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Research Paper

DISSIPATION PATTERN OF ETHION AND PROFENOPHOS IN CURRY LEAF

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Abstract

Field experiment was carried out during *kharif* 2015and 16 with curry leaf variety Suwasini to study the dissipation pattern of ethion 50 EC and profenophos 50 EC at 500 g a.i. ha^{-1}. Two sprays were given, first at vegetative stage and second at 10 days after first spray. The leaf samples were collected at 0, 1, 3, 5, 7, 10, 15, 20, 25, 30 and 45th day after second spray and soil samples at 45th day for residue analysis at AINP on Pesticide Residues, Rajendranagar, Hyderabad. An initial deposits of ethion and profenophos were 21.04 and 19.83 mg kg^{-1} after second spray, respectively. No residues were observed in soil samples. The residues reached to Below Determination Level (BDL) at 25th day for both insecticides. As there were no pesticide recommendations and MRLs fixed for any of the pesticide in curry leaf, the day at which residues reach BDL can be suggested as the safe harvest for curry leaf.

Key words: Dissipation pattern, ethion, profenophos, curry leaf.

INTRODUCTION

Curry leaf [*Murraya koenigii* (L.) Sprengel] is a leaf spice of the citrus family Rutaceae. Curry leaves, an inevitable part of spicing up dishes are not a part of mere garnishing. They are rich in medicinal, nutraceutical properties and have even cosmetic uses. In India, of late it is cultivated on the commercial scale (Tamil Nadu, Karnataka, Andhra Pradesh and Telangana) and has gained importance as a major spice crop with high export potential (Mohan, 2012). A total of 12 insect pests belonging to 10 families of 5 insect orders were recorded infesting curry leaf plants (Tara and Monika Sharma, 2010). As per Insecticides act, 1968 there is no pesticide recommendation for spray on curry leaf as on today and hence there are no MRL's suggested by Codex Alimentarius Commission. But, farmers were using pesticides indiscriminately that are designed to control the pest even if there are no recommendations for the crop and whether the pest is present or not. Hence, residues were detected at the farm gate level or export location and led to the rejection of the consignment (Ramakrishnan *et al.,* 2015). According to the report of the Indian delegation at 45th session of the Codex Committee on Pesticide Residues (CCPR) held at Beijing P. R. China (May 6-11, 2013) in agenda no. 11, India is considered for fixation of new and revised MRL's of profenophos, chlorpyriphos, cypermethrin, methyl parathion, triazophos, ethion and quinalphos in

curry leaves based on good agricultural practice (GAP) trials and monitoring data and should submit in the prescribed format to Joint FAO/WHO Meeting on Pesticide Residues (JMPR), as a follow-up, for evaluation in 2014 for fixation of MRL on curry leaves. By keeping in view all these most important issues of concern, the present comprehensive study, on dissipation of newer insecticides viz., ethion and profenophos was carried out in curry leaf.

MATERIAL AND METHODS

A field experiment was conducted during *kharif*, 2015 and 2016 at Department of Agroforestry, College of Agriculture, Professor Jayashankar Telangana State Agricultural University (PJTSAU), Rajendranagar, Hyderabad with seven treatments and three replications laid out in Randomized Block Design (RBD)and the laboratory studies were carried out in the All India Network Project on Pesticide Residues, Hyderabad Centre. The insecticidal treatments *viz.,* ethion 50 EC and profenophos 50 EC at 500 g a.i. ha^{-1} were sprayed twice, first at vegetative stage and second at 10 days after first spray with hand compression knapsack sprayer and the amount of spray fluid used was 500 l ha^{-1}.

Sample Collection

The curry leaf samples were collected at 0, 1, 3, 5, 7, 10, 15, 20, 25, 30 and 45th day after second spray and soil samples at 45th day for residue analysis. The leaf samples (1 kg) were collected randomly from each treatment in polythene bags and brought to the laboratory immediately for further sample processing. Soil samples (2 kg) were collected following "Z" sampling plan, air dried, grounded, and sub-sampling was done following quartering method.

Extraction and clean up procedure for leaf samples

The leaf samples were analyzed for both ethion and profenophos residues following the AOAC official method 2007.01 (QuEChERS) after validation of the method at the laboratory. One kg of leaf samples collected from all treatments were homogenized with robot coupe blixer separately. 7.5 g sample was taken in 50 ml centrifuge tube and added with 30±0.1 ml acetonitrile. The sample was homogenized at 14000-15000 rpm for 2-3 min using Heidolph silent crusher. The samples were then added with 3±0.1 g sodium chloride and mixed by shaking gently followed by centrifugation for 3 min at 2500-3000 rpm to separate the organic layer. The top organic layer of about 16 ml was taken into the 50 ml centrifuge tube and added with 9±0.1 g anhydrous sodium sulphate to remove the moisture content. 8 ml of extract was taken into 15 ml tube, containing 0.4±0.01 g PSA sorbent (for dispersive solid phase d-SPE cleanup), 1.2±0.01 g anhydrous magnesium sulphate and 0.05±0.01 g GCB (remove chlorophyll). The sample tube was vortexed for 30 sec, followed by centrifugation for 5 min at 2500-3000 rpm. The extract of 1 ml acetonitrile was transferred into 2 ml vial by filtering through 0.22µm filter paper for the analysis on LC-MS under standard operational conditions (Table 1).

Extraction and clean up procedure for soil samples

The soil samples were analyzed for both ethion and profenophos residues following the QuEChERS method after validation of the method in the laboratory. Two kg of soil was collected from each plot in polythene bags. The soil samples were pooled treatment wise, mixed well and about 200 g of the representative sample was drawn by quartering method. The soil samples were dried at room temperature under shade, ground, passed through 2 mm sieve and a representative 10 g sample was taken into 50 ml centrifuge tube. The sample tube is then added with 20±0.1 ml acetonitrile. The sample is then

added with 1±0.1 g sodium chloride and 4±0.1 g Magnesium sulphate mixed by shaking gently followed by centrifugation for 3 min at 3300 rpm to separate the organic layer. The top organic layer of about 10 ml was taken into the 15 ml centrifuge tube containing 1.5±0.1 g magnesium sulphate and 0.25 g PSA and sonicated for 1 min to remove air bubbles and centrifuged for 10 min at 3000rpm. The extract of about 1 ml (0.5 g sample) was transferred into vials for LCMS/MS analysis under standard operational conditions (Table 1).

Fortification and Recovery studies

The untreated control leaf and soil samples were fortified with required quantity of ethion and profenophos so as to obtain 0.5, 0.25 and 0.05 mg kg^{-1} fortification levels and the samples were extracted and cleaned up as per QuEChERS method to validate the suitability of method.

The recovery of ethion is 97.84, 120.42 and 92.13 per cent from the curry leaf fortified at 0.5, 0.25 and 0.05 mg kg^{-1}, while the profenophos fortified at 0.5, 0.25 and 0.05 mg kg^{-1} have shown the recovery of 99.91, 123.08 and 90.99 per cent. Hence, the limit of quantification (LOQ) is 0.05 mg kg^{-1} in curry leaf for both ethion and profenophos.

In soil samples, the recovery of ethion is 104.94, 84.69 and 83.71 per cent at 0.5, 0.25 and 0.05 mg kg^{-1} level of fortification, while the profenophos fortified at 0.5, 0.25 and 0.05 mg kg^{-1} have shown the recovery of 99.84, 85.05 and 86.76 per cent. Hence, the limit of quantification (LOQ) is 0.05 mg kg^{-1} in soil for both ethion and profenophos.

Table 1. LCMS parameters for analysis

LC-MS/MS	**SHIMADZU LC-MS/MS 8040**
Detector	Mass Spectrophotometer
Column	KINETEX, 2.6µ, C18 Column, 100 x 3.0
Column Oven Temperature	40°C
Retention Time (RT)	Ethion - 16.76 min
	Profenophos - 16.18 min
Nebulizing gas	Nitrogen
Nebulizing flow gas	2.0 litres/min
Pump Mode/ flow	Gradient/ 0.4 ml/min
LC Programme	A : Ammonium formate in water (10 Mm) - 65%
	B : Ammonium formate in methanol (10 Mm) - 35%
Total Time of Programme	24 min

RESULTS AND DISCUSSION

The initial deposits of ethion 50 EC and profenophos 50 EC at 500 g a.i. ha^{-1} were 21.04 and 19.83 mg kg^{-1}, respectively which dissipated to below determination levels (< 0.05 mg kg^{-1}) at 25 days. The half-life values were 3.14 and 3.40 days, respectively. As there were no MRLs fixed, the day at which residues reached to BDL was suggested as safe harvest i.e., 25days. Soil samples collected at harvest (45th day) were free from the residues of both pesticides (Table 2, 3 and fig 1 and 2). Parmar *et al*. (2012) reported that ethion sprayed@0.254 mg kg^{-1} in okra showed initial deposits of 0.254 mg kg^{-1} and half-life of 1.27 days. In chilli when ethion 50 EC was sprayed @ 500 and 1000 g a.i. ha^{-1} showed initial deposits of 2.4 and 4.84 mg kg^{-1}, respectively. They showed half-life values of 1.81 and 2.32 days and safe waiting periods of 2.65 and 5.63 days, respectively

(Sharma and Parihar, 2013). Results are in agreement with Pallavi *et al.* (2013), who detected a level as high as 25.63 mg kg^{-1} of profenophos in curry leaf. Renuka *et al.* (2006) on dissipation of profenophos on different crops clearly indicate that when applied at recommended dose, the initial deposits are less than 3 mg kg^{-1} and dissipate to BDL in 7-10 days depending on the crop, except on cardamom. The difference in reports of earlier workers to the present work may be due to difference in the matrix of curry leaf with other crops

Table 2. Dissipation of ethion in curry leaf

Days after last spray	Residues of ethion (mg kg^{-1})				Dissipation (%)
	R_1	R_2	R_3	Average	
0	22.13	19.98	21.01	21.04	0.00
1	19.09	17.45	18.05	18.20	13.50
3	13.89	13.01	14.89	13.93	33.79
5	6.57	7.88	8.05	7.50	64.35
7	4.89	5.54	5.89	5.44	74.14
10	2.98	1.94	2.01	2.31	89.02
15	1.04	2.09	1.99	1.71	91.87
20	0.09	0.25	0.21	0.18	99.14
25	BDL	BDL	BDL	BDL	100.00
Soil (45th day)	BDL	BDL	BDL	BDL	-
Regression equation	Y = 4.381 + (-0.096) X				
R^2	0.96				
Half-life (Days)	3.14				
BDL :	Below Determination Level (< 0.05 mg kg^{-1})				

Table 3. Dissipation of profenophos in curry leaf

Days after last spray	Residues of profenophos (mg kg^{-1})				Dissipation (%)
	R_1	R_2	R_3	Average	
0	20.01	19.91	19.56	19.83	0.00
1	16.92	16.69	15.91	16.51	16.74
3	14.73	14.04	13.91	14.23	28.24
5	11.39	12.01	11.92	11.77	40.65
7	6.91	6.29	6.49	6.56	66.92
10	3.39	4.02	3.77	3.73	81.19
15	1.91	1.80	1.29	1.67	91.58
20	0.21	0.35	0.29	0.28	98.59
25	BDL	BDL	BDL	BDL	100.00
Soil (45th day)	BDL	BDL	BDL	BDL	-
Regression equation	Y = 4.399 + (-0.088) X				
R^2	0.97				
Half-life (Days)	3.40				
BDL :	Below Determination Level (< 0.05 mg kg^{-1})				

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