



Sensitivity and terminal residues of various herbicides screened for the control of broomrape in tomato

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ABSTRACT

Sensitivity of sulfosulfuron, ethoxysulfuron, mesosulfuron + iodosulfuron (RM), glyphosate, metribuzin and imazethapyr against Himsona and Rocky hybrids of tomato and their residues in fruits and soil were evaluated in a field and screen house study during *Kharif* 2015 and 2016, respectively. Herbicides applied at different stages and doses caused injury to tomato seedlings in both hybrids up to 30 days after treatment (DAT). Phyto-toxicity was more prominent in case of mesosulfuron + iodosulfuron (RM) followed by ethoxysulfuron and glyphosate. Among both hybrids, herbicide toxicity was more in Rocky as compared to Himsona irrespective of dose and time of application. But crop recovered fully within 30 days in plots treated with sulfosulfuron at 25 g/ha either used as pre-plant incorporation (PPI) or pre-emergence (PE) and post emergence (PoE) applications of 25 g/ha twice on 15 and 45 DAT, respectively resulting in more number of fruits per plant and was on par with untreated check. For herbicides residues estimation, recovery experiments were performed by validation of analytical method at two fortification levels of 0.01 and 0.05 µg/g which gave average recoveries of different herbicides from 80.4 to 91.3%. The limit of detection (LOD) and limit of quantification (LOQ) of various herbicides were ranged from 0.003 to 0.01 µg/g. In tomato fruits, residues of these herbicides were below maximum residues limit (MRL) of 0.05 µg/g. Residues in soil ranged from 0.023 to 0.186 µg/g in various herbicide treatments. Sulfosulfuron application at 25 g/ha was found safe for *Orobanche* management in tomato.

INTRODUCTION

Broomrape (*Orobanche aegyptiaca*), an annual parasitic weed is one of the major biotic constraints to tomato, cauliflower, cabbage, mustard, sunflower, tobacco, fababean and lentil crops which extract all nutrients, water and minerals from their host plants (Punia 2014, Punia and Duhan 2015). There have been reports of branched broomrape causing yield losses of up to 75% in tomatoes and 90% in rapeseed (Parker and Riches 1993, Eizenberg *et al.* 2004). In Haryana state, infestation of *O. aegyptiaca* locally known as Margoja/Rukhri/Khumbhi has been observed in tomato (*Lycopersicon esculentum* Mill) and mustard (*Brassica juncea* Czern and Cosson). During weed flora survey of tomato fields in 2014, the crop in Nuh, Ferozpur Jhirka, Nagina, Taoru areas of Mewat, Sahlawas of Jhajjar, Charkhi Dadri and Loharu of district Bhiwani was found seriously infested with this obnoxious weed threatening the cultivation of this crop in this region (Anonymous 2014).

The management of *Orobanche* is often difficult as it is closely associated with the host during its complete life cycle. Despite many management strategies like crop rotation and other mechanical weed control practices tried against broomrape, the use of selective herbicides were found most effective (Punia 2015, Punia and Duhan 2015). But, indiscriminate use of herbicides may lead to severe ecological consequences like residual carry over effects, destruction of natural enemy fauna, effect on non-target organisms, residues in food products and environmental factors like soil and water *etc.* Such treatments may suppress soil micro-flora and hence affect soil properties. Herbicides may have potential to bind to soil; the extent of which depends greatly on the nature of the chemical used. As per world trade organization (WTO) agreements, our agricultural produce must be free from pesticide residues, which can only be achieved through the application of modern production and protection logics. In order to avoid harmful effects of herbicides to mankind and ecosystem, top priority may be given to the use of

low dose eco-friendly herbicides. Study of herbicides used for weed control and estimation of their bio-efficacy, phyto-toxicity, persistence behaviour, half-life period, safe waiting period in consumable products and environmental factors provide good interdisciplinary correlation between agriculture and residue chemistry.

Keeping in view, the present study was designed to know the sensitivity of Himsona and Rocky hybrids of tomato to three sulfonyl-urea herbicides viz. sulfosulfuron, ethoxysulfuron, meso + iodosulfuron (RM) and a phosphono-glycine herbicide, glyphosate at different doses and time of applications. Selected doses of sulfosulfuron, ethoxysulfuron and glyphosate from field study were also evaluated in screen house along with two more herbicides- metribuzin and imazethapyr. The major objectives of this study was to evaluate these herbicides for their phyto-toxic effects on tomato hybrids before using best applications for effective control of *Orobanchae* infestation at farmers' field along-with estimation of herbicides residue in soil and tomato fruits.

MATERIALS AND METHODS

Field study

Tomato hybrids, Himsona and Rocky were planted in Vegetable Research Area of CCS Haryana Agricultural University Hisar, Haryana, India. Crop was raised as per university recommended package of practices for tomato except herbicide treatments in plots having 5.7 x 2.4 m size with factorial RBD design. The soil was sandy loam in texture with 65.5% sand, 18.1% silt and 16.3% clay, 0.4 EC (dS/m)², 7.6 pH and 0.35% organic carbon. There were 18 treatments having different application doses and time of applications (Table 1 and 2). All herbicides were sprayed by knap sack sprayer fitted with flat fan nozzle using 500 litres of water/ha. Observations on plant height and number of leaves per plant were recorded on 35 days after transplanting (DAT) while number of fruits per plant were recorded from five tagged plants at 90 DAT and averaged to compute values per plant. Tomato fruits picked in four flushes were weighed and thus total yield/ha was computed. These observations were subjected to ANOVA and means were compared with appropriate Fisher's protected LSD test at 5% level of significance. Crop phyto-toxicity due to different treatments was assessed at 30, 60 and 90 DAT on a scale of 0-10, where 0 means no injury and 10 means complete mortality of tomato plant. Foliar necrosis, yellowing, stunting and wilting were the main symptoms considered while making estimates of visual

injury on tomato plants. Injury data were arc sine transformed prior to analysis but expressed in their original form also for clarity.

Screen house studies

In another experiment performed at screen house, only selected doses of sulfosulfuron, ethoxysulfuron and glyphosate as tested from field study along with two other herbicides metribuzin and imazethapyr were applied for only screening of herbicides sensitivity against the tomato crop. Seedlings of Himsona hybrid planted for field studies, were used for the pot experiment using plastic pots of 30 cm height and top diameter. Pots were filled with field soil mixed with vermi-compost (4:1 ratio by volume) and inoculated with *Orobanche* seed collected from affected tomato fields of Mewat area. Tomato seedlings were transplanted on 25 January - 2016 with three plants per pot. Herbicides, sulfosulfuron, metribuzin, imazethapyr, ethoxysulfuron and glyphosate were applied at different rates and time with three pots per treatment. PE application of sulfosulfuron (50 and 75 g/ha), metribuzin (125 and 250 g), imazethapyr (50 and 75 g) and ethoxysulfuron (50 and 75 g) were sprayed using backpack sprayer fitted with flat fan nozzles delivering a spray volume of 500 l/ha, before transplanting tomato seedlings. PoE application of the above herbicides and glyphosate (50 and 75 g/ha) was done at 30 DAT and 50 DAT for sequential sprays. Untreated control with and without *Orobanche* seed inoculation was maintained for comparison. Total 36 treatments were arranged in a CRD design in the screen house. Plants were watered as and when required. Since no emergence of *Orobanche* was observed, data on herbicide selectivity was recorded. Visual mortality (%) was recorded on 20, 35, 45 and 65 days after spray at 0-10 scale. Fruit number and yield was recorded six times from April to May and data was summed for fruits per plant and weight for ANOVA using SPSS. One way ANOVA was performed to separate effect of herbicides.

Herbicides residue study

The residue analysis was carried in the Agrochemicals Residues Testing Laboratory at Department of Agronomy, CCS Haryana Agricultural University, Hisar. Tomato and soil samples were collected in triplicate at crop maturity from experimental trial conducted for screening of different herbicides with two tomato hybrids.

Chemicals and reagents

The technical grade analytical standard of sulfosulfuron, ethoxysulfuron, mesosulfuron,

iodosulfuron and glyphosate were procured from Fluka Sigma Aldrich, Germany. Other chemicals like acetonitrile (HPLC grade), analytical grade ammonium carbonate, sodium chloride, sodium sulfate (anhydrous), dichloromethane, ammonium hydroxide, methanol, HCl, phosphoric acid, trifluoroacetic anhydride, trifluoroethanol, ethyl acetate were purchased from Merck.

A standard stock solution of different sulfonyl-urea herbicides like sulfosulfuron, ethoxysulfuron, mesosulfuron and iodosulfuron were prepared in acetonitrile (HPLC grade). Standard stock solution of glyphosate was prepared in HPLC grade water (18 mΩ). The standard solutions required for constructing a calibration curve (0.003 to 1.0 µg/ml) were prepared from stock solution by serial dilutions with acetonitrile in case of different sulfonyl-urea herbicides and with HPLC grade water in case of glyphosate. All standard solutions were stored at 4°C before use.

Extraction and clean-up

Sulfosulfuron, ethoxysulfuron, meso + iodosulfuron (RM) were extracted by methods developed by Anjana *et al.* (2006) with slight modifications. For extraction of these herbicides fifty gram of the finely grinded, sieved soil and 25 g crushed tomato samples were taken in separate conical flasks and 50 ml of acetonitrile and ammonium carbonate mixture (9:1 v/v) was added to each flask. The flask was shaken for one hour on shaker and the content was filtered in another flask. The residues were again extracted with another 50 ml mixture of acetonitrile and ammonium carbonate (9:1 v/v). The content was again filtered in the same flask containing the first fraction. The combined content was concentrated on Heidolph rota-vapour to 20 ml at 40°C and was partitioned thrice (50, 30 and 20 ml) with dichloromethane after adding 20 ml of 10% brine solution. The combined dichloromethane extract was collected and passed through anhydrous sodium sulphate packed in a funnel so as to remove the moisture. Filtrate was collected, pooled and dried at 40°C on flash evaporator to near dryness. The residues were finally dissolved in 2 ml of HPLC grade acetonitrile and filtered through 0.45µm syringe filter before analysis on HPLC.

Extraction and clean-up of glyphosate from tomato and soil was achieved by the method of Hu *et al.* (2008). 50 g of dried and finely grinded sieved soil and 25 g of crushed tomato samples were taken in separate 250 ml conical flasks and extracted by shaking for one hour with 50 ml of 2M ammonium hydroxide. The process was repeated twice and the

combined extract was taken in 250 ml of spherical flask. The content was evaporated to dryness at 75°C under vacuum. The sample was dissolved with 5 ml of water: methanol: HCl (160:40:2.7 v/v) thrice and collected in a centrifugal tube. The tube was kept at room temperature for one hour and then centrifuged at 5000 rpm for 15 min. The supernatant was transferred to a derivatization tube and blown to dryness with stream of nitrogen at 80°C. The tube was cooled to room temperature and was added with 1 ml of trifluoroacetic anhydride (TFAA) and 0.5 ml of trifluoroethane (TFE). The derivatization tube was kept in ice cooled water during this process. The content was then heated at 100°C on an oil bath for one hour. The excess reagents were removed by gentle stream of nitrogen again at 40°C. The content obtained after derivatization tube was transferred in a separatory funnel, 20 ml of water was added and partitioned thrice with 50 ml of dichloromethane each time and collected in a separate conical flask by passing through 2 cm bed of anhydrous sodium sulphate taken in funnel. The combined dichloromethane content was dried over a rotary evaporator at 40°C. Finally the residues were reconstituted by dissolving in 2 ml of ethyl acetate, filtered through 0.45 µm syringe filter before analysis over GC-NPD.

Estimation

Analysis of the different herbicides mentioned above was carried using HPLC and GC-NPD. The instruments were tuned properly before injection of standard samples of all herbicides. Sulfosulfuron, ethoxysulfuron, meso + iodosulfuron (RM) were estimated by high performance liquid chromatography (HPLC) (Waters e-alliance 2695) having RP C-18 column (250 x 4.6 mm) and 5µ particle size. Acetonitrile: water (70:30 v/v) was used as mobile phase with an isocratic flow rate of 1 ml/min. Injection volume was maintained at 10 µl for each herbicide. Photodiode array detector (PDA, Waters 2998) was used at 254 µm for sulfosulfuron, 271 µm for ethoxysulfuron and 245 µm wavelengths for meso + iodosulfuron (RM) quantification. Retention time (Rt) of sulfosulfuron, ethoxysulfuron, mesosulfuron and iodosulfuron were 1.83, 1.79, 1.52 and 1.92 min, respectively. Glyphosate was analyzed on Shimadzu 2010 gas chromatograph (GC) equipped with capillary column, HP-I (30 m x 0.32 mm i.d. x 0.25 µm film thickness of film thickness of 5 per cent diphenyl and 95 percent dimethyl polysiloxane) and nitrogen phosphorous detector (NPD). Injection volume was 2 ml at split ratio of 1:5. The operating parameters of the instrument were: injection port was maintained at 270°C, column oven

temperature ramping was started from 70°C (2 min) → at 25°C/min → 150°C (0 min) → at 15°C/min → 200°C (0 min) → at 8°C/min → 280°C (2 min), carrier gas was N₂ at flow rate of 1.8 ml/min, H₂ at 1.5 ml/min and zero air at 130 ml/min. Detector temperature was 280°C. Under these operating conditions, the retention time of glyphosate was found to be 14.61 min.

Calibration details, linearity check and validation of method

Different known concentrations of respective herbicides were prepared by diluting the stock solution as mentioned above and injected into the instruments for measuring the peak area resulting after elution of compound. A calibration curve was plotted for concentration of the standard injected versus area observed and the curve was found linear up to the lowest range from 0.003 to 1.0 µg/ml. The method for estimation of selected herbicides residues in tomato crop and soil using HPLC and GC-NPD was validated by performing recovery experiments. A representative 25 g of meshed tomato fruits and 50 g of soil sample was taken in 250 ml Erlenmeyer flasks and fortified at 0.01 and 0.05 µg/g spiking levels with standard solution of the selected herbicides mentioned above. These flasks were kept undisturbed overnight. On next day, extraction, clean-up and analysis were done according to the procedures mentioned above.

RESULTS AND DISCUSSION

Bio-efficacy and phyto-toxicity of herbicides under field conditions

Both tomato hybrids Himsona and Rocky responded differentially to herbicide doses and time of applications. Hybrid Rocky was more sensitive to sulfosulfuron, ethoxysulfuron and meso + iodosulfuron (RM) irrespective of dose and time of application as seen by visual crop injury to tomato seedlings (**Table 1** and **2**). Glyphosate at all doses was found more injurious to Himsona than Rocky. Ready mix combination of meso + iodosulfuron (RM) caused extreme toxicity to both tomato hybrids resulting in complete death of seedlings at 60 and 90 DAT. Although sulfosulfuron at 25 g/ha (PPI or PE) and its application twice at 25 and 25 g/ha at 15 and 45 DAT, caused mild toxicity in the form of leaf yellowing to the range of 9-11% in Himsona and 21-34% in Rocky, but crop recovered fully up-to 60 DAT reflecting no adverse effect on number of fruits/plant and fruit yield of tomato. Post emergence use of glyphosate at 25 g/ha (4 weeks after transplantation, WAT) although caused slight chlorosis and bleaching

of leaves but crop recovered within 30 days of application with wrinkled leaves and lower number of tomato fruits and fruit weight in comparison to untreated check. Effect of herbicide treatments had significant effect on fruit yield of tomato. Maximum fruit yield 178.9 q/ha was obtained with use of sulfosulfuron (PE) at 25 g/ha which was significantly at par with sulfosulfuron 25 g/ha (PPI), untreated control and PoE application of sulfosulfuron at 25 g/ha at 15 and 45 DAT, respectively.

Bio-efficacy and phyto-toxicity of various herbicides under screen house studies

Metribuzin 250 g/ha was the safest treatment applied either PE or PoE (**Figure 1**). Even its higher dose (250 g/ha) or repeat applications of 125 and 250 g on 30 and 50 days caused minimum crop injury (<15%) when recorded 65 DAT. Application of sulfosulfuron 50 or 75 g/ha was more injurious when applied PE than PoE. Repeat applications of sulfosulfuron 50 followed by (*fb*) 50 g/ha or 75 g or 75 *fb* 75 g/ha at 30 and 50 days caused similar toxicity to that of single PE application and no significant variations in application rates were observed. Similar results were observed for ethoxysulfuron whereas, imazethapyr caused maximum crop damage. Application of glyphosate was less injurious than sulfosulfuron but affected the plant growth resulting in twisted leaves and affecting fruit size and weight.

Maximum tomato yield and fruit numbers were recorded with metribuzin 125 g/ha applied PE and non-sprayed plants followed by repeat application of ethoxysulfuron 75 *fb* 75 g/ha and metribuzin 125 g/ha applied at 30 DAT (**Figure 1**). Imazethapyr treated plants had no fruiting as there was complete plant mortality. Total fruit yield and fruit numbers were less with repeat applications of glyphosate compared to single application and also lower than other herbicides.

Herbicides residues study

The method for the estimation of selected herbicides residues in tomato crop and soil using HPLC or GC-NPD was validated by performing recovery experiments. Percent recoveries in all the samples of soil and tomato were found to be greater than 80%, so no recovery factor was needed for final calculations (**Table 3**). The harvest time residues status of different herbicides in tomato hybrids Himsona and Rocky and in soil under tomato crop has been presented in **Table 4**. It was observed that residues of sulfosulfuron in soil and both hybrids of tomato were below detectable level (BDL) at 25 g/ha

Table 1. Effect of different herbicide treatments on plant height and visual phyto-toxicity on tomato crop

Treatment	Crop phyto-toxicity (%)						Plant height (cms) 30 DAT	
	30 DAT		60 DAT		90 DAT		Himsona	Rocky
	Himsona	Rocky	Himsona	Rocky	Himsona	Rocky		
Sulfosulfuron (25 g/ha) PPI	19.4 (11.1)	29.7 (24.7)	0 (0)	0 (0)	0 (0)	0 (0)	21.2	20.6
Sulfosulfuron (25 g/ha) PE	17.8 (9.3)	27.7 (21.7)	0 (0)	0 (0)	0 (0)	0 (0)	23.0	21.3
Sulfosulfuron (25 and 25 g/ha) 15 and 45 DAT	17.9 (9.4)	28.2 (22.3)	0 (0)	0 (0)	0 (0)	0 (0)	21.8	21.1
Sulfosulfuron (50 and 50 g/ha) 15 and 45 DAT	18.4 (10.0)	28.8 (23.3)	0.0 (0.0)	22.6 (13.8)	18 (10.0)	25.3 (18.3)	21.5	20.0
Ethoxysulfuron (50 g/ha) PE	26.6 (20.0)	37.2 (36.7)	13.7 (8.3)	24.0 (17.5)	21.3 (13.3)	24 (16.7)	17.7	17.3
Ethoxysulfuron (50 and 50 g/ha) 15 and 30 DAT	29.9 (25.0)	33.2 (30.0)	21.3 (13.3)	26.6 (23.8)	15 (10.0)	33.1 (30.0)	20.1	18.5
Ethoxysulfuron (50 and 50 g/ha) PE and 45 DAT	35.2 (33.3)	52.7 (63.3)	39.2 (40.0)	59.0 (67.5)	46.9 (53.3)	55.1 (66.7)	17.3	17.0
Ethoxysulfuron (75 g/ha) PE	49.8 (58.3)	53.3 (64.3)	51.8 (61.7)	67.2 (76.3)	45 (50.0)	59.8 (73.3)	15.3	17.2
Ethoxysulfuron (75 and 75 g/ha) PE and 30 DAT	51.4 (61.1)	58.9 (73.3)	50.7 (60.0)	68.8 (77.5)	45.9 (51.7)	60.0 (73.3)	16.9	15.9
Meso+iodosulfuron (RM) (30 g/ha) PE	63.4 (80.0)	63.4 (80.0)	68.0 (85.0)	82.4 (83.8)	68.9 (86.7)	67.4 (78.3)	14.2	14.6
Meso+iodosulfuron (RM) (60 and 60 g/ha) PE and 45 DAT	65.8 (82.7)	67.4 (85.0)	90.0 (100)	90.0 (100)	90.0 (100)	90.0 (100)	12.1	14.0
Meso+iodosulfuron (RM) (90 and 90 g/ha) PE and 45 DAT	63.9 (80.0)	63.4 (80.0)	90.0 (100.0)	90.0 (100)	90.0 (100)	90.0 (100)	10.1	12.0
Glyphosate (25 g/ha) 4 WAT	0 (0)	0 (0)	6.1 (3.3)	0 (0)	0 (0)	0 (0)	19.4	20.8
Glyphosate (25 and 25 g/ha) 4 and 8 WAT	0 (0)	0 (0)	45.0 (50.0)	39.2 (40.0)	44 (48.3)	48.8 (56.7)	19.7	20.7
Glyphosate (50 g/ha) 4 WAT	25.9 (19.1)	26.6 (20.0)	40.1 (41.7)	39.2 (40.0)	49.3 (57.4)	49.8 (58.3)	19.7	20.2
Glyphosate (50 and 50 g/ha) 4 and 8 WAT	26.6 (20.0)	26.6 (20.0)	55.1 (66.7)	52.1 (61.5)	60.1 (75.0)	63.5 (80.0)	19.9	20.9
Glyphosate (25 and 50 g/ha) 4 and 8 WAT	18.4 (10.0)	18.4 (10.0)	50.8 (60.0)	54.8 (65.0)	49.8 (58.3)	50.8 (60.0)	19.9	21.2
Untreated check (control)	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)	22.4	22.2
LSD (p=0.05)	4.38	3.22	10.1	7.4	8.2	12.3	3.12	2.1

*PPI- Pre plant incorporation; DAT- Days after transplanting; PE- Pre-emergence; WAT: Weeks after transplantation
Original values are given in parenthesis which were transformed to arc sine transformation before analysis

Table 2. Effect of different herbicide treatments on number of leaves, number of fruits and fruit yield of tomato

Treatment	No. of leaves/plant (35 DAT)		No of fruits /plant (90 DAT)		Fruit field (t/ha)	
	Himsona	Rocky	Himsona	Rocky	Himsona	Rocky
	Sulfosulfuron (25 g/ha) PPI	6.9	6.9	24.4	28.1	17.10
Sulfosulfuron (25 g/ha) PE	6.8	6.6	25.0	28.7	17.89	14.26
Sulfosulfuron (25 and 25 g/ha) 15 and 45 DAT	7.1	6.8	25.1	26.5	16.11	14.11
Sulfosulfuron (50 and 50 g/ha) 15 and 45 DAT	6.5	6.4	22.5	25.5	12.62	13.42
Ethoxysulfuron (50 g/ha) PE	4.5	4.4	21.2	15.4	10.34	10.19
Ethoxysulfuron (50 and 50 g/ha) 15 and 30 DAT	6.5	6.2	21.7	20.4	12.34	6.53
Ethoxysulfuron (50 and 50 g/ha) PE and 45 DAT	5.6	5.2	13.5	8.4	9.97	3.82
Ethoxysulfuron (75 g/ha) PE	5.3	5.3	10.4	5.3	4.64	3.50
Ethoxysulfuron (75 and 75 g/ha) PE and 30 DAT	4.9	4.9	13.7	9.1	4.51	2.74
Meso+iodosulfuron (RM) (30 g/ha) PE	3.1	3.1	5.9	13.9	1.97	2.98
Meso+iodosulfuron (RM) (60 and 60 g/ha) PE and 45 DAT	2.4	2.5	0.0	0.0	0	0
Meso+iodosulfuron (RM) (90 and 90 g/ha) PE and 45 DAT	1.8	1.8	0.0	0.0	0	0
Glyphosate (25 g/ha) 4 WAT	7.9	7.5	15.2	16.5	11.8	10.29
Glyphosate (25 and 25 g/ha) 4 and 8 WAT	6.9	7.0	11.8	13.7	6.37	6.17
Glyphosate (50 g/ha) 4 WAT	6.5	6.2	13.2	12.3	5.26	5.09
Glyphosate (50 and 50 g/ha) 4 and 8 WAT	6.1	6.0	6.4	7.6	2.56	1.84
Glyphosate (25 and 50 g/ha) 4 and 8 WAT	7.1	7.1	16.6	10.9	5.28	6.05
Untreated check (control)	7.6	7.5	26.8	32.2	15.75	14.54
LSD (p=0.05)	1.3	1.0	4.7	4.8	1.24	0.81

either applied PPI and PE. Residues of sulfosulfuron were not observed in fruits of both tomato hybrids with early post-emergence application at 25 g/ha on 15 DAT followed by its sequential use at same rate on 45 DAT. But residues to the level of 0.025 to 0.045 µg/g were observed in soil treated with PoE applications of sulfosulfuron.

Ethoxysulfuron, when applied at 50 g/ha at all applications *i.e.* PE and PoE (15 and 30 DAT) did not show any residues in tomato fruits of both hybrids. But in soil, residues of ethoxysulfuron at same application rates varied from 0.028 to 0.039 µg/g. Ethoxysulfuron applications at 50 and 75 g/ha as PE and 45 DAT showed 0.019 and 0.011 µg/g residues in

Table 3. Percent recovery of different herbicides in tomato and soil

Herbicides	Calibration parameter						Average* recovery (%)			
	Linearity check		Limits of analysis				Tomato		Soil	
	Regression equation	R ²	Tomato		Soil		Fortification levels		Fortification levels	
			LOD (µg/g)	LOQ (µg/g)	LOD (µg/g)	LOQ (µg/g)	(0.01 µg/g)	(0.05 µg/g)	(0.01 µg/g)	(0.05 µg/g)
Sulfosulfuron	77320x+443.9	0.995	0.007	0.01	0.005	0.008	81.92	88.95	91.30	81.27
Ethoxysulfuron	49494x+117.1	0.999	0.006	0.009	0.006	0.01	86.66	84.31	89.42	83.62
Mesosulfuron	46382x+23.45	1.0	0.008	0.01	0.005	0.009	83.24	80.44	87.32	82.24
Iodosulfuron	10033x+359.2	0.997	0.008	0.01	0.008	0.01	82.45	87.85	84.15	81.58
Glyphosate	10745x+47.88	0.998	0.005	0.01	0.003	0.006	84.22	83.94	86.40	84.45

*Average of three replicates

Table 4. Residues of different herbicides in tomato fruits (*var. Himsona and Rocky*) and soil

Treatment	Residues* (µg/g)			
	Tomato		Soil	
	Himsona	Rocky	Himsona	Rocky
Sulfosulfuron (25 g/ha) PPI	BDL	BDL	BDL	BDL
Sulfosulfuron (25 g/ha) PE	BDL	BDL	BDL	BDL
Sulfosulfuron (25 and 25 g/ha) 15 and 45 DAT	BDL	BDL	0.025	0.032
Sulfosulfuron (50 and 50 g/ha) 15 and 45 DAT	BDL	BDL	0.068	0.045
Ethoxysulfuron (50 g/ha) PE	BDL	BDL	0.034	0.028
Ethoxysulfuron (50 and 50 g/ha) 15 and 30 DAT	BDL	BDL	0.029	0.039
Ethoxysulfuron (50 and 50 g/ha) PE and 45 DAT	0.019	0.011	0.107	0.113
Ethoxysulfuron (75 g/ha) PE	BDL	BDL	0.062	0.045
Ethoxysulfuron (75 and 75 g/ha) PE and 30 DAT	0.022	0.018	0.070	0.086
Meso+iodosulfuron (RM) (30 g/ha) PE	BDL	BDL	0.033	0.023
Meso+iodosulfuron (RM) (60 and 60 g/ha) PE and 45 DAT	-	-	0.152	0.178
Meso+iodosulfuron (RM) (90 and 90 g/ha) PE and 45 DAT	-	-	0.166	0.186
Glyphosate (25 g/ha) 4 WAT	BDL	BDL	BDL	BDL
Glyphosate (25 and 25 g/ha) 4 and 8 WAT	BDL	BDL	BDL	BDL
Glyphosate (50 g/ha) 4 WAT	BDL	BDL	BDL	BDL
Glyphosate (50 and 50 g/ha) 4 and 8 WAT	BDL	BDL	0.075	0.061
Glyphosate (25 and 50 g/ha) 4 and 8 WAT	BDL	BDL	0.062	0.068
Untreated check (control)	BDL	BDL	BDL	BDL

*Average of three replicates; BDL (Below Detectable Level)

tomato fruits of both hybrids respectively and soil under both hybrids of tomato showed residues as 0.107 and 0.113 µg/g. The residues of ethoxysulfuron at application of 75 g/ha as PE and 30 DAT were found less in comparison to the residues at application rate of 50 g/ha at PE and 45 DAT. Meso + iodosulfuron (RM) herbicide at application rate of 30 g/ha did not accumulate in tomato fruits but showed little residue build up (0.033 and 0.023 µg/g) in soil. Meso + iodosulfuron (RM) at application rate of 60 and 90 g/ha on PE and 45 DAT, respectively completely killed tomato crop and hence no tomato fruits for residues analysis were available. But, in soil the residues of meso + iodosulfuron (RM) varied from 0.152 to 186 µg/g. It can be inferred from the above study that meso + iodosulfuron (RM) dissipation was slightly less in soil under tomato crop when compared with sulfosulfuron and ethoxysulfuron.

Residues of glyphosate at application rate of 25 g/ha at 4 and 8 WAT were not found in fruits as well

as soil. Glyphosate, when applied at 25 and 50 g/ha at 4 and 8 WAT, respectively resulted in build up of residues in soil within range of 0.61 to 0.68 µg/g. This may be due to slow degradation of glyphosate in soil. Glyphosate have higher binding tendency to soil particles (Nomura and Hilton 1977, Reuppel *et al.* 1977, Newton 1984, Roy *et al.* 1989, Feng and Thompson 1990, Anton 1990) which prevents its leaching and runoff and hence greater persistence for longer duration. The glyphosate residues in tomato fruit of both varieties were found below detectable limit (BDL). It may be due to faster degradation of glyphosate in plant. The finding about fast degradation of glyphosate on foliage by Newton, 1984 further supported the results of present study in relation to prevention of glyphosate accumulation in fruits.

From the above study, it can be concluded that irrespective of dose and time of application, herbicides caused injury to tomato seedlings in both

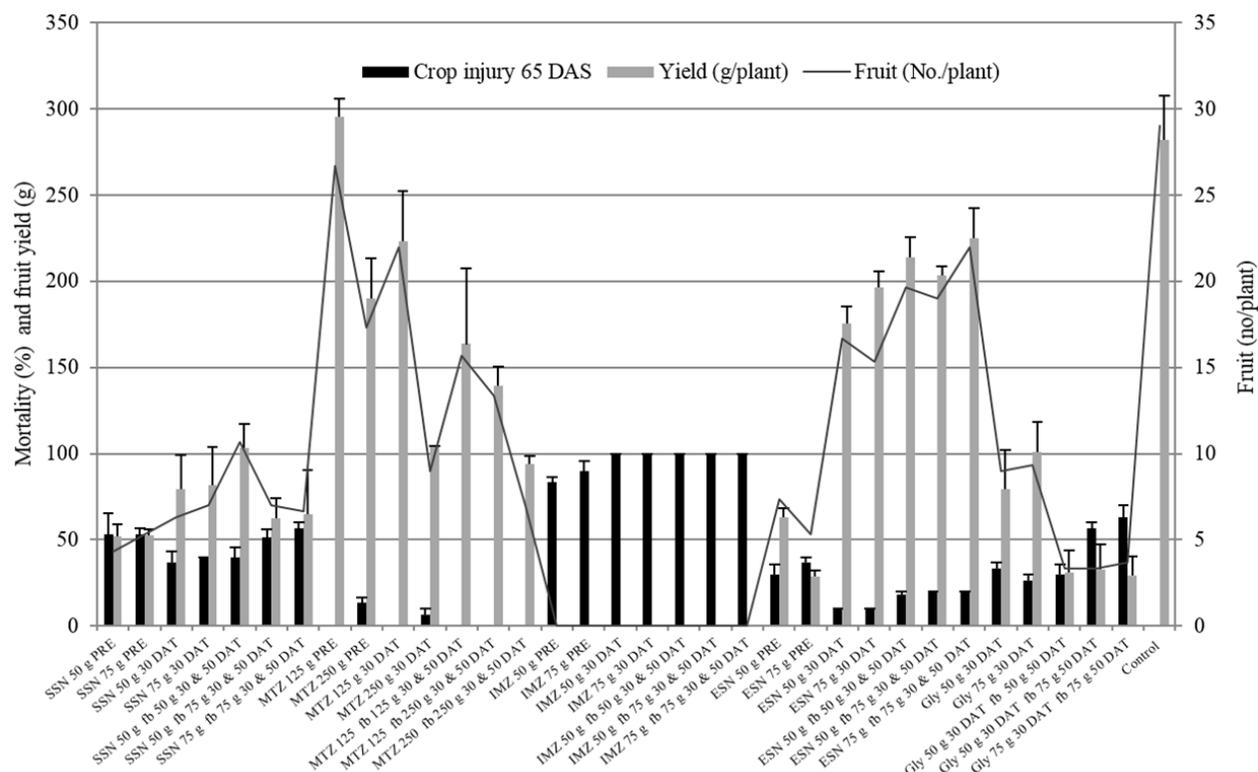


Figure 1. Effect of sulfosulfuron (SSN), metribuzin (MTZ), imazethapyr (IMZ), ethoxysulfuron (ESN) and glyphosate (Gly) on tomato mortality, fruit yield and fruit number per plant as observed in screen house study

Himsona and Rocky hybrids up to 30 DAT. Herbicide toxicity was more in Rocky as compared to Himsona irrespective of doses and time of applications. But crop recovered fully within 30 days in plots treated with sulfosulfuron at 25 g/ha either used as PPI or PE and its application at 25 g/ha twice on 15 and 45 DAT, respectively resulting in more number of fruits per plant and fruit yield was at par with untreated check. Sulfosulfuron when applied at 25 h/ha did not show any residues in tomato fruits and soil. In screen house study, ethoxysulfuron 75 fb 75 g/ha and metribuzin 125 g/ha applied 30 DAT were found safest treatment applied either PE or PoE. But, sulfosulfuron at 25 g/ha can be safely used in Himsona and Rocky hybrids of tomato for effective management of *Orobanche*.

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