

Analysis of Herbicide Residues in Onion Bulbs and Soil under Different Planting Patterns and Straw Management Techniques

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ABSTRACT

An experiment was conducted at PAU, Ludhiana, during the **rabi** seasons of 2006-07 and 2007-08 to study the effect of different planting patterns and straw management techniques on residues of different herbicides in onion bulbs and soil. The experiment was laid out in split plot design with planting patterns of onion (flat and bed planting with and without rice straw incorporation) in main plots and weed control treatments in sub-plots. Oxyflourfen and pendimethalin were applied one week after sowing (after first irrigation) and fluchloralin was applied as pre-plant at their respective doses as per given herbicidal treatments. Soil samples were collected from the experimental plots at 1, 30 and 60 days after spray and at uprooting from each plot from 0-15 and 15-30 cm depth and samples of onion bulbs were taken at 30, 60, and 90 days after spray and at uprooting. Soil samples of oxyflourfen 0.225 kg/ha, pendimethalin 0.75 kg/ha and fluchloralin 1.125 kg/ha treated plots taken 1 day after spray showed residues of respective herbicides. Samples of onion bulbs at 30, 60 and 90 days after spray and at uprooting stage showed no residues of applied herbicides in onion bulbs.

Key words : Herbicides, onion, planting patterns, residues

INTRODUCTION

Onion (*Allium cepa* L.) is one of the most important vegetable crops in India. Our country accounts for one third of the world production in onion due to intensive cultivation. In India, it occupies an area of 0.53 m ha with production of 6.66 m tonnes during 2006-07 and 0.48 m ha of area with production of 7.45 m tonnes during 2007-08 (Anonymous, 2008a). In Punjab, it covered an area of 7.99 thousand hectares with a production of 17.17 m tonnes during 2007-08 (Anonymous, 2008b). Severe weed problems in onion and huge losses due to weed competition are a global problem. Weeds interfere with the development of onion bulb by competing with moisture, nutrient, light and space thereby reducing bulb yield to the extent of 40-80% (Verma and Singh, 1996). Owing to its inherent characteristics such as short stature, non-branching habit, sparse foliage, shallow root system and extremely slow growth in initial stages, weeds offer severe competition throughout the crop growth. The conventional method of weed control i. e. hand weeding is, no doubt, effective but it is time consuming, cumbersome and under many situations becomes uneconomical. Pendimethalin, oxyflourfen and

fluchloralin are few selective herbicides recommended for controlling weeds in onion. The applied herbicides persist in soil and may leave residues in onion bulbs as these are consumed as raw at any time and hence information on the herbicide residues in onion bulb is very important. The problem of herbicide residues is more acute in vegetable crops as compared to field crops because firstly vegetables are consumed as raw and secondly time gap between application of herbicide and harvest of vegetable crops is very less. So, keeping in view the health hazardous effect of these agrochemicals, it is considered important to study the herbicide residue in vegetable crops like onion.

MATERIALS AND METHODS

A field experiment was carried out during **rabi** seasons of 2006-07 and 2007-08 at Research Farm, Department of Agronomy, Punjab Agricultural University, Ludhiana. The experimental site was loamy sand in texture. The experiment consisting of four planting patterns viz., flat sowing (with and without rice straw incorporation) and bed sowing (with and without rice straw incorporation) in main plots and five weed control treatments viz., pendimethalin 0.75 kg/ha, oxyflourfen

0.225 kg/ha, fluchloralin 1.125 kg/ha, two hand weedings 3 & 6 WAS and control (unweeded) in sub-plots. The experiment was laid out in split plot design with four replications. Oxyflourfen and pendimethalin were applied one week after sowing (after first irrigation) and fluchloralin was applied as pre-plant incorporation at their respective doses as per given herbicidal treatments. For the estimation of residues of herbicides, soil samples were taken from 0-15 and 15-30 cm depth at 1, 30 and 60 days after herbicide application. Samples of onion bulbs were uprooted at 30, 60 and 90 days after spray.

Method of Extraction

1. Pendimethalin

Ten gram of ground sample of soil and chopped onion was weighed into a stoppered conical flask and extracted with 20 ml acidified methanol and 20 ml methanol, respectively, over a mechanical shaker for 30 min. The solution was filtered through Whatman's No. 42 filter paper and washed it with respective solutions. The sample was concentrated by rotary evaporation.

Partitioning : Twenty ml of distilled water (DW) was added to concentrated extract and 4 ml 0.1 N HCl was added to this and transferred the whole solution to separatory funnel. Twenty-five ml n-hexane was added and shaken vigorously. Lower layer of water was removed in the funnel after partitioning. The remaining solution (organic phase) was filtered through cotton and collected over anhydrous Na_2SO_4 . The procedure was repeated to collect the organic phase for two more times. The hexane was evaporated to dryness on a rotary vacuum evaporator.

Clean up : The dried extract was pre-dissolved in n-hexane and subjected to column chromatography over Florisil column containing 10 g Florisil (Merck 60-100 mesh ASTM) topped with 1 cm anhydrous Na_2SO_4 and pre-washed with 50 ml n-hexane. The elute was evaporated to dryness. The precipitates were dissolved in 3 ml hexane (HPLC grade), filtered and analyzed by GC/MS using method developed by Tsiropoulos and Miliadis (1998) and Sankaran *et al.* (1993), respectively.

2. Fluchloralin

Fifty gram of onion bulbs sample was extracted

twice with 250 ml of acetone for 5 min in a blender. Each extract was filtered through filter paper placed on a Buchner funnel. The acetone from the combined filtrate was evaporated using 45°C water bath temperature and water aspirator vacuum. Any remaining oily residue was extracted twice with 50 ml hexane and 50 ml acetonitrile was added and shaken vigorously. After separation of the phases, the hexane was discarded. The acetonitrile phase was washed once more with 10 ml hexane. The acetonitrile extract was brought to dryness.

Fifty gram of the soil was extracted with 150 ml of benzene : water (2 : 1) for 30 min on a shaker, centrifuged and 80 ml benzene was removed. The benzene extract was brought to dryness.

Clean up : The residues was taken up in 5 ml of benzene and applied to a 20 g florisil column. The column was eluted with 100 ml benzene. The elute was brought to dryness in a rotary evaporator and the residues were dissolved in benzene. The residues from onion bulbs were dissolved in 5 ml and soil 4 ml of benzene. The precipitates were dissolved in 2 ml hexane (HPLC grade), filter and analyze through GC/MS using method developed by Tsiropoulos and Miliadis (1998) and Sankaran *et al.* (1993), respectively.

3. Oxyflourfen

Ten gram of ground sample of soil and chopped onion was weighed into a stoppered conical flask and extracted with 20 ml acetone over a mechanical shaker for 30 min. The solution was filtered through sodium sulphate and charcoal and washed it with respective solutions and later on concentrated to a smaller volume using vacuum rotary evaporator. The sample was dissolved in mobile phase and then analysed by High Performance Liquid Chromatography method developed by Riley *et al.* (1994).

Chromatographic Separation Parameters

Oxyflourfen was analysed by High Performance Liquid Chromatography (HPLC).

Method of Calculation

During the process of routine sample analysis, wherever necessary, the concentration of sample solution was adjusted and injected. To assure the integrity of the

HPLC Chromatograph Parameters

Instrument	Waters High Performance Liquid Chromatograph Model Water 2487 Dual.
Detector	UV-VIS detector
Column	Phenomnax C ₁₈ (25 cm length × 4.6 mm i. d.)
Detector sensitivity	0.01 A. U. F. S.
Volume injected	10 µl using fixed loop Rheodyne injector
Flow rate (ml/min)	1.00
Mobile phase (Acetonitrile : water)	60 : 40
Wave length (nm)	206

samples, analytical standards were injected after each seven sample injections. The residues were determined by using the formula given below :

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

Where,

- A₁=Peak area of sample (µV-sec)
- A₂=Peak area of standard (µV-sec)
- V=Volume of sample extracts (ml)
- W=Weight of the sample (g)
- C=Concentration of herbicide (ppm)

RESULTS AND DISCUSSION

The residues of the herbicides under flat planting (rice straw incorporation), flat planting (no rice straw incorporation), bed planting (rice straw incorporation), bed planting (no rice straw incorporation), respectively, and at different soil depths (0-15 and 15-30 cm) are presented in Tables 1 and 2. Residual concentrations of herbicides viz., pendimethalin, oxyflourfen and fluchloralin in soil were determined at 1, 30 and 60 days after spray (DAS) and uprooting in onion bulbs was

determined at 30, 60 and 90 days after spray and at uprooting during both the years (2006-07 and 2007-08).

One day after spray, the concentration of pendimethalin, oxyflourfen and fluchloralin in the 0-15 cm soil depth was reduced to less than 0.025, 0.01 and 0.05 ppm in case of pendimethalin, oxyflourfen and fluchloralin, respectively, which were the minimum detectable limits. The residue of the herbicides at 30 and 60 days after sowing and at uprooting under all the main plot treatments and at the soil depths (0-15 and 15-30 cm) was found to be below 0.025, 0.01 and 0.05 ppm in case of pendimethalin, oxyflourfen and fluchloralin, respectively, which were the minimum detectable limit. Randhawa *et al.* (2007) reported that no residue of oxyflourfen was detected at 15 and 30 days after spray and at harvest in onion and garlic bulbs.

Analysis of plant samples was also carried out to estimate the amount of herbicides translocated to the plant system from soil. Upon analysis of samples of onion bulbs at 30, 60, 90 days after spray and at uprooting, no residue of pendimethalin, oxyflourfen and fluchloralin was detected during both the years of trial (Table 3) as in none of the samples the peak was obtained at the respective retention time. Jayakumar and Bharti (2003) also concluded that application of fluchloralin

Table 1. Residue of herbicides (ppm) in soil at 0-15 cm depth under different planting patterns at 0 DAS in onion crop

Planting pattern	0 DAS (at 0-15 cm)					
	Pendimethalin		Oxyflourfen		Fluchloralin	
	2006-07	2007-08	2006-07	2007-08	2006-07	2007-08
Flat planting (rice straw incorporation)	0.03	0.04	0.02	0.03	0.05	0.06
Flat planting (no straw incorporation)	0.03	0.04	0.02	0.04	0.06	0.06
Bed planting (rice straw incorporation)	0.04	0.03	0.02	0.03	0.06	0.06
Bed planting (no straw incorporation)	0.035	0.03	0.02	0.03	0.05	0.06

Table 2. Residue of herbicides (ppm) in soil at different depths and in onion bulbs under different planting patterns at different intervals during 2006-07 and 2007-08

Planting pattern	In soil						In onion bulbs																		
	Days after spray						Days after spray																		
	30		60		At harvest		30		60		90		At harvest												
(0-15 & 15-30 cm)						(0-15 & 15-30 cm) (0-15 & 15-30 cm)						Herbicides													
Herbicides						Herbicides						Herbicides													
Pendi Oxy Flu						Pendi Oxy Flu						Pendi Oxy Flu													
Flat planting (rice straw incorporation)	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Flat planting (no straw incorporation)	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Bed planting (rice straw incorporation)	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Bed planting (no straw incorporation)	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Flat planting (rice straw incorporation)	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Flat planting (rice straw incorporation)	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL

BDL : Below detectable limit.
 Minimum detectable limit for Pendimethalin < 0.025 ppm, oxyflourfen < 0.01 ppm and fluchloralin < 0.05 ppm.

and pendimethalin in onion could be considered safe from the point of view of consumption of green and mature onion bulbs as the residues of these herbicides were below maximum residual limits. Devi *et al.* (1998) reported that oxyflourfen lost its activity by 60-80 days after spray in red and black soils.

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