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PERSISTENCE OF PROFENOFOS AND CYPERMETHRIN IN TOMATO GROWN UNDER MID HILL CONDITIONS OF HIMACHAL PRADESH

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ABSTRACT

Profenofos and cypermethrin both individually and as ready-mix product were applied at recommended rate 400 g and 40 g a.i./ha and at double the recommended rate 800 g and 80 g a.i./ha, on tomato. The fruits and soil samples were collected after second spray. Residues of both insecticides were determined by using gas chromatograph, Agilent 6890N having electron capture detector. In fruits, the residues of profenofos and cypermethrin reached below the limit of determination in 10 and 5 days when applied at recommended rate and in 15 and 7 days when applied at double recommended rate. In soil, residues of profenofos persisted upto10 days, whereas residues of cypermethrin were below determination limit in 10th day sampling in individual insecticides. However, profenofos residues persisted for 0 day in soil whereas, cypermethrin residues were not detected even on the 0 day sampled soil in ready-mix formulation.The study revealed that the persistence behavior in fruits were almost same when applied individually or as ready-mix insecticides. Therefore, safe waiting period of 1 day is suggested for ready-mix as well as for individual insecticides at recommended and double the recommended rate to reduce the risk before consumption of tomato fruits.

KEY WORDS
Fruits
Soil
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INTRODUCTION

Tomato (*Solanum lycopersicum* L.) is one of the largest grown vegetable crop under mid hill conditions of Himachal Pradesh. It is known as protective food for its special nutritive value as low in fat and calories, free of cholesterol and rich in vitamins A and C, b-carotene, lycopene and potassium (Pawar, 2012). Here, it is consumed in raw form as salad, home-cooked or processed as juice, paste or sauce. As it is a main cash crop in Himachal Pradesh it gives good monetary returns to the farmers. The production of tomato crop is affected by a variety of limiting factors including insect-pests. Hence, various insecticides are being used by farmers inHimachal Pradesh for their effective control. The present study were undertaken to evaluate the persistence behavior of ready-mix and individual insecticides on tomato crop.

In India, a large number of ready-mix insecticide formulations have been registered for use on various crops (Regupathy *et al.*, 2004). Ready-mix insecticide formulations have been found effective against insect pests of many vegetables (Dharne and Kabre, 2009; Kumar and Shivaraju, 2009). In tomato, profenofos and cypermethrin separately or in ready-mix, have been found effective in controlling insect-pests (Tripathi *et al.*, 2003; Mishra 2002; Sarangdevot *et al.*, 2010 a & b). This effectiveness of these two insecticides on tomato has propelled the use of combination product Roket 44EC on tomato. However, information available on the persistence behavior of these ready-mix products in/on tomato fruit and soil are scare under present environmental conditions. So, Roket 44EC a combination of profenofos 40% + cypermethrin 4% is one such ready-mix formulation which is abundantly available in the market and was tested to know the residues of these insecticides in the environment.

This crop is susceptible to attack by a large number of pests including tomato fruit borer, mites, leaf miner, aphids, whiteflies etc. (Dikshit and Pachauri, 2000; Gavkare *et al.*, 2013). In order to prevent the damage to the crop the farmers rely heavily on the usage of many pesticides viz. quinalphos, phosalone, fenvalerate, cypermethrin, deltamethrin etc. (Awasthi, 1986). Improper and injudicious use of pesticide, besides posing health threat to the farm workers, also leave harmful pesticide residues on the crop and soil and causes development of pest resistance leading to the losses to the crops (Kumar and Singh, 2014). To combat this menace, usage of insecticide mixtures is a promising option.

Therefore, the present studies were contemplated with an objective to study the persistence behavior of the combination product and its comparison with the residue status when applied individually, following spray application.

MATERIALS AND METHODS

The experiment was laid out in randomized block design at the experimental farm of the Department of Entomology, UHF Nauni, Solan (H.P.) during 2009 and

2010 and each treatment was replicated thrice. The crop was raised by following package of practices of vegetable crops (Anonymous, 2009). Tomato (Him Sohna var.) was sprayed at fruit formation stage with individual insecticides profenofos (Profex 50EC), cypermethrin (Challenger 25EC) and the ready-mix formulation Roket 44EC (profenofos 40% and cypermethrin 4%) at the recommended rate (RR) 400 g and 40 g a.i./ha and at double recommended rate (DRR) 800 g and 80 g a.i./ha. A total of two sprays were given at 15 days interval. Control plots with only water spray were maintained simultaneously for comparison. After the second spray, fruit samples (1kg) from each replication were collected randomly at 0 (2 hours after spray), 1, 3, 5, 7, 10 and 15 days intervals. Soil samples (1 kg) from each replication were collected on 0, 10 and 20 days after application. The tomato fruits were homogenized and analysed for respective insecticides. Soil samples were shade dried and sieved.Tomato fruit samples were analyzed according to multi residue method (Sharma, 2007).Homogenized tomato fruit sample (100 g) was extracted with 200 mL of acetone, filtered through Buchner funnel under low suction and rinsed with 50 mL acetone. From total extract, an aliquot of 60 ml (30 g equivalent of sample) was transferred to 1 litreseparatory flask and extracted with 200 ml mixture of hexane and dichloromethane (1:1, v/v). The separatory flask was shaken vigorously for 1 min and then allowed the phases to separate into organic and aqueous phase. The lower aqueous phase was transferred to another 1 litre separatory flask and remaining organic phase was retained in the same separatory flask. Ten millilitre saturated sodium chloride solution was added to the left amount of aqueous phase and again partitioned twice with 100 ml dichloromethane. Lower aqueous phase was discarded and upper organic phase was transferred to the 1st separatory flask. Pooled organic phase was passed through anhydrous sodium sulfate and evaporated to dryness by using vacuum rotary evaporator at 40^{0}C. Finally, the residues were taken up in 3 mL (1 + 2) acetone for cleanup. One millilitre sample extract was diluted with 10 ml of acetone: hexane (1:9) mixture, loaded on 4 g Florisil column (22 mmi.d.), overlaid with 2 g layer of sodium sulphate and eluted with 50 mL solvent mixture (50 % dichloromethane: 48.5 % hexane: 1.5 % acetonitrile). Another fraction, 2 mL of sample was loaded on a charcoal column which was prepared by placing one inch layer of Celite 545, 6 g absorbent mixture (1:4 w/w Charcoal: Celite 545) and then overlaid with 2 g sodium sulfate. The sample extract was loaded on to the column and eluted with 200 ml of 2:1 acetone: dichloromethane mixture. Eluate from both the column fractions was pooled evaporated to dryness in vacuum rotary evaporator at 50^{0}C. The residues were redissolved in 3 mL toluene and injected one μl into gas chromatograph for residue estimation.

Soil samples were analyzed according to the multi residue method (Brar, 2003). A dried and sieved representative soil sample of 20 g was mixed with 0.5 g activated charcoal + 0.5 g Florisil and packed in 2×40 cm glass column containing about 5 cm layer of anhydrous sodium sulphate over a plug of cotton at the bottom. Eluted the column with 50 mL mixture of acetone and hexane (1:4). The eluate was evaporated to dryness and residues were taken up in 1mL toluene. Finally, one µL was injected into gas chromatograph for residue estimation.

The chemicals used were of analytical grade obtained from M/S Merck Specialties Private Limited, Mumbai, India. Residues were estimated by using Gas-Chromatograph (Agilent 6890N) having ECD detector and DB-5 Ultra Performance Capillary column (Cross-linked Methyl Silicon, length 30 m, 0.250 mm internal diameter with 0.25 µm film thickness).The analytical method employed to estimate residues was validated by spiking the control fruit and soil samples at five different concentrations viz., 0.05, 0.10, 0.20, 0.50 and 1.0 mg/kg for profenofos and cypermethrin. The limit of determination (LOD) of profenofos and cypermethrin was 0.05mg/kg. The residue data were subjected to statistical analysis (Hoskins, 1961).

RESULTS AND DISCUSSION

Data presented in Table 1 depicts reliability of analytical method tested by spiking of untreated tomato fruits and soil samples at different concentrations. Recovery of profenofos was between 86.00-93.00 per cent with relative standard deviation (RSD) of 0.034-0.870 per cent in fruits and 86.00-92.00 per cent with 0.033-0.710 per cent RSD in soil fortified samples. Recovery of cypermethrin was between 88.00-90.00 per cent with relative standard deviation (RSD) of0.034-0.738 per cent in fruits and 86.80-90.00 per cent recovery with 0.062-0.753 per cent RSD in soil fortified samples.The results are in agreement with Pal (2011) who has observed recovery 88.80-91.39 per cent for malathion and 86.60-92.31 per cent for cypermethrin in capsicum fruits. Ehlang *et al.* (1989) reported 89.00-108.00 per cent recovery of profenofos in sandy loam soil while Brar (2003) reported 78.60 recovery of pyrethroids

Table 1: Recovery of profenofos and cypermethrin from tomato fruits and soil samples.

Insecticides		Fruits		Soil	
	Fortification level, (mg/kg)	Mean recovery (%)	Relative standard deviation (%RSD)	Mean recovery (%)	Relative standard deviation (%RSD)
Profenofos	0.05	88.00	0.870	88.00	0.679
	0.10	86.00	0.349	86.00	0.710
	0.20	89.00	0.086	89.00	0.142
	0.50	91.00	0.034	91.00	0.079
	1.00	93.00	0.038	92.00	0.033
Cypermethrin	0.05	88.00	0.738	88.00	0.728
	0.10	88.00	0.286	88.00	0.301
	0.20	89.00	0.202	87.50	0.753
	0.50	89.00	0.147	86.80	0.401
	1.00	90.00	0.034	90.00	0.062

Table 2: Statistical constants of profenofos and cypermethrin on tomato fruits

Years,Insecticides	Dosage	Profenofos Regression equation (y=—)	r	RL_{50}	Cypermethrin Regression equation (y=—)	r	RL_{50}
2009, Combination	RR	- 0.101-0.159X	-0.990	1.89	-0.518-0.252X	-0.991	1.19
	DRR	0.101-0.131X	-0.990	2.30	-0.507-0.148X	-0.984	2.03
Individual	RR	-0.233-0.142X	-0.991	2.11	-0.605-0.232X	-0.997	1.29
	DRR	0.080-0.133X	-0.996	2.26	-0.491-0.148X	-0.971	2.04
2010, Combination	RR	-0.030-0.166X	-0.989	1.81	-0.459-0.272X	-0.998	1.10
	DRR	0.143-0.136X	-0.993	2.21	-0.253-0.194X	-0.983	1.54
Individual	RR	-0.172-0.148X	-0.990	2.02	-0.500-0.258X	-0.996	1.16
	DRR	0.102-0.135X	-0.996	2.22	-0.278-0.187X	-0.981	1.61

RR = Recommended rate, DRR = Double recommended rate, r = Correlation, RL50 = Residue half-life

Table 3: Residues of profenofos (400 g a.i./ha) and cypermethrin (40 g a.i./ha) in tomato cropped soil.

Interval (Days)	2009 Combination Profenofos Residues ± SD(mg/kg)	Cypermethrin Residues ± SD(mg/kg)	Individual Profenofos Residues ± SD(mg/kg)	Cypermethrin Residues ± SD(mg/kg)	2010 Combination Profenofos Residues ± SD(mg/kg)	Cypermethrin Residues ± SD(mg/kg)	Individual Profenofos Residues ± SD(mg/kg)	Cypermethrin Residues ± SD(mg/kg)
0	0.220 ± 0.005	BDL	0.316 ± 0.005	0.073 ± 0.003	0.278 ± 0.003	BDL	0.371 ± 0.001	0.078 ± 0.004
10	BDL		0.103 ± 0.005	BDL	BDL		0.109 ± 0.007	BDL
20			BDL				BDL	
30								

BDL- Below determination limit

in soil. The decrease in level of residues in individual and combi insecticides treatments at different days interval in fruits are presented in Fig. 1.

Profenofos initial deposits on tomato fruits from mixture (Roket 44EC) and individual insecticide formulation (Profex 50EC) were 0.713-0.832 mg/kg and 0.527-0.611 mg/kg which dissipated to 0.051-0.053 mg/kg on 7th day, respectively at recommended rate. In double the recommended rate, initial deposits of profenofos from mixture were 1.103-1.275 mg/kg which dissipated to 0.052 mg/kg and deposits 1.098-1.166 mg/kg from individual profenofos dissipated to 0.051 mg/kg in 10 days. Two years persistence data showed that they followed almost the same dissipation pattern in the same day whether applied individually or as ready-mix formulation. The present findings are in agreement with those of Shah *et al.* (1999) who reported 0.762 mg/kg profenofos initial deposits on okra fruits sprayed with 0.044 per cent Polytrin-C (profenofos 40% + cypermethrin 4%).

Initial deposits of cypermethrin on tomato fruits from mixture with profenofos applied @ 40 ga.i./ha were 0.278-0.333 mg/kg whereas at double recommended rate, the initial deposits were 0.305-0.513 mg/kg. When applied individually, cypermethrin initial deposits at recommended rate were 0.259-0.298 mg/kg and at double the recommended rate cypermethrin deposits were 0.300- 0.500 mg/kg. Rai *et al.* (1986) observed 0.46 mg/kg initial deposits of cypermethrin on cauliflower at 0.0075 per cent spray concentration. Bhupinder and Udeaan (1989) reported 0.65 mg/kg and 1.43 mg/kg initial deposits at 50 ga.i./ha and 100 ga.i./ha doses of cypermethrin, respectively in okra fruits.

Data contained in Table 2 revealed that there is decline in residues with the time lapse at both the level of application. The persistence of insecticides is generally expressed in terms of RL_{50} *i.e.* time for disappearance of insecticide initial deposits to 50 per cent. The RL_{50} values are often obtained by fitting first-order kinetics to observed degradation pattern. Shah *et al.* (1999) sprayed Polytrin-C (Profenofos 40% + Cypermethrin 4%) on okra crop at 0.044% and studied half-life values of 1.35 and 3.95 days forprofenofos and cypermethrin, respectively. The slight variation of half-life values in our study as depicted below in table 2 may be due to variation in treatment doses, age, vigor, type and varieties of crop.

Profenofos initial deposits were below the MRL in tomato fruits from ready-mix formulation as well as inindividual insecticide formulation (MRLs as per Codex Alimentarius Commission: 2.0 mg/kg for profenofosand 0.5 mg/kg for cypermethrin). Cypermethrin initial deposits were also below the MRL on tomato fruits when applied individually and as ready-mix product at all recommended rates whereas at double recommended rate, initial deposits were above MRL but reduced below MRL within a day. These findings are in agreement with Gupta *et al.* (2011) who suggested waiting period of 1-day from consumer's safety point of view.

Profenofos residues 0.220-0.278 mg/kg were detected in soil at the recommended rate400 g a.i./ha in combi-mix formulationwhich became non-detectable on 10th day (Table3).When profenofos applied individually on the crop then residues in soil became non-detectable on 20 days sampled soil at recommended rate. Gupta *et al.* (2011) observed 0.048 mg/kg profenofos residues in 0 day after the application of Roket 44 EC (profenofos 40% + cypermethrin 4%) @ 1 L/ha on tomato crop and residues became non detectable on 7th day in soil at recommended rate.

Cypermethrin residues in soil were below determination limit in 0 day at recommended rate (40 g a.i./ha). However, in individually applied cypermethrin, its residues were detected in soil on 0 day and became below the determination limit in 10 days. Present findings are in accordance with findings of

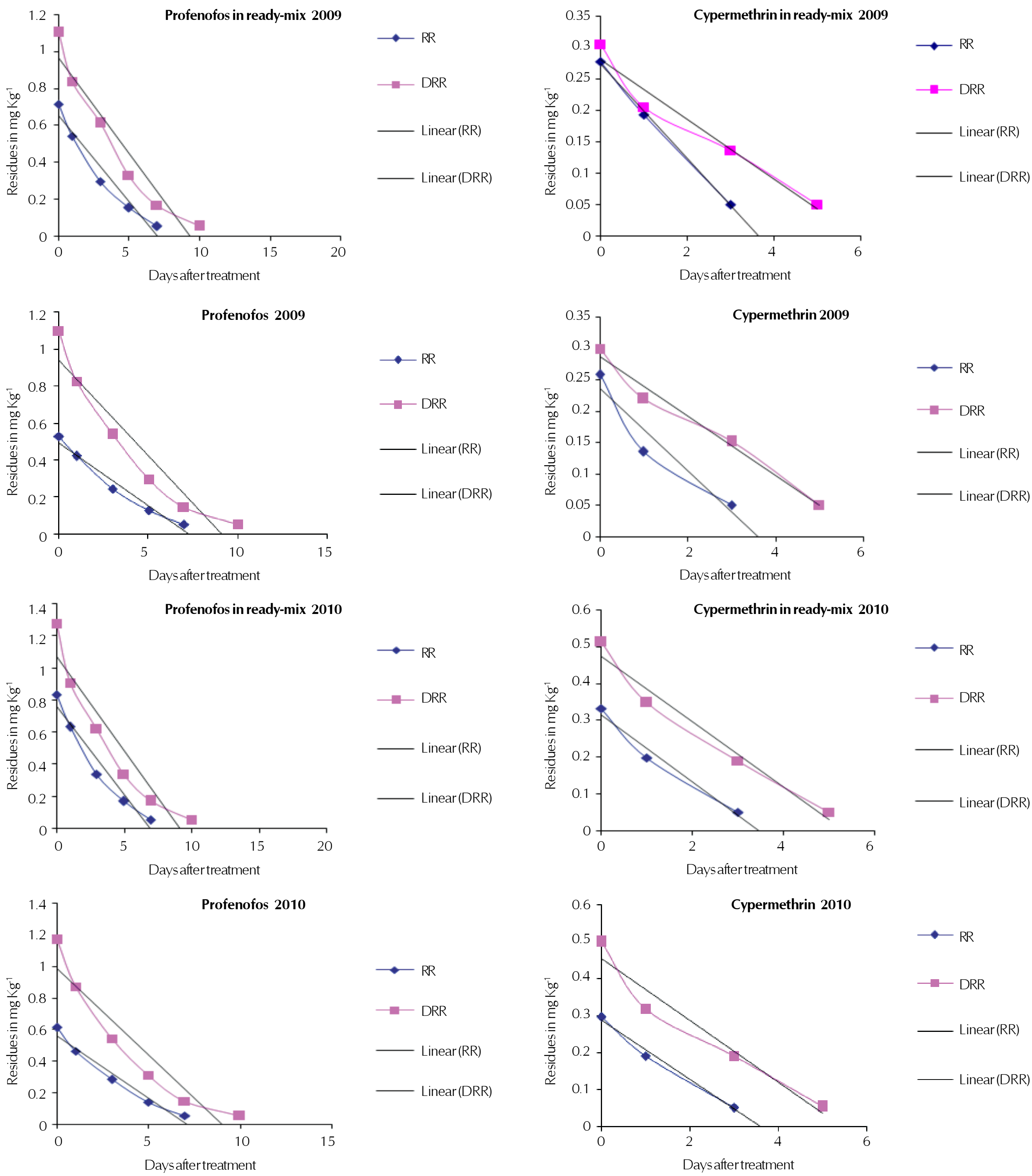

Figure 1: Dissipation behaviour of Profenofos and cypermethrin on tomato fruits.

Gupta et al. (2011) who observed cypermethrin residues below detection limit in soil samples after the application of Roket 44 EC @ 1 L/ha on tomato crop. Studies revealed that when individual insecticides were applied alone on tomato crop then higher residues were detected in soil in comparison to their application in ready-mix formulation. Residues also persisted for longer period in alone treatment in comparison to ready-mix treatment. The present findings are in agreement with the findings of Swarcewicz and Gregorczyk (2011) who observed longer persistence of pendimethalin alone (RL50

44.4 days) in comparison to pendimethalin+metribuzin mixture (RL50 37.7 days) in clay loam soil.

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