Environ Monit Assess (2015) 187: 610
DOI 10.1007/s10661-015-4826-6

Behavior and bioefficacy of tribenuron-methyl in wheat (*Triticum astevum* L.) under irrigated agro-ecosystem in India

Irani Mukherjee · T.K. Das · A. Kumar · Bipasa Sarkar · K.K. Sharma

Received: 23 March 2015 / Accepted: 24 August 2015 / Published online: 4 September 2015

Abstract Possible bioaccumulation of pesticides in crop produce may cause ill effects on animals and humans. Tribenuron-methyl is a new post-emergence herbicide and is highly efficient to control the broad-leaf weeds in cereals, pasture, and plantation crops. There are scarce studies on its bioefficacy, sensitivity to weeds, tolerance to wheat, and persistence in crop produce, which are important information required before recommending an herbicide for use by the farmers. Weed control efficiency of an herbicide is dose-sensitive and site/soil-specific. Tribenuron-methyl (75 % DF) was applied at 22.5 and 45.0 g a.i./ha along with the surfactant 300.12 mL/ha as a tank mixes after 30 days of sowing in wheat as post-emergence herbicide. The samples of wheat foliage, soil, and grains at harvest were processed and analyzed for residues by high-performance liquid chromatography using a UV detector at 240 nm. The study revealed that there was a significant reduction in weed population and dry matter accumulation due to tribenuron-methyl application at a higher dose (45.0 g/ha) compared to a lower dose (of 22.5 g/ha). The weed density was found to be from 16.1 to 44.3 no/m^2 for application rate of 22.5 g/ha while at the 45.0 g/ha application, the weed density was 5.3–5.9 as compared to untreated control, where 184.3–120.5 no/m^2 was observed. The yield varied from 4.30 to 4.80 t/ha as compared to 2.25–3.55 t/ha in unweeded control with the LSD value being 21.5–16.3 to 0.27–0.19. Residues were below detectable level (BDL, <0.005 mg/kg) of tribenuron-methyl since they were detected in wheat grains at 22.5 g a.i./ha rates. However, 0.012 μg/g residues were detected in wheat foliage at an application rate of 45.0 g a.i./ha. It can be concluded that it is safe to use tribenuron-methyl at 22.5 g a.i./ha on wheat crop as post-emergence herbicide.

Keywords Foliage · HPLC analysis · Persistence · Soil · Tribenuron-methyl · Wheat

I. Mukherjee (✉) · B. Sarkar
Division of Agricultural Chemicals, Indian Agricultural Research Institute, New Delhi 110012, India
e-mail: mukrj_irani@yahoo.com

B. Sarkar
e-mail: bipasasarkar@yahoo.co.in

T. Das
Division of Agronomy, Indian Agricultural Research Institute, New Delhi -110012, India
e-mail: tkdas@iari.res.in

A. Kumar
Agricultural Knowledge Management Unit, Indian Agricultural Research Institute, New Delhi -110012, India
e-mail: akjha@iari.res.in

K. Sharma
Network Cordinating Cell AINP on pesticide Residues, Indian Agricultural Research Institute, New Delhi -110012, India
e-mail: kksaicrp@yahoo.co.in

Introduction

Crop protection agents (CPA) are the integral part in sustainable agriculture. The loss due to insect pests,

diseases, and weeds is about 10–15 % of the total agricultural produce globally. The timely use of inputs such as crop protection agents, high yielding varieties, and tolerant varieties of crops may reduce the loss of yield by about 15–30 %. Weeds pose the biggest threat to agriculture. Therefore, herbicides are now extensively used in Indian agriculture. Though, they are designed to be biologically active but are often found in soil, plant part, and finally in groundwater (Fletcher et al. 1993; Xu et al. 2002; Sondhia 2008). Tribenuron is a post-emergence herbicide (2-[4-methoxy-6-methyl-1,3,5-triazin-2-yl(methyl)-carbamoylsulfamoyl] benzoic acid). A variant methyl ester of tribenuron is used in the formulated product. Tribenuron and tribenuron-methyl, respectively, belong to the class of sulfonylurea herbicides. Tribenuron-methyl is used for the control of a wide range of broad-leaf weeds in cereal crops including wheat (winter, spring, and durum), barley (winter and spring), oats (winter and spring), rye, and triticale (Ferguson et al. 1985). It is also registered for use as a pre-emergence burndown broadcast application for wheat and barley. It is a systemic herbicide which is absorbed by the roots as well as leaves and then spreads throughout the whole plant. Among sulfonylurea herbicides, tribenuron-methyl {[methyl 2-(4-methoxy-6-methyl-1, 3, 5-triazine-2-yl) (methyl) carbamoylsulfamoyl] benzoate} (Fig. 1) is widely used due to its selectivity against a wide range of weeds in cereal, pasture, and plantation crops (Beyer et al. 1988; Brown 1990; Pons and Barriuso 1998). It inhibits acetolactate synthase (ALS) enzyme that catalyzes the first common reaction in the biosynthesis of branched amino acids namely valine, leucine, and isoleucine (Ray 1984; Brown et al. 1994; Vencill 2002). Animals have a different system of division of acetolactate synthase, and therefore, it is of low toxicity to mammals, birds, invertebrates, bees, or biological control agents such as parasitic wasps and predatory mites. The structure of tribenuron-methyl is characterized by a sulfonylurea bridge, an aryl group, and a nitrogen-containing heterocyclic portion. Under controlled conditions, tribenuron-methyl did not change any expression profile of gene immediately after treatment, but defense-related genes were upregulated after 1 week of its treatment. Sulfonylurea compounds such as tribenuron-methyl specifically inhibit acetolactate synthase and are quickly detoxified, but the activity of some of the resulting metabolites could explain later changes in gene expression in wheat (Pasquer et al. 2006) due to the formation of the metabolites after about 120 days (Anonymous 2004). Chemical hydrolysis and microbial degradation are the two major pathways of sulfonylurea degradation in soil, whereas photolysis (Bhattacherjee and Dureja 1999; Pusino et al. 1999; Vulliet et al. 2002) and volatilization are relatively minor processes (Molinari et al. 1999). Tribenuron-methyl binds to the soil matrix through a mixed interaction mode. Herbicide-soil interaction depends on the chemical structure of the herbicide along with the properties of the soil. In the harvested crop, possible bioaccumulation of pesticides may cause adverse effects on animals and human health. Therefore, there is an urgent need to evaluate this chemical in the crop during the time of harvest. Tribenuron-methyl is a new post-emergence herbicide and is highly efficient to control broad-leaf weeds in cereals, pasture, and plantation crops. Intensive literature survey indicates that there are scarce studies on its bioefficacy (sensitivity to weeds and tolerance to wheat) and persistence in crop produce, which are important information required before for its commercial use as an herbicide. Weed control efficiency of an herbicide is dose-sensitive and site/soil-specific (Das 2008).

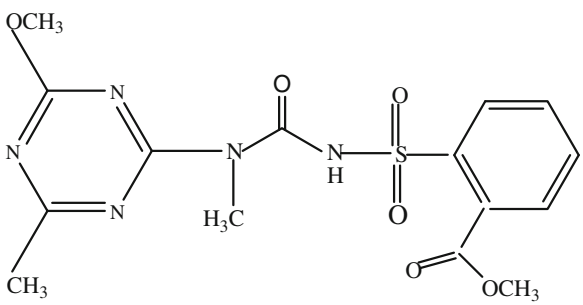

Fig. 1 Structure of tribenuron-methyl

In India, wheat is the second most important cereal crop in terms of consumption as well as production which is heavily infested with many broad-leaf weeds. Tribenuron proves to be very effective for controlling the broad-leaf weeds. Though, tribenuron-methyl is largely used for the control of pre- and post-emergence broad-leaf weeds of cereal crops. However, the presence of an extremely low concentration of tribenuron-methyl in soil causes greater phytotoxicity of several crops such as sugar beet, maize, cotton, sunflower, soybean, and rice (Brown 1990; Kotoula Syka et al. 1993; Junilla et al. 1994; Yao et al. 1997). However, little is known about the bioavailability of tribenuron-methyl in soil and crop produce. Hence, the present study was conducted to evaluate the persistence of tribenuron-methyl residues in wheat grains, straw, and soil at different time

intervals up to the harvesting. Also, the present study might help to recommend the suitable dose in terms of reducing the possible risk of contamination of the soil, crop produce, and phytotoxicity to wheat by tribenuron-methyl residues.

Material and methods

Reagents

Tribenuron-methyl (Analytical standard, purity 99.1 %) was purchased from Sigma Aldrich, USA. High-performance liquid chromatography (HPLC)-grade acetonitrile, HPLC-grade methanol, and water were taken from Merck, India. Laboratory-reagent-grade solvents like dichloromethane (DCM), acetone were glass-distilled at their respective boiling point and used. Magnesium sulfate anhydrous, sodium chloride, PSA, were purchased from Merck, Germany, Merck, India, and Agilent Technologies, USA, respectively. Herbicide Tribenuron-methyl (75 % DF) formulation was obtained gratis from DuPont India.

Preparation of stock solution

Tribenuron-methyl was accurately weighed (50 mg) and transferred into a volumetric flask (50 mL capacity) and dissolved in ~5 mL acetonitrile. The volume was made up to the mark with acetonitrile. A stock solution of 1000 μg/mL was obtained. Working standard solutions of lower concentrations were prepared from the stock solution by serial dilution. One milliliter of stock solution (1000 μg/mL) of tribenuron-methyl was taken in a 10-mL volumetric flask and diluted up to the mark with acetonitrile to get 100 μg/mL. This mixture was serially diluted to get standard solutions of lower concentrations.

Field experiment

Field experiments on the bioefficacy as well as soil were conducted consecutively for 3 years (2010–11, 2011–12, and 2012–13) and persistence of tribenuron-methyl in wheat in 2012–2013, at the Indian Agricultural Research Institute, New Delhi, India, during the winter (*rabi*) season (November to April) in a randomized block design with three replications each. Soil of the experimental field was alluvial (type ustochrepts; order inceptisol) in origin and sandy-loam (62.1 % sand, 16.5 % silt, and 19.8 % clay) with 0.54 % organic carbon and pH 7.6. The available phosphorous (18.5 kg/ha) and potassium (191.5 kg/ha) were medium, but available nitrogen (272.5 kg/ha) was low in soil. The size of each plot was 4 m × 1.6 m. Wheat variety, PBW 343, was sown in rows at 22.5 cm spacing between rows with a seed rate of 100 kg/ha. Tribenuron-methyl (75 % DF), tank-mixed with surfactant 300.12 mL/ha, was applied at two doses of 22.5 and 45.0 g active in gradient a.i./ha as post-emergence at 30 days after sowing (DAS) of wheat using a knapsack sprayer fitted with a flat fan nozzle. The volume rate of application was 400 L/ha water. Nitrogen, phosphorous, and potassium were applied to the wheat crop at 120 kg N, 60 kg P_2O_5, and 40 kg K_2O/ha in the form urea, single superphosphate, and muriate of potash, respectively. Half the dose of nitrogen and full dose of phosphorous and potassium were applied at the time of sowing, and the remaining nitrogen was top-dressed in two equal halves at 30 and 60 DAS. At 60 DAS, a quadrate of 0.5 m × 0.5 m area was randomly placed in each plot across replications. Weeds were counted, collected, and dried in a hot-air oven at 70 °C for 72 h for estimating dry weight. Some wheat plants were randomly sampled from each plot before harvest, and their straw and grains were separated. Harvested total produce of wheat from each plot was weighed, threshed, and cleaned and grain yield recorded.

Sampling of soil

Soil samples were drawn randomly from 0 to 15 cm depth using a tube auger from 6 to 7 spots in each plot. Around 500 g soil was collected from each plot. The samples were randomly drawn on initially 1 h, then 1, 3, 5,7,10, 15, 20, 30 days after treatment (DAT) and at the time of crop harvest (110 DAT) from all the treated and untreated control plots. Soil was mixed thoroughly, air-dried, grounded, and passed through 2 mm sieve. Around 50 g representative sample was taken by quartering for the final extraction before analysis.

Sampling of wheat foliage

The samples of wheat foliage were collected periodically from each treatment after spray on days 0, 1, 3, 5,7,10, 15, 20, and 30 and finally at the time of harvest, i.e., 110 days. The initial sample was collected after 3 h of herbicide application keeping in view the proper drying of spray on the leaves of plants.

Weather data

During the period of the study, average maximum and minimum temperatures were 26.3 and 12.49 °C, respectively. The average rainfall recorded during the period of the study was 16.33 mm and the % relative humidity was 61.22 with the average sunshine hours of 7.4 (Table 1).

Extraction and clean up

The wheat foliage and soil samples were extracted with acetonitrile, following the QuChERS method AOAC 2007.01 (AOAC Official Method 2007.01). The samples were homogenized to generate a uniform sample and then taken in a clean tube. After that, 10 ml acetonitrile was added along with 6 g magnesium sulfate and 1.5 g sodium chloride. The sample was centrifuged at 5000 rpm for 10 min. After which, 2 ml of the supernatant was taken, and 150 mg magnesium sulfate and 50 mg PSA were added and centrifuged at 5000 rpm for 10 min. Finally, 2 mL of supernatant was analyzed by HPLC and further by liquid chromatography with mass spectrometry-mass spectrometry (LC-MS-MS).

Analysis

The samples were analyzed by HPLC-UV detector at 240 nm using acetonitrile-water as the mobile phase.

Table 1 Weather data for the 3 years during the experimental period

Year	Nov	Dec	Jan	Feb	March	April
Rainfall (mm)						
2010–2011	0.0	8.6	40.8	109.4	12.6	11.6
2011–2012	0.0	0.0	14.8	0.0	19.2	9.0
2012–2013	13.4	0.3	0.0	49.9	2.3	2.2
Avg. rainfall/month	4.46	2.96	18.53	53.1	11.37	7.6
Avg. rainfall	16.33					
Maximum temperature(°C)						
2010–2011	25.9	20.2	20.7	23.9	33.0	37.1
2011–2012	26.1	22.1	19.3	24.0	30.2	35.0
2012–2013	26.3	19.5	20.2	23.1	30.1	37.2
Avg. max. temp/month	26.1	20.6	20.06	23.66	31.1	36.3
Avg. max. temp	26.3					
Minimum temperature(°C)						
2010–2011	18.2	7.3	8.2	11.1	13.6	20.9
2011–2012	10.2	8.2	7.9	12.3	16.3	20.6
2012–2013	11.9	8.3	6.3	11.8	12.8	19.3
Avg. min. temp/month	13.43	7.93	7.4	11.73	14.23	20.26
Avg. min. temp	12.49					
Relative humidity (%)						
2010–2011	78	74	62	73	50	34
2011–2012	68	70	71	51	40	38
2012–2013	60	80	82	80	58	33
Avg. relative humidity/month	68.66	74.66	71.66	68	49.33	35
Avg. relative humidity	61.22					
2010–2011	8.2	7.0	6.9	7.7	8.0	7.2
2011–2012	7.9	6.8	6.8	7.6	7.8	7.5
2012–2013	7.8	6.8	6.6	7.4	7.9	7.6
Avg. sunshine h/month	7.96	6.86	6.76	7.5	7.9	7.43
Avg. sunshine h	7.4					

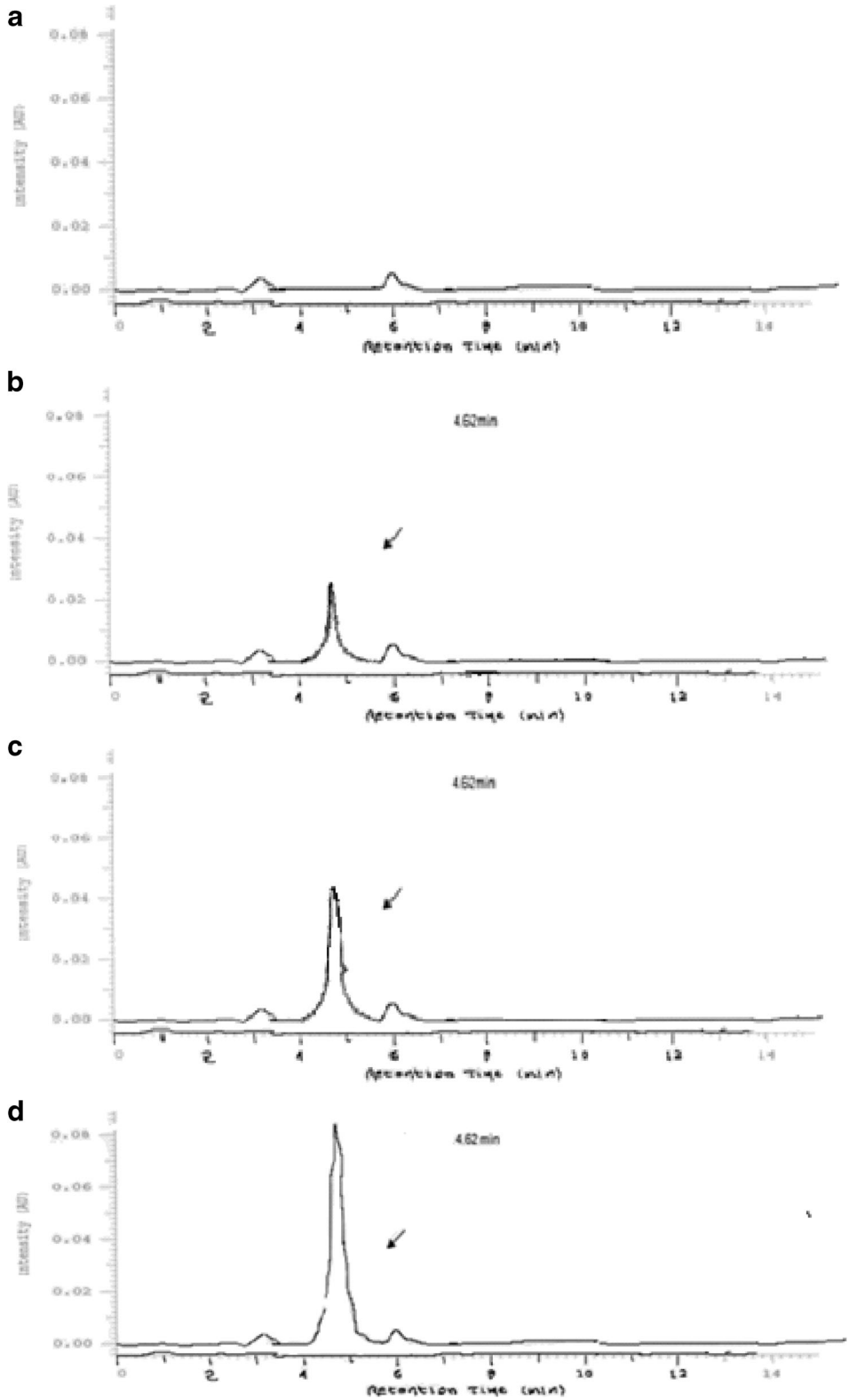

Fig. 2 HPLC profile of tribenuron-methyl, untreated control foliage (**a**), standard tribenuron-methyl 0.01 μL/mL (**b**), standard tribenuron-methyl 0.05 μL/mL (**c**), standard tribenuron-methyl 1.0 μL/mL (**d**)

Tribenuron-methyl was quantified by Merck High-Performance Liquid Chromatography (HPLC, Hitachi™) equipped with Lichrospher_RP-8 column (250 mm × 4.60 mm, 5 μm) and a UV detector set at 240 nm. HPLC-grade solvents (water and acetonitrile) were filtered through 0.22 μm filter and degassed before use. Mixture of acetonitrile and water (80:20 *v*/*v*) was used as a mobile phase with a flow rate of 0.5 mL/min. The injection volume was 10 μL. Under the standardized conditions, tribenuron-methyl was eluted at 4.62 min (Fig. 2). Calibration curve was drawn by injecting different concentrations of tribenuron-methyl to determine the linearity range.

The presence of tribenuron-methyl in field soil and foliage samples was confirmed by LC-MS-MS (Shimadzu LC-MS-MS–8030) (Fig. 3). The column Zorbax Eclipse plus C18 (3 mm × 100 mm, 3.5 μm), the mobile phase was programmed A, (80:20, water/methanol) with 5 mM ammonium formate; B, (90:10, methanol/water) with 5 mM ammonium formate, with a flow rate 0.2 ml/min and total run time 13 min. The organic mobile phase concentration was maintained at 45 % from 0.01 to 1 min and increased to 100 % till 12 min, and at 13 min, the mobile phase was bought to the initial concentration. The dwell time was 100, Q1 prebias 15 V, collision energy −18.0, and Q3 Prebias −19.0 V. The DL temperature was 250 °C and heat block was 400 °C. The drying gas was maintained at 15 L/min. The multiple reaction monitoring (MRM) for tribenuron-methyl was optimized at 396.0 > 155.0 under the above conditions. The analysis was carried out at an ambient oven temperature (Fig. 4).

Kinetic study and statistical analysis

To determine the degradation of herbicide residue on soil and wheat foliage, log concentration against time was plotted and then a linear regression analysis was performed of various data sets. The value of *K* (rate constant) along with corresponding half-life values were obtained. All the statistical analysis was done as per the protocol described by Sebai et al. (2010). Two-way analysis of variance was performed on the data sets using SAS 9.3 software and significant effects were noted.

Result and discussion

The effect of tribenuron-methyl on weed control in wheat was studied for 3 years for a valid recommendation. The reduction in weed populations and their dry weights vis-à-vis wheat grain yield across the treatments were recorded. Weed flora existed in wheat field were mostly broad-leaved ones, namely *Convolvulus arvensis*,*Rumex dentatus*,*Spergula arvensis*,*Coronopus*

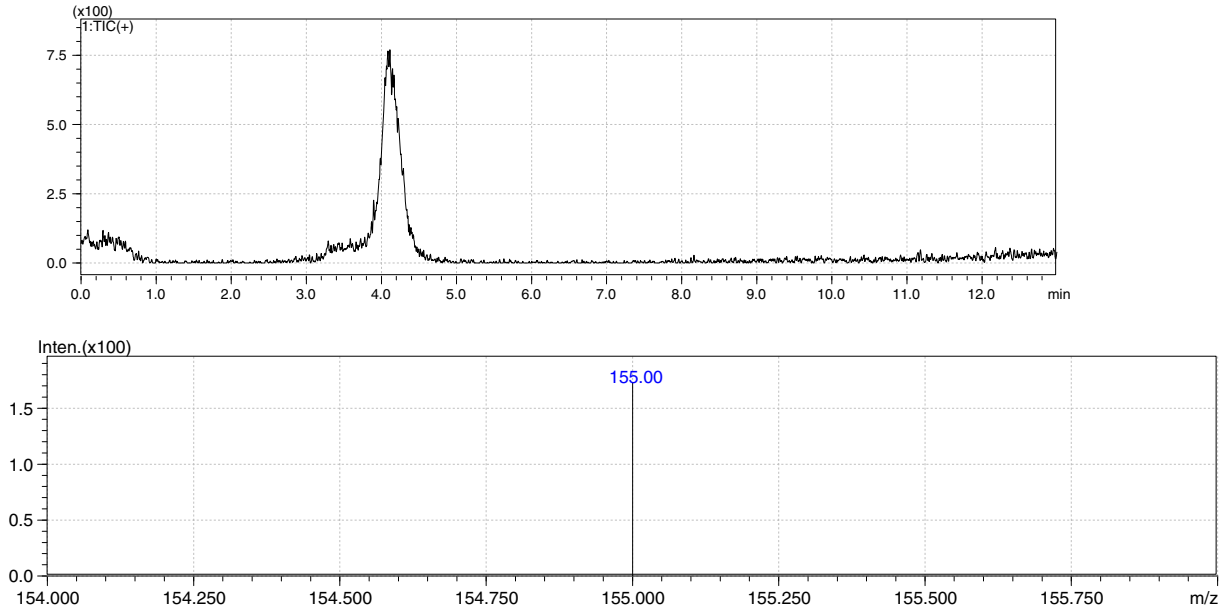

Fig. 3 LC-MS-MS TIC chromatogram and spectra of tribenuron-methyl

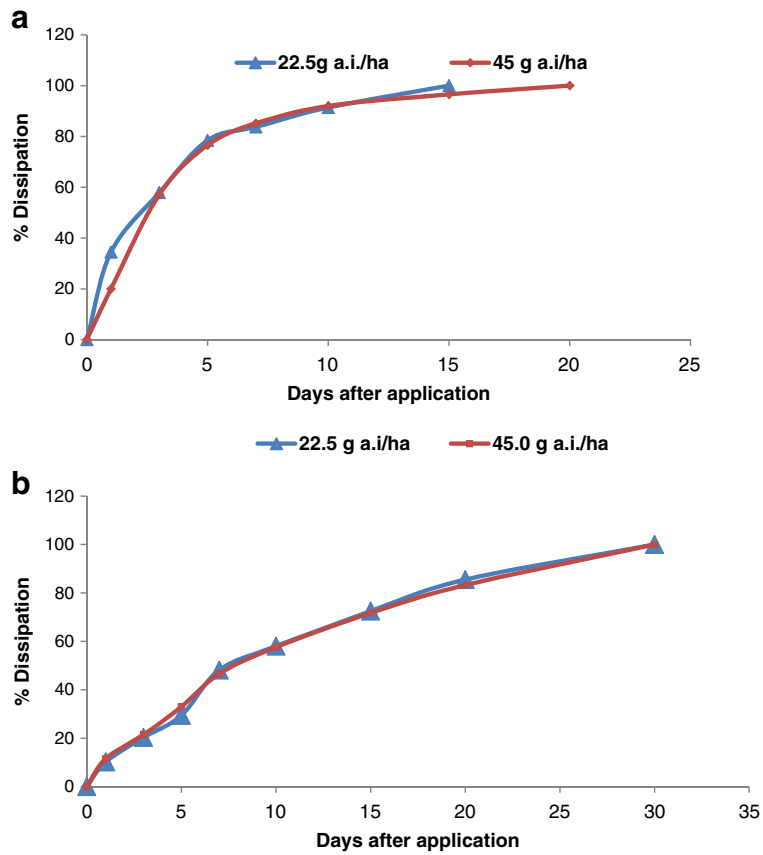

Fig. 4 Dissipation of tribenuron-methyl on wheat foliage (**a**) and in soil (**b**)

didymus,*Melilotus indica*,*Chenopodium album*,*Anagallis arvensis*,*Sisybrium irio*, and *Fumaria parviflora.* Besides, monocot grassy weeds, namely *Avena sterilis* ssp *ludoviciana* and *Phalaris minor* were also present in insignificant numbers.

This study (Table 2) revealed that weed density ranged from 16.1 to 49.3 plants/m^2 and 5.3 to 5.9 plants/m^2 at tribenuron-methyl dose of 22.5 and 45 g/ha, respectively, compared with 120.5 to 184.3 plants/m^2 in unweeded control over the years. Similarly, the yield varied from 4.30 to 4.80 t/ha and 4.07 to 4.52 t/ha at tribenuron-methyl doses of 22.5 and 45 g/ha compared with 2.55 to 3.55 t/ha in unweeded control. The sunshine, rainfall, and relative humidity play a significant role in the emergence of weeds, as compared to the treatment with the herbicide, recording maximum weed density in 2010–2011 (Table 2) where higher rainfall and % relative humidity was obtained (Table 1). There was a significant reduction in population and dry matter accumulation of weeds due to tribenuron-methyl treatment at both the doses, i.e., 22.5 and 45 g/ha, but the higher dose 45.0 g/ha was

Table 2 Effects of tribenuron-methyl on the density and dry weight of weeds and wheat grain yield over the years

Treatment	Weed density (no./m^2)			Weed dry weight (g/m^2)			Wheat grain yield (t/ha)		
	2010–11	2011–12	2012–13	2010–11	2011–12	2012–13	2010–11	2011–12	2012–13
Tribenuron-methyl at 22.5 g/ha	16.1	49.3	32.2	39.33	41.04	43.21	4.30	4.80	4.71
Tribenuron-methyl at 45.0 g/ha	5.3	5.7	5.9	11.67	12.51	13.36	4.07	4.43	4.52
Unweeded control	184.3	140.1	120.5	102.0	119.02	109.05	3.25	2.55	3.55
LSD (P = 0.05)	17.4	21.5	16.3	19.3	20.5	18.1	0.22	0.27	0.19

Table 3 Residues of tribenuron-methyl in wheat foliage and soil

Sampling days	Treatment (g a.i./ha)	Average residues (mg/kg) in wheat foliage	Average residues (mg/kg) in wheat soil
0	22.5	0.209 ± 0.98	0.069 ± 0.99
	45.0	0.35 ± 1.01	0.191 ± 0.79
1	22.5	0.137 ± 0.79	0.062 ± 1.05
	45.0	0.28 ± 0.88	0.169 ± 0.86
3	22.5	0.088 ± 0.81	0.055 ± 0.77
	45.0	0.15 ± 0.77	0.150 ± 0.87
5	22.5	0.045 ± 0.85	0.0487 ± 0.94
	45.0	0.082 ± 0.91	0.128 ± 0.85
7	22.5	0.034 ± 0.96	0.0358 ± 1.03
	45.0	0.052 ± 0.85	0.102 ± 0.93
10	22.5	0.018 ± 0.97	0.029 ± 0.86
	45.0	0.028 ± 1.05	0.081 ± 0.97
15	22.5	BDL	0.019 ± 0.86
	45.0	0.012 ± 0.92	0.054 ± 0.93
20	22.5	BDL	0.01 ± 0.86
	45.0	BDL	0.032 ± 0.91
30	22.5	BDL	BDL
	45.0	BDL	BDL
Harvest wheat grains	22.5	BDL	BDL
	45.0	BDL	BDL

BDL <0.01 mg/kg

superior to the lower dose of 22.5 g/ha on weed control. But the higher dose of tribenuron-methyl inflicted phytotoxicity to wheat crop, which resulted in lower grain yields of wheat by 0.19–0.37 t/ha over the years than the lower dose. Thus, tribenuron-methyl 22.5 g/ha was by far the best treatment in terms of weed control as well as wheat grain yield. It did not inflict phytotoxicity to wheat plants vis-à-vis gave better weed control. Similar results on persistence were recorded in metsulfuron methyl in wheat (Sondhia, 2008). Tribenuron-methyl controlled all broad-leaved present in wheat and could be a most promising broad-leaved killer herbicide of the sulfonylureas, inhibiting acetolactate synthase, which catalyzes the synthesis of three branched-chain amino acids, namely leucine, isoleucine, and valine (Das 2008).

Green foliage of wheat and soil samples was fortified at three concentrations of tribenuron-methyl at 0.01, 0.05, 0.5, and 1.0 μg/mL levels. The average recovery ranged from 68.9 to 91.9 and 73.6 to 95.7 %, respectively. The overall mean recoveries of tribenuron-methyl in wheat foliage were found to be 68.9 ± 1.62 %, 76.3 ± 0.98 %, 84.9 ± 0.81 %, and 91.9 ± 1.51 %, whereas in soil were found to be 73.6 ± 0.84 %, 78.4 ± 1.50 %, 88.4 ± 0.71 %, and 95.7 ± 0.56 % at 0.01, 0.05, 0.5, and 1.0 μg/mL fortification levels, respectively. The linearity of tribenuron-methyl was in the range of 2.5–0.005 μg/mL. The limit of detection (LOD) and limit of quantitation (LOQ) of tribenuron-methyl were 0.005 and 0.01 mg/kg, respectively. Tribenuron-methyl has been estimated using electron spray tandem spectroscopy (Degenhardt et al., 2010). The initial residues in wheat foliage was 0.209 mg/kg 0.350, respectively at 22.5 and 45.0 g a.i./ha application rate (Table 3). The residues dissipated to more than 78.4 % by day 5 (Fig. 4), indicating fast dissipation initially followed by slow dissipation till day 20 after the application of tribenuron-methyl. The residues were below detectable limit (BDL) (<0.01 mg/kg) by day 15 at the application rate of 22.5 g a.i./ha but were detected at a higher dose of application. Tribenuron-methyl dissipated with a half-life of 2.9–3.0 days in wheat foliage. In soil, the initial residues persisted till 20 days and were below detection limit day 30, recording a loss of 78.6 % (Fig. 4). A similar half-life was recorded as reported by Anonymous (2004). In the harvest, grain samples of tribenuron-methyl were below detectable limit (<0.01 mg/kg). No metabolites of tribenuron-methyl were detected during analysis by HPLC in the wheat and soil samples. Half-life values of tribenuron-methyl in soil calculated following first-order dissipation kinetics model were in the range 7.5–

Table 4 Regression equation and half-life of tribenuron-methyl in/on wheat foliage and soil under wheat field

Herbicide	Commodity	Treatment	$Y=$	DT^{50} (days)	R^2	K
Tribenuron-methyl	Soil	22.5 g a.i./ha	−0.040 × −1.143	7.52	0.990	0.0921
	Soil	45.0 g a.i./ha	−0.038 × −0.716	7.92	0.997	0.0875
	Wheat foliage	22.5 g a.i./ha	−0.104 × −0.739	2.89	0.984	0.2395
	Wheat foliage	45.0 g a.i./ha	−0.099 × −0.513	3.04	0.983	0.2279

7.9 days at both low and high doses, respectively (Table 4). The regression for residue time and algorithm of correlation of residue remained showed a good linear correlation based on R^2. A value of R^2 in different experiments was between 0.983 and 0.997. Although, no maximum residue limit (MRL) is set for tribenuron-methyl, but on the basis of risk assessment calculations based on acceptable daily intake (ADI) of tribenuron-methyl 0.02 mg/kg/day/body weight, a waiting period of 1 day can be suggested for wheat foliage and the harvest time grains are safe for consumption.

Conclusion

The study revealed that application of tribenuron-methyl at of 22.5 and 45 g/ha was significant in controlling the weed population in wheat as compared to unweeded control, enhanced yield in the crop, and the residues were below detectable limit in the wheat grains.

Acknowledgments The authors thank the Head of the Division for providing the facilities for the study.

References

AOAC Official Method 2007.01, Pesticide residues in foods by acetonitrile extraction and partitioning with magnesium sulfate.

Anonymous (2004). EFSA Scientific report peer review of the pesticide risk assessment of tribenuron) 15, 1–52.

Beyer, E. M., Duffy, M. F., Hay, J. V., & Schlueter, D. D. (1988). Sulfonylureas. In P. C. Kearney, & D. D. Kaufman (Eds.), *Herbicides: chemistry, degradation, mode of action* (vol. 3, pp. 117–189). New York: Dekker.

Bhattacherjee, A. K., & Dureja, P. (1999). Light-induced transformations of tribenuron-methyl in aqueous solution. *Pesticide Science*,*55*, 183–188.

Brown, H. M., & Cotterman, J. C. (1994). Advances in sulfonylurea herbicides. In J. Stetter, & W. Eding (Eds.), *Chemistry of plant protection—herbicides inhibiting branched chain amino acid biosynthesis* (pp. 47–81). Berlin: Springer.

Brown, H. M. (1990). Mode of action, crop selectivity, and soil relations of sulfonylurea herbicides. *Pesticide Science*,*29*, 263–281.

Das, T. K. (2008). *Weed science basics and applications* (pp. 1–901). New Delhi:Jain Brothers.

Degenhardt, D., Cessna, A. J., Raina, R., Pennock, D. J., & Farenhorst, A. (2010). Trace level determination of selected sulfonylurea herbicides in wetland sediment by liquid chromatography electrospray tandem mass spectrometry. *Journal of Environmental Science and Health Part B*,*45*, 11–24.

Ferguson, D. T., Schehl, S. E., Hageman, L. H., Lepone, G. E., & Carraro, G. A., (1985). DPXL5300—a new cereal herbicide. In: Proceedings of the British Crop Protection Conference-Weeds, 1, pp 43–48.

Fletcher, J. S., Pfleeger, T. G., & Ratsch, H. C. (1993). Potential environmental risks associated with the new sulfonylurea herbicides. *Environmental Science & Technology*,*27*, 2250–2252.

Junilla, S., Heinonorean-Tanski, H., Revio, L. R., & Laitinen, P. (1994). Phytotoxic persistence and microbiological effect of chlorsulfuron and metsulfuron in finish soil. *Weed Research*, *34*, 413–423.

Kotoula Skya, E., Elftherohorinos, I. G., & Gagianas, A. A. (1993). Phytotoxicity and persistence of chlorsulfuron, metsulfuron in three soils. *Weed Research*,*33*, 355–367.

Molinari, G. P., Cavannam, S., Bonifacinim, F., Giammarustim, L., & Barefootm, A. C. (1999). Bensulfuron-methyl and metsulfuron-methyl dissipation in water and soil of rice fields. In A. M. Del Re, C. Brown, G. Errera, & S. P. Evans (Eds.), *Proceedings of XI symposium pesticide chemistry, human and environmental exposure to xenobiotics, Cremona, Italy* (pp. 45–50). Edizioni La Goliardica-Pavese: Pavia.

Pasquer, F., Ochsner, U., Zarn, J., & Keller, B. (2006). Common and distinct gene expression patterns induced by the herbicides 2,4-dichlorophenoxyacetic acid, cinidon-ethyl and tribenuron-methyl in wheat. *Pest Management Science*,*62*, 1155–1167.

Pons, N., & Barriuso, E. (1998). Fate of metsulfuron-methyl in soils in relation to pedo-climatic conditions. *Pesticide Science*,*53*, 311–323.

Pusino, A., Braschi, I., Petretto, S., & Gessa, C. (1999). Photodegradation of herbicide triasulfuron. *Pesticide Science*,*55*, 479–481.

Ray, T. B. (1984). Site of action of chlorsulfuron: inhibition of valine and isoleucine biosynthesis in plants. *Plant Physiology*,*75*, 827–831.

Sebai, T. E., Devers, M., Lagacherie, B., Rouard, N., Soulas, G., & Martin-Laurent, F. (2010). Diuron mineralisation in a Mediterranean vineyard soil: impact of moisture content and temperature. *Pest Management Science*,*66*, 988–995.

Sondhia, S. (2008). Imazosulfuron residues in rice crop and soil. *Environmental Monitoring and Assessment*,*137*, 205–211.

Vencill, W. K. (2002). *Herbicide handbook* (eighth ed., p. 493). Lawrence:Weed Science Society of America.

Vulliet, E., Emmelin, C., Grenier-Loustallot, M. F., Païssé, O., & Chovelon, J. M. (2002). Simulated sunlight-induced photodegradations of triasulfuron and cinosulfuron in aqueous solutions. *Journal of Agricultural and Food Chemistry*, *50*, 1081–1088.

Xu, J., Wang, H., & Xie, Z., (2002). Dynamics of extractable and bound residues of ^{14}C metsulfuron-methyl in soils. 17 WCCS, Thailand, pp. 1674–1677.

Yao, D. R., Song, X. L., & Chen, J. (1997). Studies on sensitivity of after crop to metsulfuron residues in wheat field. *Jiangsu Journal of Agricultural Science*,*13*, 171–175.