

Pre-emergence grass weed control in winter wheat (*Triticum aestivum* L.) with soil applied premixed herbicides influenced by precipitations

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Abstract. The field trials were conducted during two winter wheat growing seasons (2013–2014 and 2014–2015, respectively) to estimate weed control, and influence of herbicides on grain yield with PRE premixes in winter wheat crops. The field trials were conducted with ‘Ingenio’ and ‘Pobeda’ winter wheat cultivars which were sowed in a well-prepared soil seedbed at a seeding rate of 220 and 240 kg ha⁻¹. The experimental design was a randomised complete block with four replicates and elementary plots 25 m². The efficacy of PRE herbicides varied with treatments among weed species and periods of efficacy estimation, regions and years, respectively. Overall, the performance of the PRE herbicide premixes correlated with the weather conditions. All PRE herbicide premixes effectively reduced the dominant weed species *Milium vernale*, *Papaver rhoeas*, and *Galium aparine* in the Bitola region in 2013–2014, but not in 2014–2015 due to heavy rain during the first two weeks of herbicide application. In contrast, the limited precipitation after PRE application may have contributed to the poor performance of PRE herbicides in the Probištip region in 2013 compared with 2014. In the Bitola region, the lowest crop yield was obtained in plots treated with diflufenican + isoproturon (2,960 kg ha⁻¹) in both growing seasons. In the Probištip region, the wheat grain yields in 2013–2014 following all PRE applied herbicides were significantly lower (between 520 and 800 kg ha⁻¹) than weed-free control. In 2014–2015, diflufenican + isoproturon herbicide treatment produced the lowest yield of 2,530 kg ha⁻¹, whereas chlortoluron + triasulfuron was the highest-yielding herbicide treatment (2,820 kg ha⁻¹). However, results indicated that in Bitola region comparatively higher yield were found in plots treated with chlortoluron + triasulfuron (3,450 kg ha⁻¹), in both growing seasons, also in Probištip region herbicide chlortoluron + triasulfuron achieved higher yield (2,820 kg ha⁻¹), in both growing seasons).

Key words: PRE herbicide, efficacy, weeds, wheat.

INTRODUCTION

Winter wheat is important crop grown on approximately 15% of the arable land in Republic of Macedonia (Anonymous, 2017). It is a popular rotational crop helping to maintain soil structure and break weed cycles, as weeds comprise the most undesirable,

aggressive and troublesome element of crop production (Kazi et al., 2007). Among the factors which adversely affect wheat crop yields, weed infestation is the most harmful, but less noticeable (Oad et al., 2007). Wheat often suffers from competition from numerous weed species, where the reduction of wheat yields due to weed infestation can reach on average up to 23% globally (Oerke, 2006).

For this reason, one of the most significant aspects of winter wheat production is weed management. Substantial crop yield and associated economic losses can occur if weeds are not adequately controlled. Under certain conditions, weeds have their greatest detrimental effect on the ultimate yield of winter wheat during the early stages of the crop development (Pilipavičius, 2012), so the pre-emergence herbicides can play a valuable role in controlling weeds at these critical growth stages (Shehzad et al., 2012; Kazmarek & Matysiak, 2015; Kaur et al., 2018). Pre-emergent herbicides offer an alternate mode of action to many post-emergent options, reducing selection pressure on subsequent post-emergent herbicide applications and removing much of the early season weed competitive pressure on crops by controlling weeds that emerge with winter wheat.

Herbicides registered for PRE weed control in winter wheat in the Republic of Macedonia have significantly changed over the past 4–5 years. ‘Forgotten’ and ‘old’ well-known herbicide active ingredients are in favour once again. Among them, different combinations of pendimethalin, isoproturon, chlortoluron and isoxaben are the most frequently applied. Soil residual activity may be maintained for 16 (chlortoluron) (Tomlin, 1994) to 20–25 weeks (pendimethalin, izoxaben and isoproturon) (Vouzounis & Americanos, 1995; Alletto et al., 2006) insuring long lasting weed control over the prolonged period of weed emergence.

The main objectives of the research were (i) to determine whether acceptable weed control of PRE premixes in winter wheat crop may replace usually spring POST-applied herbicides, and (ii) to evaluate their injury effect and influence on the winter wheat yield.

MATERIALS AND METHODS

The field trials were conducted during two winter wheat growing seasons in 2013–2014 and 2014–2015 on commercial wheat fields in the Bitola and Probištip wheat growing regions in south-western and north-eastern Macedonia on mollic-vertic gleysol and vertisol, respectively (Filipovski, 2006) (Table 1).

Table 1. Soil characteristics in the wheat-growing regions

Region	Soil	coarse, %	fine sand, %	clay+silt, %	organic matter, %	pH-water, %
Bitola	mollic-vertic gelysol	27.10	47.30	25.60	1.86	6.30
Probištip	Vertisol	3.50	30.00	60.30	2.40	7.20

The wheat was grown following conventional tillage practices. The soil was tilled with a field cultivator prior to sowing. Nitrogen, phosphorus and potassium were applied as per soil test-based recommendation. The field trials were carried out with ‘Ingenio’ and ‘Pobeda’ winter wheat cultivars sowed in a well-prepared soil at a seeding rate of 220 and 240 kg ha⁻¹ on 10th October 2013 and 18th October 2014 in the Bitola region, and on 12th October 2013 and 21st October 2014 in the Probištip region, respectively.

The trials were conducted in two different sites of the same commercial wheat fields. Herbicides were applied with a CO₂-pressurised backpack sprayer calibrated to deliver 300 L ha⁻¹ solution at 220 kPa. Herbicides were applied to the dry seed, at the beginning of the seed imbibitions wheat growing stage (BBCH 00-01). Weeds at the time of treatment were in the same growth stages as wheat (BBCH 00-01). The experimental design was a randomised complete block with four replicates and elementary plots 25 m², in total were six treatments (Table 2).

Table 2. Basic data for applied herbicides

Treatments	Trade name	Rate	Time of application
chlortoluron 500 g L ⁻¹ + isoxaben 18.7 g L ⁻¹	Aubaine	3.6 L ha ⁻¹	pre-emergence
pendimethalin 250 g L ⁻¹ + isoproturon 125 g L ⁻¹	Maraton 375 SC	4.0 L ha ⁻¹	pre-emergence
chlortoluron 500 g L ⁻¹ + triasulfuron 750 g kg ⁻¹	Tolurex 50 SC Logran 75 WG	3.2 L ha ⁻¹ 0.037 kg ha ⁻¹	pre-emergence
diflufenican 50 g L ⁻¹ + isoproturon 500 g L ⁻¹	Quartz Super	1.6 L ha ⁻¹	pre-emergence
Weed free control			
Control (untreated)			

The weed control plots were left untreated during the entire experimental period. Weed-free control was maintained by hand weeding, which was initiated at weed emergence and continued as required to maintain weed-free plots.

Weed control efficacy was estimated 28 days after application (DAA) (the first estimation) and in the spring, 150 DAA at the middle of wheat tillering stage (BBCH 24-26) (the second estimation) by counting the weed plants in a 1 m² area within each plot. Herbicide efficacy was calculated by equation according to Hasanuzzaman et al. (2008):

$$W_{ce} = \frac{W_{up} - W_{tp}}{W_{up}} \cdot 100 \quad (1)$$

where W_{ce} – weed control efficiency; W_{up} – number of weeds in the untreated plots; W_{tp} – number of weeds in the treated plots.

Wheat injury was visually evaluated based on a 0–100% rating scale, where 0 is no injury to wheat plants and 100 is complete death of wheat plants (Frans et al., 1986). Visual estimates of the percentage wheat 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. At full maturity, wheat was harvested manually at ground level in an area of 1 m² per plot and the yield was determined after harvest based on weights of grain containing 13% moisture.

Total weekly rainfall, as well as weekly average temperatures 1 week before and 4 weeks after PRE applications were recorded, respectively (Table 3). Precipitation 1 week before and 4 weeks after PRE applications in 2013 were in line with the average for the Bitola region, but scarce for the Probištup region. In the Bitola region, precipitation occurred on the 2nd day of the week before PRE applications, and on the first 2 and the last day of the 1st and the last 2 days of the 3rd week after PRE applications. In contrast, in the Probištup region in the same year, it rained on 7 and 11 days at intervals throughout the 1- and 4-week period before and after PRE applications. Furthermore,

the 4-week period after PRE application in autumn of 2014 in Bitola region was unusually wet, particularly the 2nd, 3rd, and 4th day of the 1st week, as well as 3 days in the middle of the 2nd and the last 2 days of 4th week. This period was also very humid, 56% above the 30-year average for the Bitola region (106 mm). There was no precipitation 1 week before PRE application, however, precipitation occurred in the 1st, 2nd, and 4th week after PRE application, as well as in the 1st week before PRE applications in the Probištip region for the same year, in the line with the average for this region (11, 13, 8, and 14 mm). The 3rd week was wetter in comparison with previous weeks (23 mm) (Table 3).

The temperature 1 week before and 4 weeks after PRE application for both years and regions was slightly above the average and that was attributed to favourable environmental conditions associated with no night frosts during the estimated 1- and 4-week period before and after PRE application. PRE treatments in both years were applied at times when herbicide applications typically occur in Macedonia wheat production, thus are representative of producer practices and label recommendations.

The data were tested for homogeneity of variance and normality of distribution (Ramsey & Schafer, 1997) and were log-transformed as required to obtain equal variances and better symmetry before ANOVA was performed. Data were transformed back to their original scale for presentation. Means were separated using the LSD test at 5% probability.

Table 3. Mean weekly temperatures and total weekly rainfall 1 week before application (WBA) and 4 weeks after application (WAA) PRE applications, respectively at Bitola and Probištip region in 2013 and 2014

	Bitola region				Probištip region			
	2013		2014		2013		2014	
Weeks	P (mm)	T (°C)	P (mm)	T (°C)	P (mm)	T (°C)	P (mm)	T (°C)
1 st WBA	8	13	0	15	5	15	11	17
1 st WAA	14	9	37	10	7	10	13	14
2 nd WAA	10	11	31	7	2	14	8	11
3 rd WAA	21	8	11	8	9	9	23	12
4 th WAA	6	6	27	7	13	9	14	9

RESULTS AND DISCUSSION

Weed control. Efficacy of PRE herbicides varied among weed species, treatments, periods of efficacy estimation, regions and years. Overall, the performance of the PRE herbicide premixes correlated with the weather conditions. Both sites were naturally infested with a high population of *Milium vernale* M. Bieb., *Papaver rhoeas* L., *Galium aparine* L. *Avena ludoviciana* Dur., *Alopecurus myosuroides* Huds., *Bifora radians* M.B. and *Polygonum convolvulus* L. Inconsistent weather patterns between the two study growing seasons probably influenced weed control. The humid autumn in 2014 (Table 3), particularly heavy rain (68 mm) during the 1st and 2nd week of herbicide applications and before weed emergence, may have promoted leaching of herbicides from the soil surface, resulting in the lower efficacy of PRE applied premixes in 2014 compared to 2013 in the Bitola region (Table 3). Weed density was higher due to the climatic condition in the non-treated control plot was 93 and 135 plants m⁻² in 2013–2014 and 2014–2015, respectively in the Bitola region, and 85 and 104 plants m⁻² in 2013–2014 and 2014–2015 respectively in the Probištip region. In contrast, the limited precipitation after PRE

application may have contributed to the poor performance of PRE herbicides in the Probištip region in 2013 compared with 2014 (Table 3).

***Milium vernale*.** The control of *M. vernale* in Bitola region was significant among years with PRE herbicides, but *M. vernale* control did not differ between periods of efficacy estimation by year. In this region in 2013–2014, 28 DAA, all PRE applied premixes controlled *M. vernale* between 87 and 100%, whereas excellent control (> 95%) was achieved with chlortoluron + isoxaben and chlortoluron + triasulfuron. Negligible lower *M. vernale* control was obtained 150 DAA with all herbicide treatments in 2014–15. The efficacy of all PRE applied premixes ranged between 82% and 96%, probably due to the residual activity of PRE herbicides and no new spring growth of *M. vernale*, a typical winter weed (Kostov, 2006). In contrast, all PRE herbicide premixes provided poor control of *M. vernale* in 2014–2015, regardless of the period of efficacy estimation, the herbicides controlled *M. vernale* between 58 and 78% (Table 4). The differences in the control of *M. vernale* between the years indicated that higher amounts of precipitation (37 mm) after herbicide application in 2014–2015 caused herbicides to leach through the soil profile, consequently decreasing weed control efficacy (Ferrell et al., 2004; Tanji & Boutfirass, 2018).

***Papaver rhoeas*.** The control of *P. rhoeas* in Bitola region was significant among years with PRE herbicides, while *P. rhoeas* control did not differ between the periods of efficacy estimation by year. A significant treatment by year interaction resulted in two distinct years for *P. rhoeas* control at Bitola region with PRE herbicide premixes. but *P. rhoeas* control did not differ among periods of efficacy estimation by year. PRE herbicide premixes controlled *P. rhoeas* more than 98% at 28 DAA in 2013–2014 and with similar efficacy at 150 DAA. PRE applied premixes in 2014–2015 did not achieve more than 74% (28 DAA) and 70% (150 DAA) control of *P. rhoeas*, respectively (Table 4). PRE herbicides require precipitation to move into the zone of active weed seed germination (Chomas & Kells, 2004), so heavy precipitation immediately after PRE applications in 2014 and leaching of herbicides out of the weed seed germination zone caused significant variability among PRE treatments in the control of *P. rhoeas* in both years. The efficacy of PRE applied mixtures pendimethalin + linuron, beflubutamid + isoproturon, and pyraflufen + isoproturon in the control of *P. rhoeas* in winter cereals ranged from 93 to 100% (Torra et al., 2010). These values are within the efficacy range for *P. rhoeas* control reported by Cirujeda et al. (2000) and Garcia de Arevalo et al. (1992) reported that similar mixtures of PRE herbicides controlled *P. rhoeas* in field beans.

***Galium aparine*.** A significant treatment by year interaction resulted in two distinct years for *G. aparine* control in Bitola region with PRE herbicide premixes. *G. aparine* control did not differ between the periods of efficacy estimation by year. In 2013–2014, all investigated PRE premixes provided more than 95% control of *G. aparine* for both periods of efficacy estimation. However, the control drastically decreased the following year, probably due to the Bitola region receiving 68 mm precipitation in the 1st and 2nd week of herbicide applications. It is likely that these extremely humid conditions contributed to the decrease in efficacy of PRE herbicides, which ranged between 57% and 67% (Table 4). Isoproturon tank-mixed with pendimethalin provided the most satisfactory residual control of *G. aparine* in winter wheat (Gianessiet al., 2003). Lovegrove et al. (1985) assessed the efficacy of a range of PRE herbicides for the control

of *G. aparine*, reporting that pre-emergence applications of pendimethalin, trifluralin with linuron and bifenox with linuron gave inadequate control.

***Avena ludoviciana*.** *Avena ludoviciana* control did not differ among regions, years, and periods of efficacy estimation for PRE herbicide premixes. PRE herbicides, regardless of the growing season, had little effect on *A. ludoviciana* in both regions. In the Bitola region, control of *A. ludoviciana* was less than 69% and 63% with any PRE treatment 28 DAA and 150 DAA, respectively in 2013–2014, with similar efficacy (< 63% and < 58%, respectively) recorded in 2014–2015 (Table 4). In the Probištíp region, PRE herbicide premixes provided no more than 49% and 67% control of *A. ludoviciana* in 2013–2014 and 2014–2015, respectively (Table 5). In general, pre-emergence herbicides do not effectively control *A. ludoviciana* (Thomas & Yaduraju 2000), because the seeds of *A. ludoviciana* have the ability to germinate from greater depths in the soil (under certain conditions from depths as great as 25 cm) (Kostov, 2006). Dormancy and multiple emerging flushes throughout the growing season results in the persistence and continual re-infestation of this weed in the soil seed bank (Fuerst et al., 2011; Beckie et al., 2012). Poor wild oat control and, at the same time, the highest dry weights of this weed (9.43, 8.70 and 8.16 g m⁻²), were recorded in plots treated with isoproturon at 1.5 a.i kg ha⁻¹, isoproturon + diflufenicon at 0.98 a.i. kg ha⁻¹, isoproturon + carfentrazone ethyl at 2.0 a.i. kg ha⁻¹, respectively (Shehzad et al., 2012). In contrast, Singh and Gosh (1992) reported that the application of pendimethalin and isoproturon before emergence provided maximal *A. ludoviciana* control.

***Alopecurus myosuroides*.** A significant treatment by year interaction resulted in two distinct years for *A. myosuroides* control in Probištíp region with PRE herbicides. However, the control of *A. myosuroides* did not differ among periods of efficacy estimation by year. In 2013–2014, PRE herbicides provided lower efficacy for control of *A. myosuroides* 52% and 61% (28 DAA) and 48% and 59% (150 DAA). The lack of effective control of *A. myosuroides* can be attributed to the low rainfall after PRE application in 2013 (Table 3). Since many of the PRE herbicides can volatilise and photodegrade on the soil surface over time, rainfall is need to transfer these herbicides into the zone where weed seeds germinate (Janak & Grichar, 2016), which explains the inconsistent control of *A. myosuroides* observed with PRE herbicide premixes under the drought conditions in the Probištíp region in autumn 2014. Control of *A. myosuroides* improved in 2014–2015, due to the sufficient precipitation (11 mm 1st WBA and 13 mm 1st WAA, respectively) increasing the efficacy of the PRE herbicide premixes; 28 DAA, all PRE applied premixes controlled *A. myosuroides* between 92 and 100%. The high efficacy in *A. myosuroides* control was maintained 150 DAA, as *A. myosuroides* shares the same growth cycle as autumn-sowed wheat (Orson & Thomas, 2001), which means it is typical winter weed (Kostov, 2006) and no new spring growth was recorded. Diflufenican + isoproturon provided at least 87% control of *A. myosuroides*, while pendimethalin + isoproturon, chlortoluron + isoxaben, and chlortoluron + triasulfuron provided better control than diflufenican + isoproturon (95–100%) (Table 5). Single applications of isoproturon or chlorotoluron in the UK have been reported to effectively control *A. myosuroides* (Orson & Harris, 1997). According Moss et al. (2009), PRE flufenacet + pendimethalin (240 + 1,200 g a.i. ha) achieved 93% reduction of *A. myosuroides*, while PRE flufenacet + pendimethalin followed by isoproturon + pendimethalin (1,500 + 1,320 g a.i. ha) achieved 97% reduction.

Table 4. *Milium vernale*, *Papaver rhoeas*, *Galium aparine* and *Avena ludoviciana* control (%) 28 and 150 days after PRE herbicide applications, in winter wheat in 2013–2014 and 2014–2015 in Bitola region

Treatments	Bitola region															
	MIVER				GALAP				PAPRH				AVELU			
	2013–2014		2014–2015		2013–2014		2014–2015		2013–2014		2014–2015		2013–2014		2014–2015	
	28	150	28	150	28	150	28	150	28	150	28	150	28	150	28	150
DAA	DAA	DAA	DAA	DAA	DAA	DAA	DAA	DAA	DAA	DAA	DAA	DAA	DAA	DAA	DAA	DAA
Weedy control	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
chlortoluron+ isoxaben	95 ^a	92 ^a	74 ^{ab}	66 ^{ab}	100 ^a	100 ^a	66 ^a	62 ^a	100 ^a	100 ^a	74 ^a	70 ^a	58 ^b	55 ^a	63 ^a	58 ^a
pendimethalin+ isoproturon	87 ^b	83 ^b	68 ^{bc}	61 ^{bc}	95 ^b	100 ^a	63 ^{ab}	57 ^{ab}	98 ^a	100 ^a	68 ^{ab}	66 ^{ab}	54 ^b	57 ^a	59 ^{ab}	53 ^a
chlortoluron + triasulfuron	100 ^a	96 ^a	78 ^a	69 ^a	100 ^a	100 ^a	67 ^a	64 ^a	100 ^a	100 ^a	69 ^{ab}	64 ^b	67 ^a	63 ^a	55 ^b	52 ^a
diflufenican + isoproturon	89 ^b	82 ^b	65 ^c	56 ^c	97 ^{ab}	98 ^a	59 ^b	50 ^b	100 ^a	100 ^a	65 ^b	63 ^b	69 ^a	59 ^a	63 ^a	54 ^a
LSD 0.05	5.27	5.76	8.36	6.35	3.05	2.00	6.15	7.35	2.45	1.01	8.03	5.46	5.89	9.08	6.09	8.81
Random effect interactions PRE herbicides treatment x year	*				*				*				NS			
PRE herbicides treatment x PEE	NS		*		NS		NS		NS		NS		NS		NS	

^aAbbreviation: PRE – preemergence; DAA – days after application; MIVER – *Milium vernale*; PAPRH – *Papaver rhoeas*; AVELU – *Avena ludoviciana*; GALAP – *Galium aparine*; PEE – periods of efficacy estimation; NS – not significant; * Significant at the 5% level according to a Fisher’s protected LSD testat $P < 0.05$.

PRE treatments were applied in the same growth stages as wheat (beginning of seed imbibitions wheat growing stage – BBCH 00-01).

Weed control efficacy was estimated 28 DAA and 180 DAA.

Means followed by the same letter within a column are not significantly different according to Fisher’s Protected LSD at $P < 0.05$.

Table 5. *Avena ludoviciana*, *Alopecurus myosuroides*, *Bifora radians* and *Polygonum convolvulus* control (%) 28 and 150 days after PRE herbicide applications, in winter wheat in 2013–2014 and 2014–2015 in Probištip region

Treatments	Probištip region															
	AVELU				ALOMY				BIFRA				POLCO			
	2013–2014		2014–2015		2013–2014		2014–2015		2013–2014		2014–2015		2013–2014		2014–2015	
	28 DAA	150 DAA	28 DAA	150 DAA	28 DAA	150 DAA	28 DAA	150 DAA	28 DAA	150 DAA						
Weedy control	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
chlortoluron + isoxaben	44 ^a	47 ^a	61 ^{ab}	63 ^a	57 ^{ab}	59 ^a	98 ^a	96 ^a	-	52 ^{ab}	-	58 ^a	-	49 ^{ab}	-	55 ^{ab}
pendimethalin + isoproturon	49 ^a	41 ^a	59 ^{ab}	64 ^a	53 ^{ab}	49 ^b	95 ^{ab}	95 ^a	-	48 ^{bc}	-	54 ^a	-	51 ^a	-	57 ^a
chlortoluron + triasulfuron	37 ^b	43 ^a	67 ^a	62 ^a	61 ^a	57 ^a	100 ^a	100 ^a	-	54 ^a	-	57 ^a	-	50 ^a	-	52 ^{ab}
diflufenican + isoproturon	28 ^c	25 ^b	55 ^b	53 ^b	52 ^b	48 ^b	92 ^b	87 ^b	-	45 ^c	-	52 ^a	-	42 ^b	-	48 ^b
LSD 0.05	6.52	7.19	8.56	7.72	8.05	7.15	4.40	5.29	-	5.67	-	7.38	-	7.58	-	8.09
Random effect interactions PRE herbicides treatment x year	NS				*				NS				NS			
PRE herbicides treatment x PEE	NS		NS		NS		NS		-		-		-		-	

Abbreviation: PRE – preemergence; DAA – days after application; AVELU – *Avena ludoviciana*; ALOMY – *Alopecurus myosuroides*; BIFRA – *Bifora radians*; POLCO – *Polygonum convolvulus*; PEE – periods of efficacy estimation; NS – not significant; * Significant at the 5% level according to a Fisher’s protected LSD test at $P < 0.05$.

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

Weed control efficacy was estimated 28 DAA and 180 DAA.

Means followed by the same letter within a column are not significantly different according to Fisher’s Protected LSD at $P < 0.05$.

Table 6. Wheat plant injury as influenced by PRE applied herbicide premixes, and grain yield as influenced by PRE applied herbicide premixes in winter wheat in Bitola and Probištip region in 2013–2014 and 2014–2015^{a-d}

Treatments	Rate (g L ha ⁻¹)	Bitola region						Probištip region					
		Wheat injury				Grain yield (kg ha ⁻¹)		Wheat injury				Grain yield (kg ha ⁻¹)	
		2013–2014		2014–2015		2013–2014	2014–2015	2013–2014		2014–2015		2013–2014	2014–2015
		7	21	7	21			7	21	7	21		
DAE		DAE				DAE		DAE					
Weedy control	-----	0	0	0	0	2,180 ^e	2,060 ^d	0	0	0	0	1,760 ^d	2,040 ^d
Weed-free control	-----	0	0	0	0	3,910 ^a	3,940 ^a	0	0	0	0	3,110 ^a	3,570 ^a
chlortoluron+ isoxaben	3.6	0	0	7	2	3,380 ^{bc}	2,880 ^b	0	0	0	0	2,430 ^{bc}	2,780 ^b
pendimethalin + isoproturon	4.0	0	0	20	16	3,230 ^c	2,750 ^c	0	0	0	0	2,370 ^{bc}	2,580 ^c
chlortoluron + triasulfuron	3.2 + 0.037	0	0	9	3	3,450 ^b	2,960 ^b	0	0	0	0	2,590 ^b	2,820 ^b
diflufenican + isoproturon	1.6	0	0	22	17	2,960 ^d	2,700 ^c	0	0	0	0	2,310 ^c	2,530 ^c
LSD 0.05						170.76	118.57					221.29	148.25
Random effect interactions PRE herbicides x year		*				*		NS				*	

Abbreviation: PRE – preemergence; DAA – days after application; NS – not significant; * Significant at the 5% level according to a Fisher’s protected LSD test at $P < 0.05$.

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

Wheat injury was estimated 7 and 21 days after emergence (DAE).

Means followed by the same letter within a column are not significantly different according to Fisher’s Protected LSD at $P < 0.05$.

Bifora radians and *Polygonum convolvulus*. Taking into consideration that *B. radians* and *P. convolvulus* are annual broadleaf early-germinating spring weeds (Mennan & Zandstra, 2005; Altop & Mennan, 2017; Majd et al., 2016), no data are presented regarding the efficacy of PRE herbicide premixes 28 DAA for both years in the Probištíp region. Soil-applied herbicides controlled *B. radians* and *P. convolvulus* 150 DAA due to their soil residual activity. However, the efficacy of PRE herbicide premixes was poor and did not differ among years, providing no more than 54% and 63% control of *B. radians* in 2013–2014 and 2014–2015, respectively. Identical efficacy was recorded for *P. convolvulus* control for both years (51% and 62%, respectively) (Table 5). Similarly, Durbin (2017) reported that PRE herbicides applied in late fall/winter cannot be fully effective because seeds of *P. convolvulus* germinate in early spring. Marković et al. (2005) also reported that chlorotoluron is insufficiently effective on many weeds, including *Bifora radians*.

Wheat injury. Higher precipitation (68 mm) in autumn 2014 directly following PRE herbicide treatments caused wheat injury in the Bitola region, ranging from 7% to 22% across PRE treatments 7 days after emergence (DAE). However, injuries as a result of premixes containing diflufenican + isoproturon and pendimethalin + isoproturon were more serious (22 and 20%, respectively).

Furthermore, in 2014–2015 in Bitola region, heavy precipitation occurred in the 1st and 2nd week of herbicide applications, causing leaching of herbicides through the soil profile. Injuries caused by chlortoluron + isoxaben and chlortoluron + triasulfuron (premixes that contain chlortoluron) significantly decreased by 7 and 21 DAA (Table 6). However, wheat injuries of diflufenican + isoproturon and pendimethalin + isoproturon were still evident at 21 DAE. By the time, wheat compensated for early injuries in its growth, the crop fully recovered and did not suffer any yield reduction. PRE applied chlortoluron (2,000 g ha⁻¹) + isoxaben (74.8 g ha⁻¹), prosulfocarb (4,000 g ha⁻¹), prosulfocarb (2,000 g ha⁻¹) + s-metolachlor (300 g ha⁻¹), and pendimethalin (1,320 g ha⁻¹) reduced wheat emergence and density because of heavy rain (about 100 mm) after herbicide treatments and before crop emergence (Tanji & Boutfirass, 2018).

Wheat grain yield. Wheat grain yields for each treatment in both regions generally reflected overall weed control (Table 6). Comparison of weedy and weed-free control indicated that weeds reduced wheat grain yield by 44 and 48% in the Bitola region, and 43% in the Probištíp region for both years (Table 6). Similar, Kaur et al. (2018) noted that the season long growth of weeds reduced the wheat yield up to 38.5%. A significant treatment by year interaction resulted in two distinct years for wheat grain yield in the Bitola region. In both years, the lowest wheat grain yield was recorded in untreated control plots (2,180 and 2,060 kg ha⁻¹, respectively). The lowest yield between herbicide premixes in 2013–2014 was obtained in plots treated with diflufenican + isoproturon (2,960 kg ha⁻¹).

The yield was no higher than the weed-free control with any PRE applied herbicide in both growing seasons. Wheat yields were more closely related to the percentage weed control than wheat injury, with only the weed-free control producing a statistically higher wheat yield compared to all evaluated herbicides (Table 6). A significant treatment by year interaction also resulted in two distinct years for wheat yields in the Probištíp region with PRE herbicides. In 2013–2014, wheat grain yields following all PRE applied herbicides were significantly lower (between-520 and-800 kg ha⁻¹) than weed-free

control (Table 6), whereas in 2014–2015, wheat yields ranged from 2,040 to 3,570 kg ha⁻¹. Diflufenican + isoproturon was the lowest-yielding herbicide treatment with 2,530 kg ha⁻¹, whereas chlortoluron + triasulfuron was the highest-yielding herbicide treatment (2,820 kg ha⁻¹) (Table 6).

The increases in grain yield resulting from isoproturon+ diflufenican application amounted to 54.9% in the first season and 57.9% in the second season over the unweeded (El Metwally et al., 2015).

Moreover, Kieloch & Rola (2010) reported that impact of the mixture of pendimethalin + isoproturon on grain yield of Clever cultivar was observed only in the season with hard winter conditions. Application of pendimethalin 1.0 kg ha⁻¹ or metribuzin 0.21 kg ha⁻¹ or a tank-mix of pendimethalin 0.75 and 1.0 kg ha⁻¹ + metribuzin at 0.175 kg ha⁻¹ resulted in statistically similar wheat grain yields (4.68 and 4.74 t ha⁻¹) to two hand weeding (4.8 t ha⁻¹) (Kaur et al., 2018). Significantly, a negative linear relationship between winter wheat yield and the number of *Lolium rigidum* was reported in study by Tanji & Boutfirass (2018). According to their results, chlortoluron + isoxaben provided 93% control of *Lolium rigidum* and was the highest-yielding herbicide treatment with 9.8, 6.8 and 8.9 t ha⁻¹ in three separate experiments.

CONCLUSIONS

These results demonstrated that the efficacy of PRE herbicides in wheat crops are strongly dependent on the amount of precipitation and weed populations. The humid autumn in second year of study and particularly heavy rain after two weeks of herbicide applications probably resulted in leaching of herbicides from soil surface in Bitola region. Furthermore, for that efficacy of herbicides were lower in 2014 compared to 2013 in Bitola region. Consequently, chlortoluron + isoxaben, pendimethalin + isoproturon, chlortoluron + triasulfuron, and diflufenican + isoproturon effectively reduced the dominant *Milium vernale*, *Papaver rhoeas*, and *Galium aparine* in 2013–2014, but not in 2014–2015. In contrast, the limited precipitation 7 days before and after herbicide application in the Probištip region in 2013 reduced the efficacy of PRE herbicides that require precipitation for optimal activity and weed control. Dry or very humid environmental and soil conditions immediately after PRE autumn herbicide application were accompanied by poor control of the deeper germinating *A. ludoviciana*, as well as poor control of the spring germinating *B. radians* and *P. convolvulus*, indicating that any combination of PRE herbicides must be followed by POST herbicides for control of escaped and newly emerging weeds. Therefore, the amount of precipitation and weed flora should be considered when selecting the most appropriate PRE weed management strategy for winter wheat crops. Based on the results presented, we recommend the usage of triasulfuron+dicamba or tribenuron-methyl in the study region for successful weed control and high wheat grain yields.

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