Dissipation Kinetics of Deltamethrin 10 EC in Capsicum (*Capsicum Annum var. Frutescens*) and Cropped Soil Under Field and Polyhouse Conditions

Guru P.N.
CIPHET: Central Institute of Post-Harvest Engineering and Technology
PATIL C.S.
Mahatma Phule Krishi Vidyapeeth
DEORE B.V.
Mahatma Phule Krishi Vidyapeeth
Mahesh Kumar Samota (mahesh.iari1@gmail.com)
CIPHET: Central Institute of Post-Harvest Engineering and Technology https://orcid.org/0000-0003-3901-3290

Research Article

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Abstract

The dissipation behavior of deltamethrin 10 EC (Decis®) in capsicum (*Capsicum annuum* var. frutescens) grown in polyhouse and open field was investigated. Two sprays of deltamethrin 10 EC @ 15 and 30g a.i./ha (150 ml/ha -recommended and 300 ml/ha- double the recommended dose), respectively were applied at 10 days intervals, initiating at button sized fruit stage. Fruit samples were collected at 0, 1, 3, 5, 7, 10, 15, 20, 25 days after second spray and the soil samples were collected at final harvest for residue analyses. Modified QuEChERS multi-residue analysis showed initial residues of 0.976 and 1.161 mg/kg recorded at 15 and 30 g a.i./ha doses, respectively in polyhouse. The residues persisted upto 7th and 10th day, reaching below quantification limit (BQL) by 10th and 15th day, respectively at both the doses. In open field, the initial deposits recorded were 0.607 and 1.924 mg/kg reaching BQL on 5th and 7th day, respectively @ 15 and 30 g.a.i./ha. The comparative results showed that half life (*syn*. Dissipation time 50- DT_{50}) for deltamethrin in capsicum fruits varied from 2.05–2.45 days in polyhouse. The corresponding values in open field were 1.51–1.86 days suggesting that dissipation of deltamethrin residues took longer time in controlled conditions than open field. The soil samples analysed at harvest contained no residues. The insecticide degradation followed the first order kinetics. Since, the insecticide is not recommended under capsicum in India; the data may help to fix maximum residue limit (MRL) and pre harvest interval (PHI).

Introduction

In India, *Capsicum annuum* L. var. frutescens, commonly called as Sweet Pepper, Bell Pepper or Shimla Mirchi (Solanaceae) is one of the leading vegetables grown under protected conditions and is mainly cultivated for export purpose. In India, capsicum is grown in *kharif*, *rabi* (Jharkand, Karnataka, Maharashtra and Tamil Nadu) and summer (hills of Himachal Pradesh, Uttar Pradesh, Jammu and Kashmir and Nilgiri hills) as field crop. Under controlled conditions, the crop achieved early and heavy yields, higher returns to the farmers (Singh et al., 2004). The support of Government of India through subsidies for construction and cultivation of capsicum in polyhouses encouraged the farmers to take up cultivation under controlled conditions (Anonymous, 2005). Sucking insects like thrips and mites have become severe and hard to manage on capsicum (Reddy et al., 2006). In handling this pest severity, the farmers rely only on insecticides. Farmers adopted higher doses of agrochemicals to get better yields. This led to accumulation of chemical residues in crops and the ecosystems.

The dissipation of chemicals in crop ecosystem is mainly influenced by weather factors like temperature, humidity, light intensity and rainfall (Sharma et al., 2012; Rahman et al., 2020). However, capsicum is grown in field and under controlled conditions. Under controlled conditions, the weather parameters were regulated to certain extent leading to slower dissipation rate. This leads to residue retention for longer time compared to open field. Slower degradation of applied insecticides in polyhouse was supported (Pathipati et al., 2015; Buddidathi et al., 2016) as they found that the dissipation rates of chlorantraniliprole, flubendamide, spiromesifen, thiamethaxam and spinosad was slower and the insecticides persisted for longer time in capsicum grown under polyhouse than in field-grown crop.

Since, it was found that deltamethrin was commonly used (Dhore, 2016; Sali, 2016) on capsicum in the region; the present study was conducted to analyse degradation variations of deltamethrin in polyhouse and field grown capsicum. Deltamethrin is a pyrethroid ester insecticide, found effective against sucking insect pests on glasshouse cucumbers, tomatoes, peppers and ornamentals (Baptista et al., 2008) besides managing numerous insect pests of field crops with low application rates (Deo et al., 1991). Deltamethrin is not recommended or label claimed in capsicum under recommended insecticides list of Central Insecticide Board and Registration Committee (CIB and RC) of the Government of India. Even then, farmers are applying deltamethrin frequently. Although, referred ADI for deltamethrin is 0.1 mg/kg under EU (EU, 2017) but, MRLs for deltamethrin was not listed by FSSAI (Food Safety Standards Authority of India) and CAC (Codex Alimentarius Commission) in capsicum. The need for fixing safety standards for harvest and consumption of capsicum under Indian conditions is urgently required. The objectives of this study were to evaluate the behavior of deltamethrin residues applied as different formulations in a capsicum crop and in cropped soil.

Materials And Methods

Field Experiment

The trials were laid out in randomized block design (RBD) with three replications. The crop was grown under two conditions *i.e.*, polyhouse and field conditions during *kharif*. The capsicum var. *Bachata* was cultivated by following standard package of practices except plant protection measures (Anonymous, 2017).

Table 1
Weather conditions during the period of experimentation

Weather parameters	Readings
Minimum temperature	22.21° C
Maximum temperature	33.07° C
Minimum relative humidity	49.41 %
Maximum relative humidity	72.77 %
Rainfall	123.70 mm
Source: Department of Irrigation water management, MPKV, Rahuri, India	

The experiment comprised of three treatments i.e. Recommended dose (15g a.i./ha), Double the recommended dose (30g a.i./ha) and Untreated control (Water spray). Two foliar sprays of deltamethrin 10 EC were applied at 10 days interval initiating the first spray at fruit initiation (button-sized) stage. Quantity of spray fluid required per plot was calculated after spraying control plot with water.

Residues Analysis

Laboratory studies on dissipation of insecticide residues were carried out at the Pesticide Residue Laboratory, Mahatma Phule Krishi Vidyapeeth, Rahuri (Maharashtra), India.

Chemicals and reagents

The Certified Reference Material (CRM) of deltamethrin with purity of 99.9 % was obtained from Sigma Aldrich and commercial formulation (Decis®) was purchased from the local market. HPLC grade ethyl acetate was obtained from M/s. Avantor Performance Material India Limited, Thane. Primary-Secondary Amine and sodium sulphate anhydrous were procured from Agilent Technology, Bangalore and S.D. Fine-Chem Ltd., Mumbai, respectively. Working standards were prepared by dissolving reference standards in ethyl acetate.

Standard preparation

An accurately weighed 10 mg of analytical grade insecticide was dissolved in 10 ml volumetric flask using suitable solvent to prepare the standard stock solution of 1000 mg/kg. Standard stock solution was further diluted to obtain intermediate lower concentration of 100 and 10 mg/kg. They were stored in a refrigerator at -40°C. From intermediate standards, working standards were prepared by suitably diluting the stock solution in n-hexane and used as standard check in analysis, linearity and recovery studies.

Method validation

Prior to analysis, linearity of deltamethrin was established on GC-ECD. Accuracy and precision of the method was determined by per cent mean recovery and per cent relative standard deviation (RSD). The limit of detection (LOD) was determined by considering a signal-to-noise ratio of three with reference to the background noise obtained for the blank sample. The limits of quantification (LOQ) is a response that could be quantified with RSD lower than 20 per cent.

Recovery analysis

Capsicum fruit sample (10 g) was taken in 50 ml centrifuge tubes, each in triplicates and each sample was spiked with deltamethrin at the required fortification levels *i.e.* LOQ, 5 × LOQ and 10× LOQ, adding an appropriate volume of working standard

of 10 mg/kg. The extraction and clean-up was performed using methodology as described here under. Flow diagram for sampling, extraction, cleanup, and analysis of Deltamethrin in capsicum and cropped soil showed in Fig. 1. The per cent recovery was calculated using the formula.

$$Percent\ Reccovery = \frac{Quantity\ of\ insecticide\ recovered}{Quantity\ of\ insecticide\ added} \times 100$$

Soil analysis

One kg of representative soil sample was collected from each plot at harvest. In lab, 20 g of air dried sample was taken in 100 ml beaker. To soil, 0.5 ml of ammonia solution was added, mixed thoroughly and kept for half an hour till the odour stopped. Then 0.5 g each of fluorosil and activated charcoal and 10 g of anhydrous sodium sulphate were added and uniform mixing was ensured.

Preparation of column: A cotton plug was put at the bottom of the clean and dried glass column (600 mm × 22mm i.d.), over which 1 cm layer of anhydrous sodium sulphate was laid. After tapping gently for ten times, sample mixture was transferred into column and tapped again to ensure compact packing of the column. A layer of anhydrous sodium sulphate was added above the soil mixture. The column was eluted with 125 ml solution of hexane: acetone (9:1 v/v). The elute was concentrated to near-dryness on rotary vacuum flash evaporator by putting one drop of mineral oil followed by gas manifold evaporator. The final volume was made to 2 ml in n-hexane or ethyl acetate for GC-analysis

Residue determination

Sample Collection

Medium sized, marketable capsicum fruit samples (1 kg) were collected from each treatment plots separately, at regular time intervals of 0 (2 h after spraying), 1, 3, 5, 7, 10 and 15 days after the second spray. The collected samples (capsicum fruits) were transferred immediately to the laboratory in an ice box and were immediately processed. The soil samples were collected after crop harvest.

Extraction and Clean-up

The samples were extracted and cleaned up using modified QuEChERS method (Quick, Easy, Cheap, Effective, Rugged and Safe (Sharma, 2013). The entire laboratory sample (1 kg capsicum fruits) was macerated thoroughly in a grinder and approximately 10 g homogenized sample was weighed in a 50 ml polypropylene tube. The tube was kept in deep freezer for 10 min. Homogenised samples were extracted with 10 ml ethyl acetate in 10 g anhydrous Na_2SO_4 and centrifuged at 3500 rpm for 5 min. Two ml supernatant was transferred to 15 ml tube containing 50 mg PSA. The content was vortexed for 30 s and then cetrifuged at 2500 rpm for 2 min. The supernatant was filtered through 0.2 µ PTFE filter and subjected to GC analysis.

GC Conditions

The residues of deltamethrin were estimated on gas chromatograph (Shimadzu 2010) equipped with electron capture detector (ECD). The analysis was performed on capillary column (DB-5, 30 m length, 0.25 mm internal diameter and 0.25µm film thickness) with oven temperature at 300°C. The injector and detector temperatures were maintained at 290 and 310°C, respectively. The quantitative analysis of deltamethrin residues was performed by comparing peak area of standard with unknown samples run under identical conditions.

Statistical Analysis

The analyses of data sets were carried out in the Microsoft Excel-2013. The mean residues, standard deviation, regression equation, R^2 and half life values were calculated and presented.

Results

Method validation

Before analyzing the actual samples, validation parameters *viz.*, linearity, Limit of Detection (LOD) and Limit of Quantification (LOQ), accuracy and precision were determined to confirm the suitability of analytical method employed, since it is an integral part of any good analytical procedure. Fairly good response was observed by injecting 0.025 mg/kg of deltamethrin standard, finalized as LOD and 0.05 mg/kg was fixed as LOQ under assessed conditions, the peak of deltamethrin was observed at 22.319 min (retention time-RT) (Fig. 2). For the linearity studies, a graph of detector response versus concentration of working standard was plotted; correlation equation and coefficients were determined. Eight linear concentrations (0.025, 0.05, 0.10, 0.25, 0.40, 0.50, 0.80 and 1.00 mg/kg) of working standards of deltamethrin were injected in triplicate and the linearity line was drawn (Fig. 3). The response was linear over the range tested and R^2 value of 0.995 was recorded, which meets the standard criteria (*i.e.*, 0.990 to 0.999) (SANTE/11813/2017).

Recovery analysis

Recovery studies were performed using capsicum fruits and soil from control plot fortified with three concentrations of deltamethrin *i.e.*, 0.05 mg/kg (LOQ), 0.25 mg/kg (5× LOQ) and 0.50 mg/kg (10×LOQ). The extraction and clean-up was performed as described earlier. The recovery percentages are presented in Table 2. Recovery for deltamethrin ranged between 94.80 to 96.00 per cent in fruits and 90.20 to 97.88 per cent in soil that revealed acceptable recovery range of 70–120 per cent (SANTE/11813/2017). In addition, repeatability (spiking by other person) and reproducibility (spiking for more than two times) tests were also conducted to assess the possible deviation during spikes at two different conditions by individuals.

Table 2
Recovery of deltamethrin in capsicum and cropped soil

Substrate	Fortified level	Per cent recovery				SD	RSD
	(mg/kg)	R1	R2	R3	Mean		
Capsicum fruits	0.05	97.45	94.67	95.89	96.00	1.39	1.45
	0.25	91.67	92.27	90.45	94.80	6.49	6.85
	0.50	91.30	96.69	98.84	95.61	3.88	4.06
Soil	0.05	97.10	87.36	86.14	90.20	6.01	5.99
	0.25	91.97	90.59	91.08	97.88	5.12	5.23
	0.50	93.33	94.44	93.53	93.76	0.59	0.56

SD- Standard deviation, RSD- Relative standard deviation

Residues in fruits

Residues of deltamethrin (mg/kg) on capsicum fruits at different time intervals after the application of the formulation of deltamethrin 10EC @ 150 ml (15 g a.i./ha) and 300 ml (30 g a.i./ha) are presented in Tables 3 and 4 and Fig. 5. In polyhouse, the mean initial deposits of deltamethrin (0th day) were 0.976 mg/kg and 1.161 mg/kg at recommended and double the recommended dose, respectively. Residues dissipated to a maximum of 92.19 and 94.41 per cent after 7th and 10th day, respectively, in recommended and double the recommended dose. Subsequently the residues were recorded below quantification limit (BQL) on 10th and 15th day of application with the half life (DT_{50}) of 2.05 and 2.45 days, respectively.

Table 3
Dissipation of deltamethrin in/on capsicum fruits and soil (grown in polyhouse)

Sampling days after second application	Control	Residues (mg/kg)									
		Deltamethrin @ 15 gm a.i. ha^{-1}					Deltamethrin @ 30 gm a.i. ha^{-1}				
		R1	R2	R3	Mean	Dissipation (%)	R1	R2	R3	Mean	Dissipation (%)
0	ND	1.004	0.907	1.019	0.976 (± 0.061)	-	1.132	1.146	1.205	1.161 (± 0.039)	-
1	ND	0.604	0.630	0.592	0.609 (± 0.019)	37.65	0.787	0.734	0.729	0.750 (± 0.032)	35.40
3	ND	0.481	0.429	0.487	0.466 (± 0.032)	52.29	0.501	0.495	0.497	0.498 (± 0.003)	57.14
5	ND	0.228	0.200	0.269	0.232 (± 0.035)	76.19	0.296	0.291	0.296	0.294 (± 0.003)	74.66
7	ND	0.068	0.095	0.067	0.076 (± 0.016)	92.19	0.146	0.146	0.142	0.145 (± 0.002)	87.54
10	ND	BQL	BQL	BQL	BQL	≈ 100.00	0.064	0.067	0.063	0.065 (± 0.002)	94.41
15	ND	-	-	-	-	-	BQL	BQL	BQL	BQL	≈ 100.00
Soil	ND	BQL	BQL	BQL	BQL	BQL	BQL	BQL	BQL	BQL	BQL
DT_{50} (days)	-	2.05					2.45				
R^2	-	0.953					0.995				

BQL - Below Quantification Limit i.e., 0.05 mg /kg

ND- Not detected, DT_{50}- Dissipation Time 50 (*syn.* Half-life), Figures in the parentheses are standard deviation values

Table 4
Dissipation of deltamethrin in/on capsicum fruits and soil (grown in open field)

Sampling days after second application	Control	Residues (mg/kg)									
		Deltamethrin @ 15 gm a.i. ha^{-1}					Deltamethrin @ 30 gm a.i. ha^{-1}				
		R1	R2	R3	Mean	Dissipation (%)	R1	R2	R3	Mean	Dissipation (%)
0	ND	0.634	0.611	0.576	0.607 (± 0.029)	-	1.871	2.032	1.869	1.924 (± 0.094)	-
1	ND	0.383	0.408	0.371	0.387 (± 0.019)	36.23	1.452	1.325	1.246	1.341 (± 0.104)	30.32
3	ND	0.186	0.134	0.140	0.153 (± 0.028)	74.77	0.775	0.642	0.792	0.736 (± 0.082)	61.73
5	ND	BQL	BQL	BQL	BQL	≈ 100.00	0.296	0.291	0.281	0.289 (± 0.008)	84.96
7	ND	-	-	-	-	-	BQL	BQL	BQL	BQL	≈ 100.00
Soil	ND	BQL	BQL	BQL	BQL	BQL	BQL	BQL	BQL	BQL	BQL
DT_{50} (days)	-	1.51					1.86				
R^2	-	0.999					0.989				
BQL - Below Quantification Limit i.e., 0.05 mg/kg											
ND- Not detected, DT_{50}- Dissipation Time 50, Figures in the parentheses are standard deviation values											

As regards, the open field conditions, the results indicated that the mean initial deposits recorded were 0.607 and 1.924 mg/kg, respectively. At recommended dose, the initial deposit got dissipated to 0.387 (36.23 %) and 0.153 (74.77 %) mg/kg on 1st and 3rd day, respectively, which reached BQL (< 0.05 mg/kg) on the 5th day. In double dose, the initial deposits were dissipated to 1.341 (30.32 %), 0.736 (61.73 %) and 0.289 mg/kg (84.96 %) on the 1st, 3rd and 5th day, respectively, which reached BQL (< 0.05 mg/kg) on the 7th day. The DT_{50} calculated for deltamethrin @ 15 and 30 gm a.i./ha were 1.51 and 1.86 days, respectively.

Discussion

The rate of dissipation of pesticide varies with the nature of the pesticide, dosage, formulation and the application methods (Montemurro et al., 2002), crop variety (Fan et al., 2013; Wang et al., 2014), weather conditions (Garau et al., 2002) and frequency of pesticide applications. All the above factors influenced the residues of pesticide. Though, in the current study, the commercial formulation, dosage and application method was similar, the residue levels still varied in polyhouse and field conditions. This may be because, in polyhouse, the volatilization and wind drift losses of pesticides are much less in plant and soil. Since most of the pesticides are UV-degradable, in controlled condition there is likelihood that pesticides persist for a longer duration in absence of UV (IIHR, 2011) compared to open field conditions. Longer time for insecticide degradation on capsicum fruits in polyhouse condition compared to open field was also reported in other insecticides like, chlorantraniliprole, triazophos, spinosad, flubendamide, spiromesifen and thiamethaxam, chlorpyriphos, ethion, triazophos and profenophos (Kavitha et al., 2016). In a multi-location supervised field trials conducted on chillies (Pandher et al., 2012), the dissipation kinetics of deltamethrin 10 EC @ 17.5 and 35 g a.i./ha showed that the initial deposits of 0.21–0.49 mg/kg @ 17.5 g a.i./ha and 0.37–0.69 mg/kg @ 35 g a.i./ha

reached BQL in 5 and 7 days. Similarly, residues of deltamethrin reached BQL of 0.01 mg/kg on the 7th day on tomato (Premchand et al., 1999) and 10 days on brinjal (Sen and Choudhary, 1999).

It was obvious that the initial deposits were directly proportional to the dose/ concentration of the sprayed chemical means the deposits of double the recommended dose was higher than that of the recommended dose. However, in the present study slight variation in initial deposits on capsicum fruits was recorded in polyhouse and open field. This might be due to the application method, and other abiotic factors. Dissipation studies of deltamethrin were also reported on tomato (Singh et al., 1989) and found initial residues of 0.07–0.09 mg/kg in single and 0.08–0.23 mg/kg in double the recommended dose. In cauliflower (Singh et al., 1990), maximum initial deposits recorded were 0.12 to 0.32 mg/kg, where as in cabbage (Singh et al., 1992) and brinjal (Das and Mukherjee, 2012) they recorded initial deposits of 0.08 to 0.30 mg/kg and 0.430 to 0.900 mg/kg, respectively.

The soil samples analysed at harvest of the capsicum fruits (40 days after 2nd spray) recorded no residues at both the growing conditions i.e., polyhouse and open field neither @ 150 ml (15 g a.i./ha) nor 300 ml (30 g a.i./ha) dose. This pesticide degradation in soil may depend on the organic matter content of the soil, soil pH or microbial fauna (Das and Mukherjee, 2012). Light is also a reason for pesticide degradation (photo-degradation), light also influences the soil characteristics (Gavrilescu, 2005). Studies also revealed that the higher moisture content of soil and soil microorganisms speed up the process of flubendamide degradation in soil (Das and Mukherjee, 2012; Katagi, 2004; Narenderan et al., 2020). In general, while applying the insecticides, insecticide release from the sprayer, drift of the chemical due to application procedures and wind was common, it may be the reason for insecticide to reach soil. Since, the capsicum vines are robustly grown and the foliage coverage was good, it is that likely higher absorption of insecticides on plant occurred surface than the accumulation in soil.

Conclusion

Capsicum fruits are increasingly gaining popularity because of use in the preparations of culinary dishes like pizza, burger, etc. In this study, deltamethrin residues persisted longer in polyhouse condition compared to the open field. Minimum 5 days in polyhouse and 3 days in open field was the time required for the initial residues of deltamethrin to reach below quantification limit. This time duration, may treat as PHI for harvesting the residue-free capsicum fruits, which avoid the insecticide poisoning. Since, there is no specific recommendation of deltamethrin against insect pests of capsicum, the present data will help to fix the Maximum Residual Limit (MRL) and Pre-Harvest Interval (PHI) for deltamethrin following application at recommended dose of 150 ml/ha (15 g.a.i./ha).

Declarations

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work

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Figures

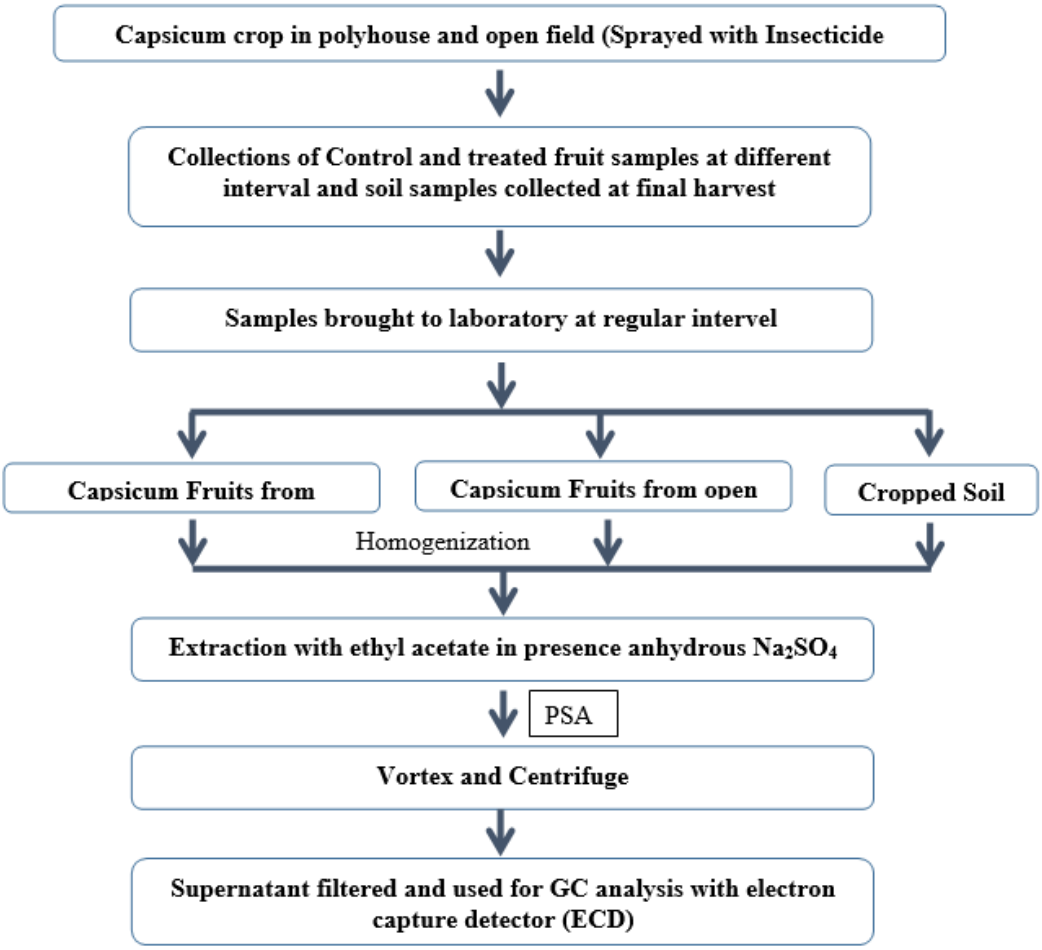

Figure 1

Flow diagram for sampling, extraction, cleanup, and analysis of Deltamethrin in capsicum and cropped soil

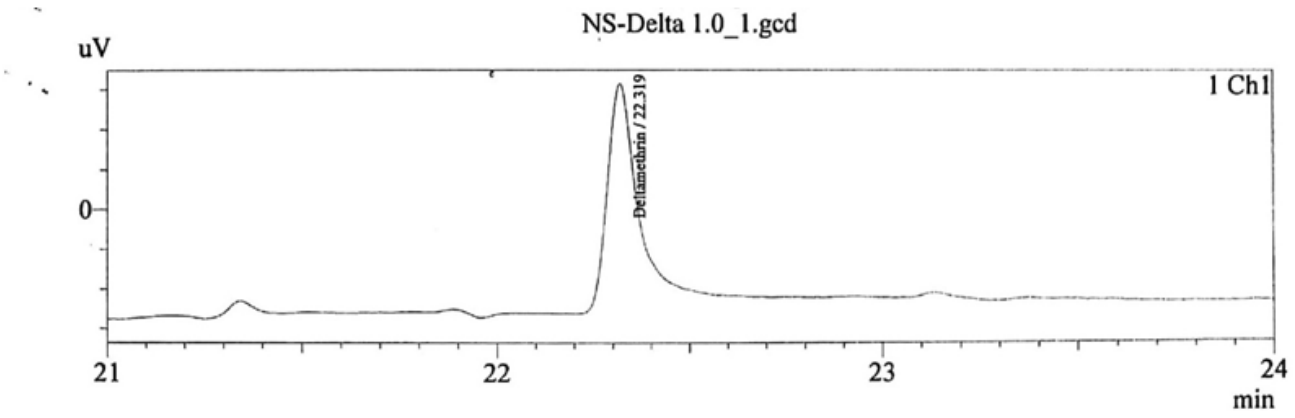

Figure 2

GC-ECD chromatogram showing deltamethrin retention time at 22.319 min.

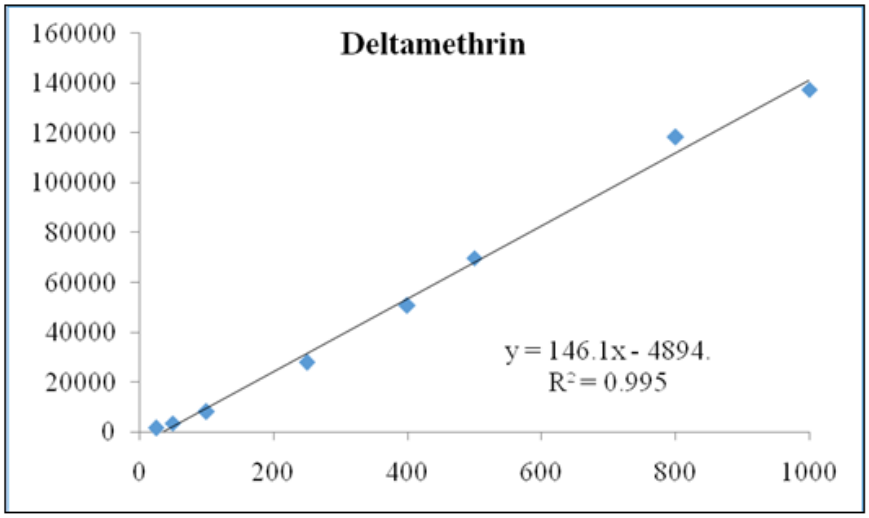

Figure 3

Linearity graph of deltamethrin

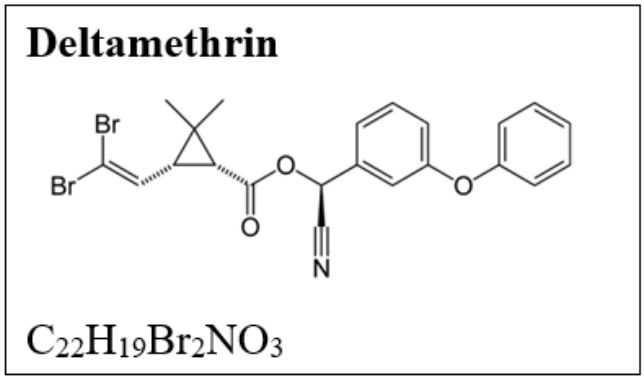

Figure 4

Structure of deltamethrin

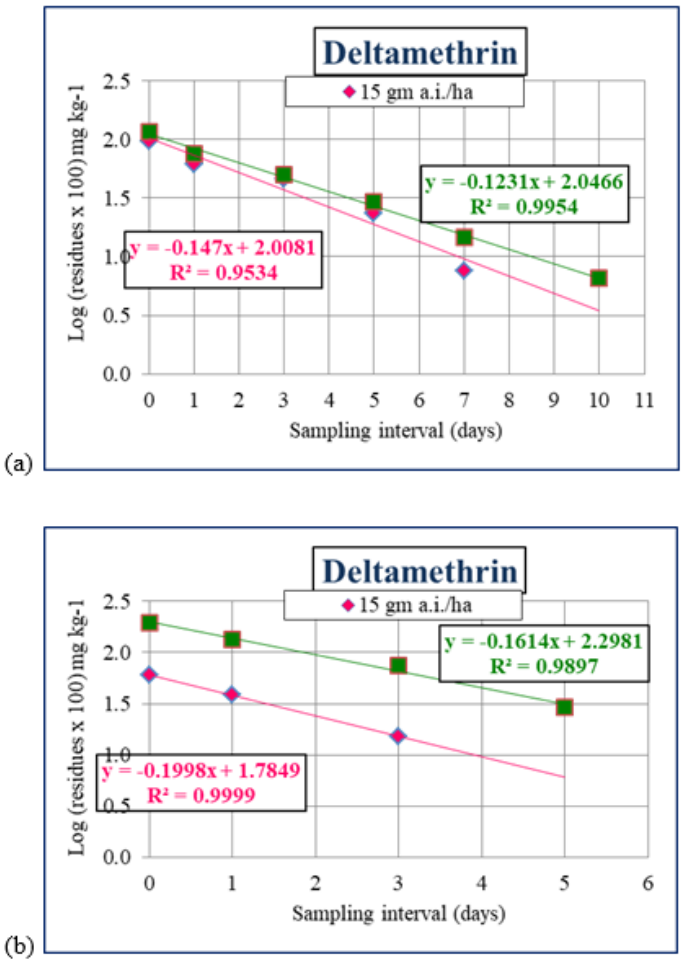

Figure 5

Semi-log graph showing dissipation pattern of deltamethrin in capsicum fruits grown in (a) polyhouse (b) open field

Supplementary Files

This is a list of supplementary files associated with this preprint. Click to download.

- Graphicalabstract.png