019. 28 DAA, S-metolachlor fully controlled 100% ECHCG, while the rest of the weeds were controlled between 95 and 99%. A negligible few per cent decreasing of S-metolachlor efficacy in the control of predominant weeds was recorded 56 DAA (Table 4). S-metolachlor provides effective weed control in the Titov Veles region in 2018. During the first estimation period 28 DAA, AMARE and SOLNI were controlled 96%, CHEAL 90%, while POROL was controlled <90%. Insignificantly lower efficacy between 78% POROL and 88% SOLNI S-metolachlor provided 56 DAA. Contrary, in 2019, due to dry soil conditions, control of weeds was less than 71% and 68%, 28 and 56 DAA, respectively (Table 5). S-metolachlor in irrigated sunflower plots nearly completely controlled AMARE and ECHCG (efficacy 93–100%). However, on treatment without irrigation, the efficacy of S-metolachlor on AMARE decreased by 8%, and the efficacy on ECHCG decreased significantly by 13% (Jursík *et al.*, 2015). In banana pepper S-metolachlor at 534 g a.i. ha⁻¹ provided control of CHEAL of 99% (2 WAT) and 85 (4 WAT), while S-metolachlor at 1 070 g a.i. ha⁻¹ provided control of CHEAL of 96 (2 WAT) and 90 (4 WAT). At the same rates, POROL was controlled between 61 and 67% (Mohseni-Moghadam, Doohan, 2015). Opposite, in spinach S-metolachlor at rates ≥ 0.56 kg ha⁻¹ provided >95% control of POROL (Fennimore *et al.*, 2001).

Dimethenamid-P

PRE-em application of dimethenamid-P resulted in two distinct years for its efficacy in both regions. However, weed control did not significantly differ among periods of efficacy estimation by year. In the Bitola region, in 2018, dimethenamid controlled weeds between 56–42% CHEAL, and 75–68% SOLNI 28 and 56 DAA, respectively. Significantly increasing in weeds control dimethenamid provided in 2019. 28 DAA, POLAV and ECHCG were fully controlled (100%), and the rest of the weeds were controlled

between 94 and 96%. The SOLNI and CHEAL were controlled <90%, while POLAV and ECHCG controlled >90%, 56 DAA (Table 4). In the Titov Veles region, in the first experimental year (2018), dimethenamid-P effectively control all predominant weeds (>91% and >83%), except POROL (88% and 79%), 28 and 56 DAA, respectively. In the second experimental year (2019), dimethenamid-P efficacy substantially decreased. Efficacy of dimethenamid gave only marginal control (<73% and <70%, 28 and 56 DAA, respectively) of predominant broadleaf weeds (Table 5). In potato crop dimethenamid-P has provided greater than 96% control of SOLNI, CHEAL, and AMARE in Idaho research trials at rates of 1.1 to 1.7 kg ha⁻¹ (Tonks *et al.*, 1999). Similar, in Idaho field research trials Alvarez, Hutchinson (2005) and Hutchinson *et al.* (2005) confirmed that dimethenamid-P provided acceptable season-long SOLNI control (>88%). Dimethenamid-P applied alone gave excellent control (>98%) of AMARE and SOLNI in dry bean (Arnold *et al.*, 2012). An evaluation of PRE herbicides for weed control in pumpkin found that 21 days after treatment dimethenamid-P at 2.24 kg ha⁻¹ resulted in 81–100% control of AMARE (Brown, Masiunas, 2002). In the investigation of Yamaji *et al.* (2016) dimethenamid-P at 1138 g a.i. ha⁻¹, provided control of ECHCG greater than 90%. Similar, in sugarbeet AMARE and CHEAL control with dimethenamid-P, applied at 0.84 kg ha⁻¹ was 99% and 91% (Bollman, Sprague, 2007).

Sunflower injury

PRE-em herbicides were applied at the time when herbicide applications typically occur in North Macedonia sunflower production and are thus representative of producer practices and label recommendations. However, in 2018 in the Bitola region, heavy precipitation occurred in the 1st WAA, which caused the leaching of herbicides through the soil profile. Possible that sunflower injury was due to higher amounts of rain (34 mm) directly following PRE-em herbicide treatments. It ranged from 9 to 28% across PRE-em treatments seven days after emergence (DAE). Injuries of oxyfluorfen and dimethenamid-P were more serious (24 and 28%, respectively). Oxyfluorfen showed phytotoxicity symptoms like slight bleaching, leaf tip burn, and stunting of sunflower growth. Stunting of sunflower growth was recorded in plots treating with dimethenamid-P, as well. Injuries caused by other PRE-em herbicides decreased by seven and 21 DAA (Table 6). However, sunflower injuries of oxyfluorfen and dimethenamid were still evident at 21 DAE. In the same line are investigations by Jursik *et al.* (2015) who concluded that the sunflower phytotoxicity caused by oxyfluorfen was the highest (25–47%) without the effect of irrigation. Sunflower growth was inhibited and regeneration was slow; however, the seed yield was not significantly reduced in any year. Similar, in the study of Andr *et al.* (2017) the highest level of sunflower injury was recorded on plots treated by oxyfluorfen

(18%). The injury caused by oxyfluorfen on sunflower was mainly caused by raindrops bouncing from the soil surface, which contaminated leaves and caused necrosis and leaf deformation. Further, the sunflower tolerance to dimethenamid-P was good (phytotoxicity less than 7%), except in the year when sunflower injury ranged from 10–12% across irrigation treatments. On the other side, the sunflower injury caused by pendimethalin, aclonifen, and S-metolachlor was minimal (between 5 and 7%) (Jursik *et al.*, 2015).

Sunflower yield

Sunflower grain yields for each treatment in both regions generally reflected overall weed control and crop injury (Table 6). Comparison of weed and weed-free control indicated that weeds reduced sunflower grain yield by 72–75% in the Bitola region, and 72–76% in the Titov Veles region for both years, respectively (Table 6). Similar, Jaykumar *et al.* (1988), Elezovic *et al.* (2012), and Alves *et al.* (2013) reported the yield reduction due to weeds in sunflower is estimated to be between 70 and 81%. A significant treatment by year interaction resulted in two distinct years for sunflower grain yield in the Bitola region. In both years, the lowest sunflower grain yield was recorded in untreated control plots (980 and 850 kg ha⁻¹, respectively). The lowest yield between PRE-em herbicides in 2018 was obtained in plots treated with aclonifen (2 030 kg ha⁻¹). No one of PRE-em applied herbicides yielded higher than the weed-free control, because sunflower yields were more closely related to the per cent of weeds control. In 2019 the effective removal of the competitive effect of the weeds led in an increase of the sunflower yield in all PRE-em herbicide treatments significantly increased and resulted in yields similar to that of the weed-free control (Table 6). A significant treatment by year interaction resulted in two distinct years for sunflower yields in the Titov Veles region with PRE-em herbicides, as well. In 2018 sunflower yields was on the line with that of weed-free control. It was ranged from 810 to 3 680 kg ha⁻¹. Aclonifen was the lowest-yielding herbicide treatment with 3 505 kg ha⁻¹, whereas oxyfluorfen was the highest yielding herbicide treatment (3 680 kg ha⁻¹). In 2019 sunflower yields following all PRE-em applied herbicides were significantly lower (between –610 and –760 kg ha⁻¹) than weed-free control (Table 6). In investigation of Pannacci *et al.* (2007) the highest average sunflower yields among PRE-em treatments were obtained in plots treated with S-metolachlor + oxyfluorfen (720 + 168 and 960 + 144 g a.i. ha⁻¹), S-metolachlor + aclonifen (960 + 720 g a.i. ha⁻¹) and pendimethalin + imazamethabenz (768 + 400 g a.i. ha⁻¹). Sunflower yield in pendimethalin and oxyfluorfen treated plots was 46 and 63% higher than in weedy control (Narender *et al.*, 2017). Regardless of irrigation and sunflower injury, in all investigated PRE-em herbicides yield was significantly higher in comparison to untreated control plots (Jursik *et al.*, 2015).

Table 6. Sunflower plant injury as influenced by PRE-em applied herbicides, and yield of sunflower in Bitola and Titov Veles region in 2018 and 2019 ^{a-d}

Treatments	Rate (1 ha ⁻¹)	Bitola region				Titov Veles region				Grain yield, kg ha ⁻¹			
		Sunflower injury		Grain yield, kg ha ⁻¹		Sunflower injury		Grain yield, kg ha ⁻¹		Grain yield, kg ha ⁻¹			
		2018	2019	2018	2019	2018	2019	2018	2019	2018	2019		
		7 DAE	21 DAE	7 DAE	21 DAE	7 DAE	21 DAE	7 DAE	21 DAE	7 DAE	21 DAE		
Weedy control		0	0	0	0	980 ^d	850 ^e	0	0	0	0	810 ^d	950 ^d
Weed-free control		0	0	0	0	3490 ^a	3340 ^{abc}	0	0	0	0	3670 ^a	3410 ^a
Pendimethalin	3.0	11	7	0	0	2320 ^b	3390 ^a	0	0	0	0	3620 ^{ab}	2740 ^{bc}
Metobromuron	3.0	14	9	0	0	2090 ^c	3220 ^{bcd}	0	0	0	0	3540 ^{bc}	2620 ^c
Oxyfluorfen	1.25	24	20	0	0	2170 ^{bc}	3330 ^{abc}	0	0	0	0	3680 ^a	2800 ^b
Aclonifen	4.0	9	6	0	0	2030 ^c	3145 ^d	0	0	0	0	3505 ^c	2650 ^c
S-metolachlor	1.5	15	11	0	0	2270 ^{bc}	3350 ^{ab}	0	0	0	0	3580 ^{abc}	2700 ^{bc}
Dimethenamid	1.7	28	22	0	0	2080 ^c	3210 ^{cd}	0	0	0	0	3540 ^{bc}	2780 ^b
LSD 0.05						195.96	170.50					99.52	146.55
Random effect interactions													
PRE-em herbicides × year			*			*			NS			*	

^aAbbreviation: PRE-em; DAA – days after application; NS – not significant; * Significant at the 5% level according to a Fisher's protected LSD test at P < 0.05

^bPRE treatments were applied in the same growth stages as sunflower (at the dry seed – beginning of seed imbibitions of sunflower growing stage – BBCH 00–01).

^cSunflower injury was estimated 7 and 21 days after emergence (DAE)

^dMeans followed by the same letter within a column are not significantly different according to Fisher's Protected LSD at P < 0.05

Conclusion

The efficacy of the PRE-em herbicides was correlated with the weather and soil conditions in both regions. The humid April in Bitola region in 2018, before weed emergence, caused herbicide leaching from soil surface which probably was the most likely reason for the lower efficacy of PRE-em herbicides in 2018 compared to their application in 2019. However, in the region of Titov Veles due to equal precipitation, particularly a few weeks after herbicide application and soil type characteristics leaching did not occur and efficacy was good to excellent. Opposite, the limited precipitation after PRE-em application contributed to the poor performance of PRE-em herbicides in the Titov Veles region in 2019 compared with 2018. The sunflower injury occurred due to heavy precipitations in the Bitola region in 2018. Sunflower grain yields for each treatment in both regions generally reflected overall weed control and crop injury. Based on the results we can recommend to the producers that the use of PRE-em herbicides in sunflower crop should be based on climatic conditions with special emphasis on rainfall and soil type.

Conflict of interest

The authors declare that there is no conflict of interest regarding the publication of this paper.

Author contributions

ZP, AM – contributed to the preparation, creation and/or presentation of the manuscript.

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