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Dissipation pattern of Cypermethrin in Curyleaf

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Abstract

The field experiments were conducted from August 2014 to March 2016 following Randomized Block Design (RBD) with three replications to study the dissipation pattern of cypermethrin at 50 and 100 g a.i. ha^{-1}. One kg of curryleaf sample was collected randomly from each plot at 0 (one h after spraying), 1, 3, 5, 7, 10, 15, 20, 25, 30 and 35 days after the last application of the insecticide. The initial deposit of cypermethrin determined one hour after application on curryleaf samples at recommended dose and double the recommended doses were 2.99 and 3.83 µg g^{-1} which dissipated to 0.16 and 0.21mg kg^{-1} respectively. The residues reached BDL at 30 days in both the doses.

Keywords: Dissipation pattern, curry leaf, cypermethrin

1. Introduction

Curryleaf (*Murraya koenigii* L. Sprengel) is a leafy spice that belongs to the family Rutaceae. It's leaves are widely used in Indian cookery for flavoring food stuffs. Curryleaf is also used in many of the Indian ayurvedic and Unani prescription (Arulselvan *et al.*, 2007) [3]. In India, it is cultivated commercially in states like Tamil Nadu, Karnataka, Andhra Pradesh, Maharashtra, Madhya Pradesh, and West Bengal (Alturi *et al.*, 2002, Krishnamoorthy *et al.*, 2004) [2, 7]. Curryleaf is infested by a number of insect pests from its early stage and the damage continues throughout the life span of the crop. The major pests of curryleaf are psyllid- *Diaphorina citri* Kuwayama, tortoise beetles- *Silana farinose* (Boheman), mealy bugs –*Planococcus citri* (Risso), scale insects- *Unaspis citri* (Comstock), citrus butterfly-*Papilio demoleus* Linnaeus and leaf miner –*Phyllocnictis citrella* Stainton. *D. citri* is a serious pest and causes considerable yield loss throughout the crop period. Curryleaf, being a crop of export significance, it is essential to get the final harvest produce free from pesticide residues. Hence, it is necessary that a pesticide should be effective against a pest along with toxicologically acceptable residue on food commodity.

2. Material and method

The field experiments were conducted from August 2014 to March 2016 following Randomized Block Design (RBD) with three replications in farmer's field. Curryleaf (variety: Sengambu) was raised in 5 cent plots with a spacing of $1.2x1.5m^2$. The crop was grown by following recommended agronomic practices. The first spraying was done one month after pruning and subsequent application was made at 10 days interval using hand operated knapsack sprayer with 500 L volume of spray fluid ha^{-1}.The plot with same size but without insecticide application was compared simultaneously. It was ensured that insecticides under investigation had not been used earlier in the experimental plot.

2.1 Sampling

One kg of curryleaf sample was collected randomly from each plot at 0 (one h after spraying), 1, 3, 5, 7, 10, 15, 20, 25, 30 and 35 days after the last application of the insecticide formulations. All the collected samples were transported to the laboratory and processed immediately or stored at -4˚ C before analysis.

2.2 Sample preparation

Leaves alone were removed from the collected sample, chopped, mixed thoroughly and homogenized with high volume blade homogenizer for 2 min.

2.3. Extraction

A representative sample of 10 g was transferred into a 50 ml centrifuge tube and mixed using a vortexer for 1 min. after adding 20 ml of acetonitrile. About 4 g of anhydrous magnesium sulphate ($MgSo_4$) and 1g of sodium chloride (NaCl) were subsequently added and again shaken well by vortexer, then centrifuged at 6000 rpm for 10 min.

2.4. Clean-up
After centrifuging, 6 ml supernatant aliquot was transferred into a 15 ml centrifuge tube containing 100 mg Primary Secondary Amine (PSA), 600 mg anhydrous $MgSO_4$ and 100 mg Graphatized Carbon Black (GCB). The mixture was vortexed for 1 min. and then centrifuged for 10 min. at 3000 rpm. The upper extract (4 ml) was transferred into a turbovap tube and concentrated to near dryness under a gentle stream of zero air in a turbovap LV at 40˚ C. The final volume was reconstituted to about 1 ml using n-hexane and transferred into a 1.5 ml glass auto sampler vial for analysis.

2.5 Chemicals and reagents
The reference standards of cypermethrin (97.3%) was purchased from Sigma Aldrich, Bangalore, India. Acetonitrile, hexane and methanol of HPLC grade, sodium chloride and anhydrous magnesium sulfate of analytical grade were purchased from Merck (Mumbai, India). PSA (Bondesil 40 µm) and GCB were purchased from Agilent technologies, USA.

2.6 Fortification and Recovery studies
Untreated control samples were fortified with required quantity of cypermethrin to obtain 0.1, 0.5 and 1 µg g^{-1} fortification levels and samples were extracted and cleaned up as per QuEcHERS method to validate the suitability of the method. The results of the recovery from curry leaves carried out in three replicates at the levels of LOQ (0.1 µg g^{-1}), 5 times of LOQ (0.5 µg g^{-1}) and 10 times of LOQ (1 µg g^{-1}) are given in table 1. The results revealed that the average recovery of cypermethrin ranged from 84.67 to 97.00 per cent in curryleaf samples with the RSD of 1.80 to 2.06 per cent. The mean recoveries of cypermethrin spiked at three levels were found to be consistent and more than 84.67 per cent.

Table 1: Recovery percentage of cypermethrin in curryleaf

Spiking level (µg g^{-1})	Recovery %				
	R1	R2	R3	Mean*±SD	RSD%
0.1	99.00	97.01	93.10	97.00±2.00	2.06
0.5	93.00	95.03	91.00	93.00 ± 2.00	2.15
1	85.00	83.10	86.00	84.67 ± 1.53	1.80

* - Mean of three replications
SD- Standard Deviation
RSD- Relative Standard Deviation

3. Results and Discussion
Curryleaves were sprayed with cypermethrin 10 EC at the rate of 50 and 100 g a.i. ha^{-1} twice with an interval of 10 days (Fig. 1). The initial deposit of cypermethrin determined one hour after application on curryleaf samples at recommended dose and double the recommended doses were 2.99 and 3.83 µg g^{-1} (Table 1 and 2). A residue of cypermethrin determined by Singh and Kalra (1992) [11] in leaves of brinjal was 2.1 and 3.69 µg g^{-1} when applied at 50 and 100 g a.i ha^{-1} which is similar to the current findings. Initial deposit of 1.99 µg g^{-1} was recorded by Abdullah *et al.* (2001) [1] in soyabean at 50 g a.i ha^{-1}.

There was decline in residue with lapse of time in both the doses. About 70 per cent of cypermethrin residues got dissipated in one week after application in both the doses. Similar results were reported by Jyot *et al.* (2013) [8] in green chillies where nearly 70 per cent of the residue dissipated in one week after application. Cypermethrin reached BDL on 30 days after application in both the doses. Nahar *et al.* (2012) recorded decline of cypermethrin residue to BDL after 20 days of application in double the recommended dose in tomato fruits.

The rate of dissipation followed the first order kinetics with half life of 4.27 and 4.62 days at 50 and 100 g a.i ha^{-1}, respectively. This is in accordance with the earlier reports of Nahar *et al.* (2012) that the dissipation of cypermethrin in tomato samples after pesticide application followed first order kinetics in recommended and double the recommended doses. Cypermethrin residues recorded half life of 5.2 days in okra, 4.6 days in tomato, 8.36 days in chickpea and 9.49 days in tomato fruits (Deen *et al*., 2009; Gupta *et al*., 2010; Samriti *et al*., 2011; Cherukuri *et al*., 2015) [5, 6, 9, 4].

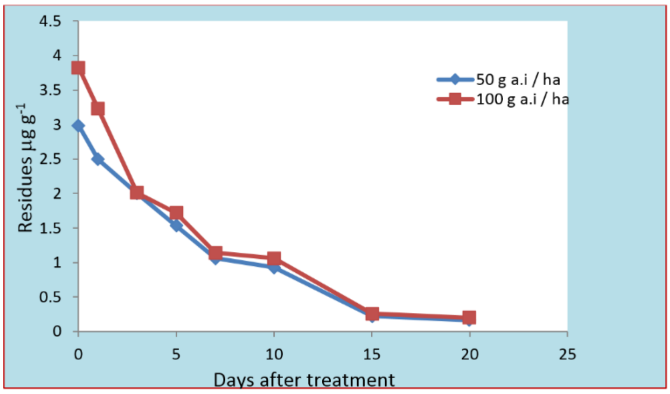

Fig 1: Dissipation pattern of cypermethrin in curryleaf sample

Table 1: Persistence and dissipation of cypermethrin 10 EC (50 g a.i ha^{-1}) residues on curryleaf

Days after application	Cypermethrin 50 g a.i ha^{-1} Residue µg g^{-1}			Mean*±SD	Dissipation%
	R1	R2	R3		
Control	ND	ND	ND	ND	ND
0	2.87	3.08	3.02	2.99±0.11	-
1	2.08	2.62	2.76	2.49±0.36	16.72
3	2.06	1.92	2.02	2.00±0.07	47.78
5	1.54	1.56	1.48	1.53±0.04	60.05
7	1.09	1.02	1.07	1.06±0.04	72.32
10	0.96	0.89	0.94	0.93±0.04	75.71
15	0.23	0.21	0.22	0.22±0.01	94.25
20	0.16	0.15	0.16	0.16±0.01	95.82
30	BDL	BDL	BDL	BDL	100

* - Mean of three replications
ND - Not detected
BDL - Below detectable level

Table 2: Persistence and dissipation of cypermethrin 10 EC (100 g a.i ha^{-1}) residues on curryleaf

Days after application	Cypermethrin 100 g a.i ha^{-1} Residue µg g^{-1}			Mean*±SD	Dissipation%
	R1	R2	R3		
control	ND	ND	ND	ND	ND
0	3.87	3.94	3.68	3.83±0.13	-
1	3.33	3.10	3.26	3.23±0.12	15.66
3	2.07	1.93	2.03	2.01±0.07	32.77
5	1.77	1.65	1.74	1.72±0.06	42.47
7	1.09	1.17	1.15	1.14±0.04	61.87
10	1.09	1.02	1.07	1.06±0.04	64.54
15	0.27	0.25	0.26	0.26±0.01	91.30
20	0.21	0.19	0.20	0.20±0.01	93.31
30	BDL	BDL	BDL	BDL	100

* - Mean of three replications
ND - Not detected
BDL - Below detectable level

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