

Trifluralin Residue in Root Vegetables

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Abstract

A field experiment was conducted during *rabi* of 2007-08 to evaluate trifluralin residues in carrot (*Daucas carota* L.) and radish (*Raphanus sativus* L.). Trifluralin at 0.6, 0.9 and 1.2 kg/ha, was applied as pre-emergent herbicide in both crops. At harvest, the crop roots were analyzed for herbicide residues by gas chromatography/mass spectrometry (GC/MS). Calibration curve for trifluralin was linear in the range between 0.02—5 ppm. The average recovery percentage for standard solutions was found to be 75%. Trifluralin residue in roots of both vegetables was below the detectable limit (0.02 ppm). It was concluded that the use of trifluralin as weedicide in carrot and radish did not pose any health hazard.

Key words : Carrot, Radish, Residue, Trifluralin.

Carrot (*Daucas carota* L. var *sativus* Hoffm.) and radish (*Raphanus sativus* L.) are important winter season root vegetables. Roots of these crops are eaten raw as salad or cooked as vegetable. Uncontrolled weed growth causes substantial yield loss of 76% in carrot (1) and 86% in radish (2). The use of herbicides is fundamental in the cultivation of vegetable crops. However, research recommendations on chemical weed control are considered incomplete if data on toxic residue of herbicide are not provided. The applied herbicides persist in soil and thus may leave residues in plants. Vegetables, owing to their short life span, are more prone to pesticide residue problem in crop roots particularly if consumed raw. When root crops such as carrot, onion and turnips were grown in soils treated with trifluralin, the herbicide residues were found to be present on their surface (3—5). Keeping in view the potential health hazards of agrochemicals, it is of utmost importance to assess herbicide residues in crops. Trifluralin (α, α, α , trifluoro-2,6,dinitro-*N, N*,-dipropyl-*p*-toluidine) is prominent among dinitroaniline herbicide family, applied pre-plant or pre-emergence for selective weed control in number of crops viz. cotton, soybean, groundnut, peas, tomato, cucurbits, cole crops and carrot (6). It inhibits cell division and elongation in both root and shoot meristems. Tolerance limits for trifluralin in carrot is reported to be 1.0 ppm (7). Very little work has been done on the residues and dissipation mode of trifluralin under Indian conditions.

The present study aimed to evaluate the residues of trifluralin in carrot and radish roots.

Methods

A field experiment was carried out during *rabi* 2007-08 at Ludhiana. The experimental site was loamy sand, normal with respect to soil pH (6.8), low in organic carbon (0.24%) and available nitrogen (180 kg/ha), medium in available phosphorus (13.4 kg/ha) and available potassium (253 kg/ha). Trifluralin at 0.6 kg, 0.9 kg and 1.2 kg/ha, was applied one day after sowing as pre-emergence, for weed control in separate trials for carrot and radish, each having a net plot size of 9.63 m². Thereafter agronomic practices as per recommendation were carried out. At harvest (about 60 days and 90 days after sowing for radish and carrot, respectively), root samples from both the crops were analyzed for trifluralin residue by gas chromatography mass spectrometry (GC/MS) using the method (8) described below.

Sample Preparation and Extraction. Ten gram of the root sample was crushed in pestle mortar and extracted with 20 ml mixture of hexane and ethyl ether (1 : 1) in a reagent bottle. The suspension was thoroughly mixed over a mechanical shaker for 30 minutes. The solution was filtered through Celite 545 bed, washed with mixture of hexane/ethyl ether to restore the volume of filtrate to 50 ml. Finally, the sample was concentrated to dryness by rotary evaporation.

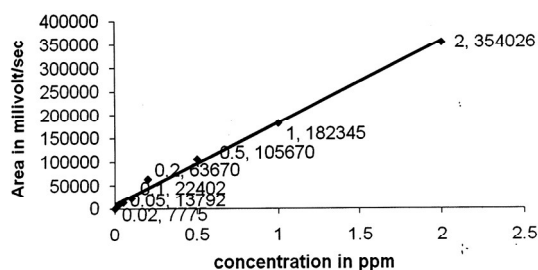


Figure 1. Calibration curve for trifluralin (Minimum detectable concentration : 0.02 ppm).

Clean-Up. Sep Pak florasil cartridge was conditioned with 10 ml of hexane : ethyl ether (3 : 7) containing 10 g florasil. The sample was added to cartridge and eluted with 24 ml hexane : ethyl ether (99 : 1) for total added volume of 25 ml at rate 1.5 ml/min and collected in 50 ml flask.

Determination. Dissolved the precipitates in 2 ml hexane (HPLC grade), filtered and analyzed through GC/MS.

GC/MS chromatographic separation parameters.

Instrument	Hewlett Packed (USA) Model 5890 A gas chromatograph coupled with 3392 A injector
Ion source	Ni ⁶³
Carrier gas	Helium
Flow rate	2.8 ml/min
Detector temperature	250 C
Volume injected	2 µl using fixed loop Rheodyne injector
Approximate retention time for standard	19.37 minutes

Validation. Validation of the method was performed in terms of recovery studies before analyzing the unknown samples. Root samples from control were fortified with known concentrations (0.5, 1.0 and 2.0 ppm) of the standard solution of trifluralin.

The residues were determined by using the formula given below :

$$\text{Residue concentration } (\mu\text{g/g}) = \frac{A_1}{A_2} \times \frac{V}{W} \times C$$

Table 1. Residues of trifluralin in carrot and radish roots at harvest. Minimum detectable concentration : 0.02 ppm.

Treatments (kg/ha)	Residue in carrot roots (ppm)	Residue in radish roots (ppm)
Trifluralin 0.60	<0.02	<0.02
Trifluralin 0.90	<0.02	<0.02
Trifluralin 1.20	<0.02	<0.02

Where, A_1 Peak area of sample (μ V-sec), A_2 Peak area of standard (μ V-sec), V Volume of sample extracts (ml), W Weight of the sample (g), C Concentration of sulfosulfuron (ppm).

Results and Discussion

The calibration curve for trifluralin was prepared by injecting different known concentrations 0.02, 0.05, 0.1, 0.2, 0.5, 1.0 and 2.0 ppm of the compound. The stock solution (1000 ppm) was obtained in pure HPLC grade acetonitrile. The calibration curve (Fig. 1) was plotted for the concentration of the standards injected versus the peak area observed. The curve was found to be linear up to the lowest concentration range of 0.02 ppm. The approximate retention time for trifluralin was obtained at 19.37 minutes.

The average recovery percentage was found to be 75% for trifluralin. The details of sample analysis and the residues under all the treatments are presented in Table 1. Radish and carrot roots upon analysis on GC/MS for residue detection showed that for none of the trifluralin levels, the peak was obtained at the respective retention time and each sample (treatment) recorded below detectable limit of herbicide (Table 1). The same can be attributed to degradation and leaching down of the herbicide due to numerous irrigations ; precipitation and a significant time gap between herbicide application and sampling for analysis. Carrot pulp at harvest had less than 0.03 mg/kg trifluralin residues (9). The results are also in conformity with (10) which reported no harvest time residues of trifluralin in carrot roots from fields treated with 0.56-2.2 kg/ha (limit of detection 0.2 parts per billion). It was concluded that trifluralin at the doses tested is safe for use in carrot and radish.

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