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Residues of pendimethalin and oxyfluorfen in radish and their persistence in soil

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ABSTRACT

Field experiment was conducted to study the persistence of pendimethalin and oxyfluorfen in soil and its residues in edible parts of raddish. At harvest in both the seasons more than 98% of initial deposit of pendimethalin was dissipated and observed half life in raddish field was 6.45 days and 10.03 days for pendimethalin applied @ 0.5 and 0.75 kg ha^{-1} respectively. More than 60 per cent of the initial deposit of oxyfluorfen was dissipated at the time of harvest of crop and 6.96 days and 12.26 days of half life was observed at 0.1 and 0.15 kg ha^{-1} of oxyfluorfen application respectively. In raddish tubers the detected residues of pendimethalin and oxyfluorfen were below maximum residue limits (MRL values).

***Key words*:** Oxyfluorfen, pendimethalin, persistence, radish, residues

Among all the chemicals use of herbicides for the control of weeds has become imminent especially in the irrigated agriculture for a wide variety of reasons like non availability of labour, high labour cost, unfavorable climatic conditions for weeding etc. Pendimethalin and oxyfluorfen is recommended as pre-emergence herbicides in most of the vegetable. After application of these herbicides to soil, undergoes decomposition and a part may be taken by plants accumulating in the edible parts, which are found to be toxic in nature. The residual activity of herbicides depends upon the soil type, soil moisture and temperature (Dharumarajan *et al.,* 2008). Application of recommended dose of herbicides may not pose serious problem for environmental pollution (Adachi *et al.*, 2007).Quantitative determination of herbicide residues helps in understanding the degradation pattern in the soil. Hence, an experiment was undertaken to study degradation pattern of pendimethalin and oxyfluorfen in soil and their residues in edible parts of radish.

MATERIALS AND METHODS

A field experiment was conducted on an Alfisol at College Farm, College of Agriculture, Rajendranagar. The soil was sandy clay loam in texture with pH 6.57, E.C 0.16 dS m^{-1} and 0.53 % of organic carbon content. Radish crop was grown with six treatments and four replications in randomized block design. The crop was treated with the common dose of fertilizers. Treatment details involving two doses of Pendimethalin @ 0.5 and 0.75 kg a.i. ha^{-1} and oxyfluorfen @ 0.1 and 0.15 kg a.i.ha^{-1} applied at 2 days after sowing. Initial soil samples from surface layer (0-15 cm) Soil samples were collected at 0, 15, 30, and 45 days after spraying of herbicides for the analysis of residues of pendimethalin and oxyfluorfen by gas chromatograph. The edible parts were also collected at the time of harvest for analysis of residues by gas chromatograph.

Standardization of Technical Grade Herbicides by Using Gas Chromatography:

Preparation of standard solutions

Technical grade pendimethalin (94.5% purity) obtained from M/S BASF India Ltd., and technical grade oxyfluorfen (97 % purity) obtained from M/s Indofil Chemicals were used in the present study. Standard solutions of pendimethalin and oxyflourfen of 100 µg mL^{-1} were prepared by dissolving 105.8 and 102.2 mg of respective technical grade herbicides in 100 ml of hexane. From this stock solution (1.0 mg ml^{-1}) was prepared for pendimethalin and oxyfluorfen in acetone. Fortification trials were conducted with 2 ppm to 0.001 ppm solutions and all stock, fortification and internal standard solutions were stored at – 20 ^{0}C in the deep freezer until use.

Method and level of fortification

The reference standards of pendimethalin and oxyfluorfen were used for quantification, recovery and determination of retention time of the herbicide. The soil and grain samples were collected from fields where no herbicide was applied. The samples were ground, sieved and the required quantity of the technical grade pendimethalin and oxyfluorfen were added to 50g soil or 20g grain sample. All samples were replicated twice. The soil and grain samples were fortified with 1ppm and 2ppm solutions. Control as well as blank samples were maintained to check for the contamination and interferences.

Extraction: A representative 10 g sieved soil /5 g edible parts was extracted with 150 ml of acetone : hexane. The samples were kept over night and filtered through buchner funnel and again the samples were rinsed with another fifty ml of acetone : hexane and the extract was evaporated.

Clean up: To a chromatographic column (2 mm i.d.) fitted below with cotton, 4 g of florosil followed by 10 g of anhydrous sodium sulphate was added. The concentrated extract was diluted to 10 ml with 10% acetone in hexane. Then the solution was transferred

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to florosil column. Container was rinsed with hexane and transferred to column. The column was eluted with about 5 ml min^{-1}. Florisil elute is concentrated to 1 ml. The extract is used for the determination of herbicide residues by GLC on ECD. The prepared solutions were injected to GC by using following requirements: Gas Chromatograph (Shimadzu GC 2010) equipped with Electron capture detector with Ni 63 (ECD), ZB-5 , 30 m length ID 0.53 mm, film thickness 1.50 um column was used for determination of residues. The following parameters were maintained for analysis. Carrier gas: Nitrogen; Carrier gas flow rate: 53.6 ml $minute^{-1}$; Injector temperature: 240; Injector split ratio: 1: 10; Detector temperature: 260 and make up gas flow: 60 ml $minute^{-1}$. Pendimethalin eluted as a peak at 10.77 minutes and oxyfluorfen at 15.96 minutes.

Estimation: One micro liter of reference standard solution of herbicide was injected. The peaks by their retention time were identified and the peak area was measured. The amount of residues of herbicide was calculated**.**

Calculation: Residues in mg kg^{-1}

$$\frac{\text{area of sample}}{\text{area of standard}} \times \frac{\mu\text{l std. injected}}{\mu\text{l sample injected}} \times \frac{\text{Final volume}}{\text{Wt. of the sample}} \times \text{recovery factor}$$

The recovery studies for pendimethalin and oxyfluorfen were carried out from 2 ppm to 0.001 ppm. Percent recovery of pendimethalin varied from 85 to 94 per cent and for Oxyfluorfen per cent recovery varied from 82 to 90 per cent. Limit of detection of pendimethalin was 0.001µg ml^{-1} and for oxyfluorfen the limit of detection is 0.004 µg ml^{-1}.

RESULTS AND DISCUSSION

The estimated residues of both herbicides were decreased from 0 DAS to at the time of harvest. Residues of pendimethalin at the time of harvest are below detectable limits. The results showed that at harvest in both the seasons more than 98% of initial deposit of pendimethalin was dissipated and observed half life in radish field was 6.45 days and 10.03 days for pendimethalin applied @ 0.5 and 0.75 kg ha^{-1} respectively. More than 60 per cent of the initial deposit of oxyfluorfen was dissipated at the time of harvest of crop and the observed half value is 6.96 days and 12.26 days at 0.1 and 0.15 kg ha^{-1} of oxyfluorfen application, respectively.

The residues of both the herbicides were decreased during summer season as compared to *rabi* season. This may be attributed to physical parameters like temperature, wind velocity and moisture level. Comparatively faster rate of degradation of herbicides in the summer season in the present study could be attributed to higher day temperature and relative humidity. Similar observations were also reported by Diwan *et al*. (1999) for the dissipation of pendimethalin and fluchloralin in soil; Guha *et al*. (1992) for the dissipation of fluchloralin in kharif paddy under West Bengal Agriculture conditions; Pandit and Choudhury, 1994 for residue and persistence of pendimethalin on groundnut.

High rainfall during summer season (122.0 mm for April, May and June) which appears to have cause very rapid loss of herbicide residues by leaching and run off, as the herbicide was sprayed on surface only. Photodecomposition might be another factor for the loss of these herbicides from soil the high temperatures during summer as compared to *rabi* season were may also helped in faster degradation of herbicides in soils.

Radish tubers analyzed for residues using gas chromatograph after harvest of crop, In both the seasons, the detected residues were below maximum residue limits (MRL values). As a part of applied herbicides were adsorbed to soil, a part may be leached down to deeper layers and the initial deposits of pendimethalin and oxyfluorfen were dissipated at the time of harvest of the crop may contributed to lower levels of herbicides in edible parts of radish (Anon., 2000).

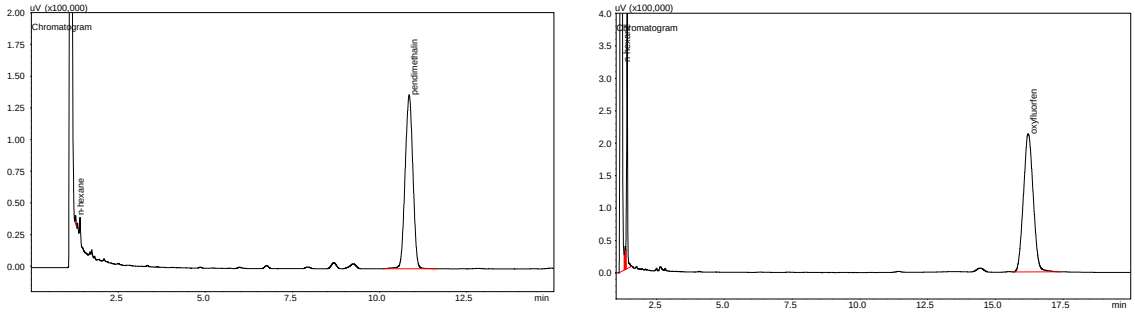

Fig.1: Standard chromatograms of pendimethalin and oxyfluorfen (1 ug ml^{-1})

Table 1: Residues (mg kg^{-1}) and dissipation of pendimethalin and oxyfluorfen in raddish grown soil

Treatment	Days after application				Half life
	0	15	30	AH	
Pendimethalin @ 0. 5 kg a.i.ha^{-1}					
I season	0.206	0.138	0.084	BDL	
II season	0.209	0.115	0.068	BDL	
Average	0.207	0.126	0.076	-	6.45 days
% of initial amount degraded		(56.10)	(73.87)	(100.00)	
		Y=0.2593+0.0466x			
Pendimethalin @ 0.75 kg a.i.ha^{-1}					
I season	0.304	0.248	0.085	BDL	
II season	0.312	0.237	0.052	BDL	
Average	0.308	0.242	0.068	-	10.03 days
% of initial amount degraded		(21.43)	(77.92)	(100.00)	
		Y=0.3194+0.03001x			
Oxyfluorfen @ 0.1 kg a.i.ha^{-1}					
I season	0.041	0.032	0.029	0.011	
II season	0.048	0.044	0.025	0.008	
Average	0.044	0.038	0.027	0.009	6.96 days
% of initial amount degraded		(13.64)	(38.64)	(78.41)	
		Y=0.04715+0.04322x			
Oxyfluorfen @ 0.15 kg a.i.ha^{-1}					
I season	0.063	0.063	0.050	0.035	
II season	0.069	0.054	0.047	0.022	
Average	0.066	0.058	0.048	0.028	12.26 days
% of initial amount degraded		(11.36)	(26.52)	(56.82)	
		Y=0.06875+0.02455x			

Table 2: Residues of pendimethalin and oxyfluorfen in edible parts of radish

Treatment	Residue (mg kg^{-1})	
	Rabi	**Summer**
T_1 : Pendimethalin @ 0.5 kg ha^{-1}	0.0026	0.0027
T_2 : Pendimethalin @ 0.75 kg ha^{-1}	0.0063	0.0056
T_3 : Oxyfluorfen @ 0.10 kg ha^{-1}	0.0072	0.0073
T_4 : Oxyfluorfen @ 0.15 kg ha^{-1}	0.0077	0.0081

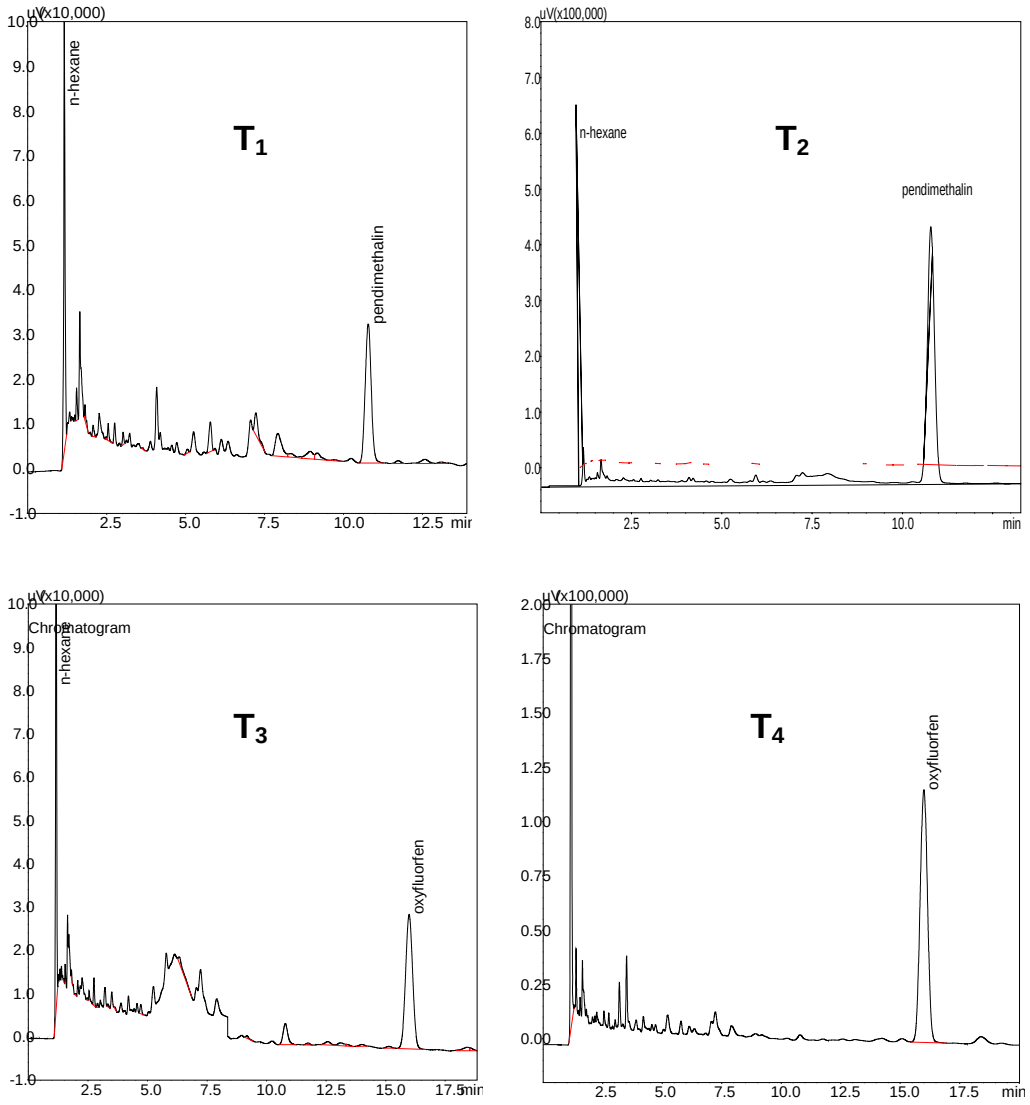

Fig. 2: Chromatograms showing pendimethalin (0.5 (T_1)and 0.75(T_2) kg a.i ha^{-1}) and oxyfluorfen (0.1(T_3) and 0.15(T_4) kg a.i. ha^{-1}) residues in soil at 0 DAS

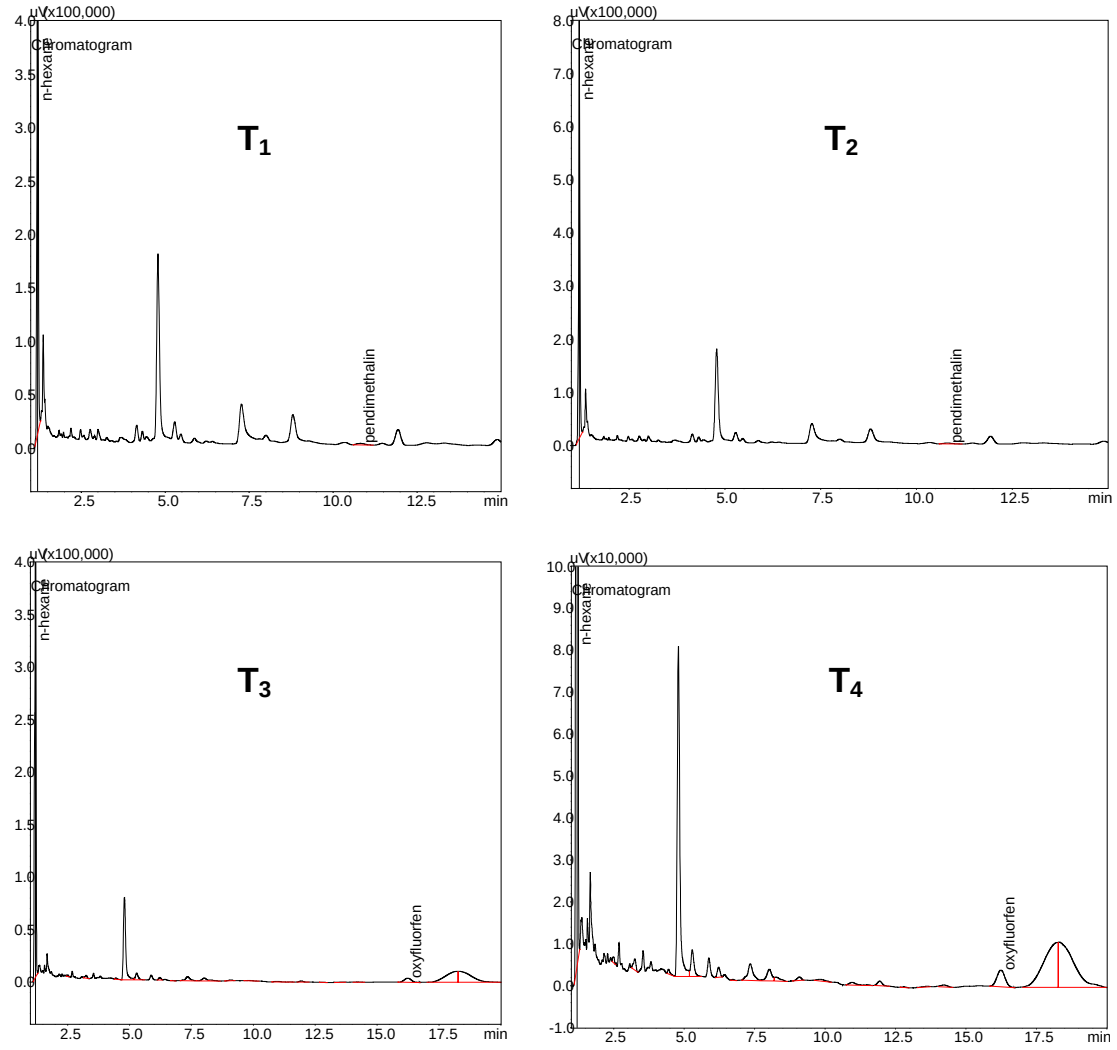

Fig. 3: Chromatograms showing pendimethalin (0.5 (T_1)and 0.75(T_2) kg a.i ha^{-1}) and oxyfluorfen (0.1(T_3) and 0.15(T_4) kg a.i. ha^{-1}) residues in soil at the time of harvest

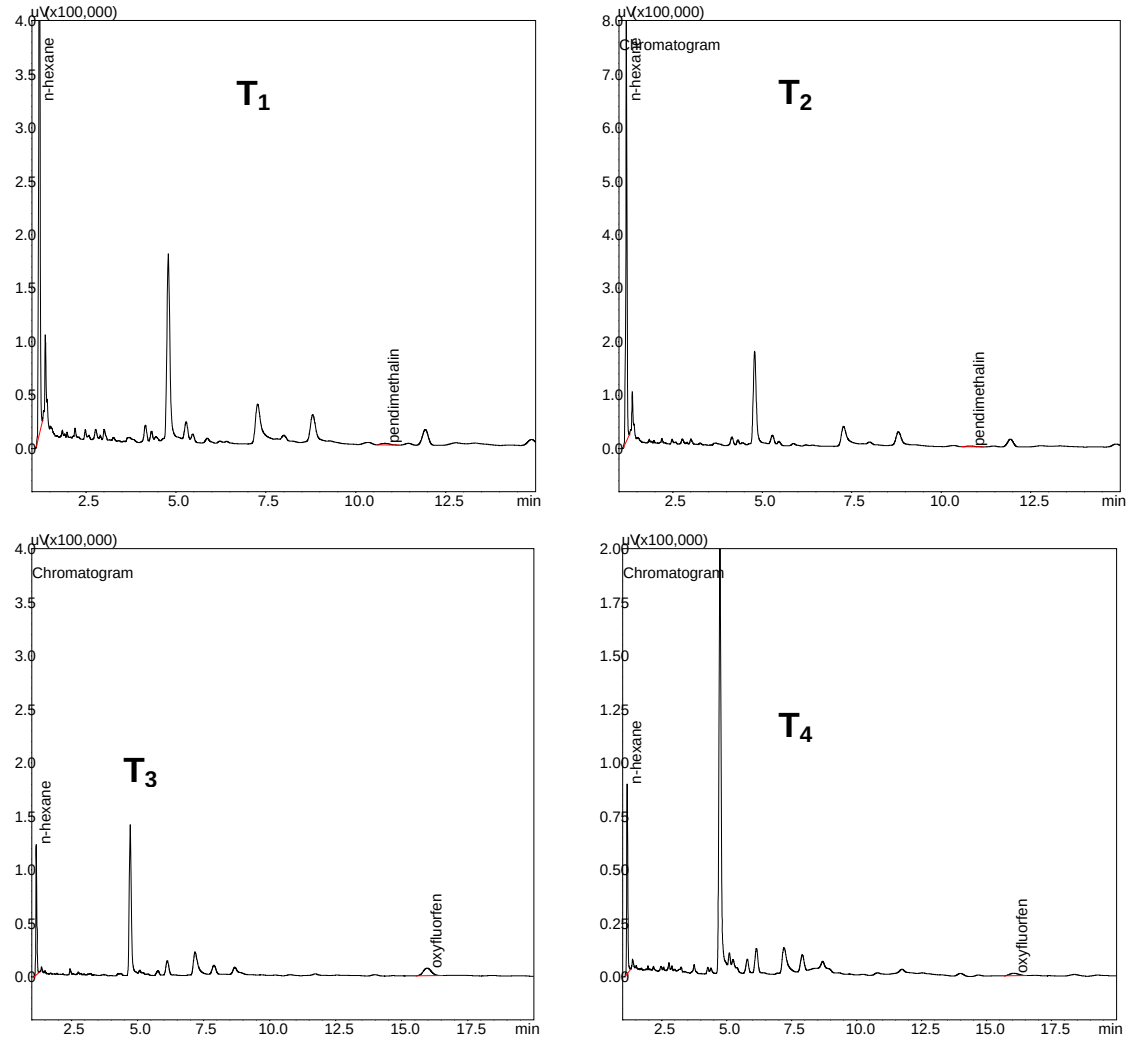

Fig.4: Chromatograms showing pendimethalin (0.5 (T_1) and 0.75(T_2) kg a.i ha^{-1}) and oxyfluorfen (0.1(T_3) and 0.15(T_4) kg a.i. ha^{-1}) residues in raddish tubers

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