

Original Research Article

Persistence and Dissipation Kinetics of λ -cyhalothrin in Okra Fruits and Soil

ABSTRACT

Understanding pesticide residue levels in food items and soil is crucial for ensuring food safety. In this context, a study on persistence and dissipation kinetics of λ -cyhalothrin residues in okra fruits and soil was conducted for two consecutive years. λ -cyhalothrin was applied in okra at the recommended dose (15 g a.i. ha⁻¹) and double dose (30 g a.i. ha⁻¹). Okra fruit and soil samples were collected at different intervals after second spray and the residues were estimated using GC-MS/MS. The initial residue level in okra fruits was higher at double dose and declined gradually falling below the limit of quantification (0.01 mg kg⁻¹) within 7 to 10 days of application. Dissipation followed first-order kinetics, with half-life of 2.17 to 2.43 days. In soil, the initial residues dropped below the limit of quantification within 3 to 5 days of application. Since the initial residue levels in both okra fruits and soil were below the corresponding EU-MRL (0.2 mg kg⁻¹), λ -cyhalothrin can be safely used in okra cultivation.

Keywords: Okra, persistence, dissipation, λ -cyhalothrin, half-life, residues, GC-MS/MS

1. INTRODUCTION

Vegetables are an important part of a healthy eating pattern and are excellent sources of many nutrients. Among these, okra, *Abelmoschus esculentus* L. (Moench), is an important crop grown in tropical and subtropical regions of the world. Okra pods are rich sources of essential minerals such as potassium, calcium, phosphorus and magnesium, while being very low in cholesterol and saturated fats [1], which are very essential for human health. It possesses significant export potential and is cultivated widely in gardens or home yards across tropical and subtropical regions worldwide [2]. In India okra is grown in 546 thousand hectare area with production of 6700 thousand MT and 12.27 tonnes productivity [3]. However, infestation of insect pest at various stages of crop growth is one of the major constraints in the production of this crop. More than 72 species of insect pests and mites have been documented to infest the okra crop [4]. To combat the problem of insect pests, farmers primarily utilize chemical pesticides, and they do so at far higher doses than recommended [5].

λ -cyhalothrin (S)- α -cyano-3-phenoxy benzyl-(Z)-(1R,3 R)-3-(2-chloro-3,3,3-trifluoro prop-1-enyl)-2,2-dimethyl cyclopropane carboxylate is a new synthetic pyrethroid. Owing to its relatively low toxicity and high knock down effect, this insecticide is extensively used in agriculture [6]. Because of broad spectrum of activity, λ -cyhalothrin is effective against a variety of insect pests such as aphids, caterpillars, beetles, leafhoppers etc. [7,8]. However, use of pesticides presents substantial risks to both the environment and public health [9,10]. Pesticides have been documented to produce toxic effects in humans, including short-term symptoms like headaches and nausea, as well as chronic issues such as cancer, reproductive harm, and endocrine disruption [11,12,13]. On the other hand, pesticide degradation impacts soil microbial diversity, along with metabolic processes and enzyme activity [14,15]. Okra being a fast growing crop is harvested at very short interval (3-4 days) and this practice increases the chance of fruits having high load of residues which can pose health hazards to the consumers [16]. Therefore, before recommending any pesticide for field application, its dissipation pattern in agricultural produce as well soil is crucial. In light of this, the present study was conducted to investigate the persistence and dissipation kinetics λ -cyhalothrin in okra fruits and soil.

2. MATERIALS AND METHODS

The present study was conducted at Chaudhary Charan Singh Haryana Agricultural University, Hisar (Haryana), India over two consecutive years, 2019 and 2020.

2.1. Field Experiment

The field experiment was conducted in a randomized block design with a plot size of 5 × 4 m. Okra (variety *Hisar Naveen*) was grown during both the seasons following recommended agronomic practices (Anonymous, 2013). λ -cyhalothrin 5 EC was applied in okra at recommended dose (15 g a.i. ha⁻¹) and double the recommended dose (30 g a.i. ha⁻¹) twice, first at fruit initiation, followed by a second spray at 15 days interval. One treatment was kept as control in which no insecticide was

applied. Three replications were maintained for each treatment. The experimental site experiences a semi-arid climate, and the meteorological parameters during the spraying period are given in Table 1.

Table 1. Meteorological parameters during the study period

Year	Period of spraying	Average temperature (°C)		Average relative humidity (%)		Rainfall (mm)
		Maximum	Minimum	Morning	Evening	
2019	September-	33.1	20.7	85.9	45.8	0.2
	October					
2020	September-	36.7	22.5	85.2	41.9	0.3
	October					

2.2. Sampling and Processing

For dissipation study, okra fruit samples (500 g) were collected in polybags from randomly selected plants in each treatment across the three replications 0 (2 h), 1, 3, 5, 7, 10, and 15 day(s) after the second spray. On the similar pattern, the soil samples (1 kg) were also drawn from the soil (up to 15 cm depth) under the okra plants in the treated plots. The soil samples were then placed on filter paper and allowed to shade dry at room temperature in the laboratory. The air dried soil samples were desegregated manually, passed through sieve and mixed thoroughly to achieve homogeneity. Okra fruit samples collected from field were brought to the laboratory immediately and processed on the same day by liquid-liquid partitioning method. For processing of soil samples, method prescribed by Kumari [17] was employed. The residues of λ -cyhalothrin were estimated by using GC-MS/MS.

2.3. Extraction and Clean-up of Residues

For extraction of residues in okra fruits, a representative sample of 20 g chopped and macerated fruits were taken in a flask and 100 ml of acetone was added to it. Then such flasks were shaken for one hour on the mechanical shaker and extract was filtered through Buchner funnel. The filtered extract was transferred to 1 litre separatory funnel. A 450 ml of brine solution (10% NaCl) was added to it and partitioned twice, first with dichloromethane (100 and 50 ml) and then hexane (100 and 50 ml) by vigorous shaking for 5 minutes to remove the non-emulsifying impurities. Each time, organic phase was collected and passed through 2-3 cm pad of anhydrous sodium sulphate to remove the trace amount of moisture and pooled together. Then 0.3 mg activated charcoal was added to this extract (for absorbing coloured impurities present in the sample) and left for 4 hours. The extract was filtered using Whatman no.44 filter paper. The organic layer was concentrated near to dryness on rotary vacuum flask evaporator and final volume of 3 ml was made with n-hexane. Cleaning of the okra fruit extract was done by column chromatography. Glass column (60 cm 9 2.2 mm i.d.) was packed with adsorbent mixture of florisil and activated charcoal (3:0.3 w/w) in between two layers (1 cm) of anhydrous sodium sulphate. The column was pre-wetted with 40 ml hexane. The extract was transferred to the column and eluted with 100 ml solution of hexane: acetone (9:1 v/v). The eluate so obtained was concentrated near to complete dryness on rotary vacuum evaporator and reconstituted the final volume of 3 ml using n-hexane.

For extraction of residues from soil, an indicative 15 g sample of fully dried soil was taken in a 100 ml beaker. Then 0.5 ml of ammonia solution was added to it and kept undisturbed for about 1 h till ammonia smell fade away. Glass column was prepared by blocking with a cotton plug and then placing 1 cm of anhydrous Na_2SO_4 layer was over it. Gradually taped the column, after that adsorbent *i.e.*, florisil and activated charcoal (3:0.3 w/w) were completely mixed and permeated down the column with 1 cm anhydrous sodium sulphate film. Soil mixture was covered with 1 cm sodium sulphate film. A 100 ml solution of hexane: acetone (9:1 v/v) was used to elute the column. The eluate was concentrated near to dryness on rotary vacuum evaporator. Final volume of 3 ml was reconstituted using n hexane and transferred to 2 ml glass vials.

2.4. Residue Estimation

GC-MS/MS system based on chromatographic technique was used for estimation of λ -cyhalothrin residue in okra fruits and soil. The system was standardised preceding to estimation of insecticide

residue. GC analysis was performed by using software GCMS solution version 2.53 SU3. The column used was Rtx-5 (Length-30 m, film thickness-0.25 μm ,). Helium was used as carrier gas with flow rate of 21 ml min^{-1} . and injection volume was 1 μl . The retention time of λ -cyhalothrin recorded was 22.152 minutes. Limit of detection (LOD) and limit of quantification (LOQ) were 0.005 and 0.01 mg kg^{-1} , respectively (Table 2).

Table 2. Parameters of GC-MS/MS used for residue study

Parameters	Details
Software	GCMS solution version 2.53 SU3
Column	Rtx-5 MS -1(30m \times 0.32 mm 1D X 0.25 μm film thickness) of 5 per cent diphenyl + 95 per cent dimethyl polysiloxane
Temperature	
Oven	80° (2 min) \rightarrow 20°C min^{-1} \rightarrow 180 (0 min) \rightarrow 5°C min^{-1} \rightarrow 300°
Iron source temp.	250°C
Interface temp.	270°C
Rates of Gas flow	
Carrier gas	Helium
Via column	1-46 ml min^{-1}
Total flow	21 ml min^{-1}
Pressure	250 k pa, high
Split ratio	Split less mode
Limit of detection (LOD)	0.005 mg kg^{-1}
Limit of quantification (LOQ)	0.01 mg kg^{-1}
R _t for ready mix formulation	λ -cyhalothrin: 22.152

2.5. Linearity Study

A concentration of 0.01 to 1.00 ppm λ -cyhalothrin was used to plot the calibration curve and the peak area against each concentration was recorded (Table 3). The calibration curve of λ -cyhalothrin was constructed by plotting the concentration against the peak area. A linear relationship was found between the different concentrations and their respective areas (Fig. 1). The retention time (R_t) for λ -cyhalothrin was 22.152 minutes as shown in the typical chromatogram (Fig 2).

Table 3. Standard curve data for lambda-cyhalothrin

Concentration (mg kg ⁻¹)	Area
0.010	103543
0.025	247086
0.050	497372
0.100	999345
0.250	1977699
0.500	3863383
1.000	7906766

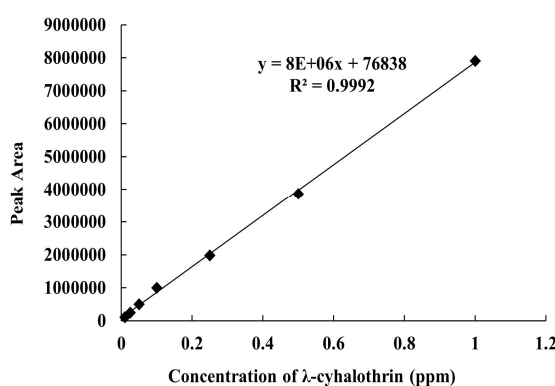


Fig. 1. Standard curve of λ-cyhalothrin on GC-MS/MS

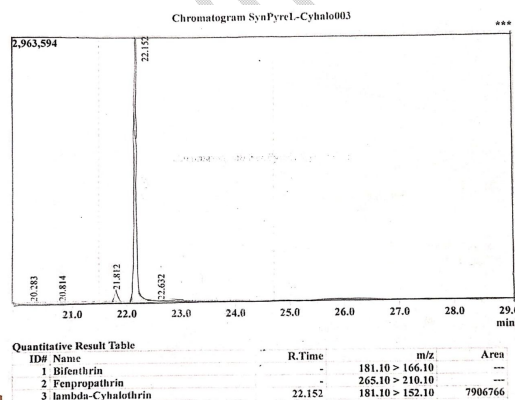


Fig. 2. Chromatogram of λ-cyhalothrin on GC-MS/MS

2.6. Method Validation

To check the validity and authenticity of the method used for estimation of λ-cyhalothrin residue in okra fruits and soil, a recovery experiment was conducted at different spiking levels. For this purpose, 20 g of crushed okra fruits and 15 g of soil were taken from the samples collected from the control plots and fortified with CRM (certified reference material) of λ-cyhalothrin at three different levels *i.e.*, 0.01, 0.05 and 0.10 mg kg⁻¹. The percentage recovery at each fortification level was estimated using the prescribed processing and analytical procedure.

2.7. Statistical Analysis

λ-cyhalothrin residues were calculated as under:

$$\text{Residue (mg kg}^{-1}\text{)} = (A_1 \times C \times I_1 \times F) / (A_2 \times W \times I_2)$$

Where, A₁ = Peak area of the sample, A₂ = Peak area of the standard, I₁ = Injected volume of standard (μl), I₂ = Injected volume of sample (μl), C = Concentration of standard solution (mg/l), F = Final volume of the sample (ml) and W = Weight of the sample (kg).

λ-cyhalothrin residues data thus obtained from the field experiments were subjected to first-order dissipation kinetics equation:

$$C_t = C_0 e^{-kt}$$

where, C_t is the pesticide concentration (mg kg⁻¹) at time t (day), e = base

C₀ is the apparent initial concentration (mg kg⁻¹), k is the dissipation rate constant [18].

The half-life of λ-cyhalothrin was calculated based on the formula given below [19,20]

$$\text{Half-life (t}_{1/2}\text{)} = \ln 2 / k$$

Where t_{1/2} = half-life of the residues, k = slope of the equation (b)

3. RESULTS AND DISCUSSION

3.1. Linearity Study

The retention time for λ -cyhalothrin was observed to be 22.152 minutes. However, in earlier studies, the retention time for λ -cyhalothrin was reported to be 6.99 minutes during the dissipation study on tomato using GC [21] and 37.33 minutes while conducting residue analysis of λ -cyhalothrin in pigeon pea on LC-MS/MS [22].

3.2. Method Validation

The average recovery of λ -cyhalothrin residues in okra fruit samples was 89.07, 90.20 and 94.92 per cent while in case of soil, it was 82.03, 94.97 and 96.97 per cent when samples were fortified at level of 0.01, 0.05 and 0.10 mg kg⁻¹, respectively (Table 4). Similarly, in another study 87 to 92 per cent and 97 to 101 per cent recovery of lambda-cyhalothrin was reported in tomato fruits and soil when fortified at the level of 0.25 mg kg⁻¹ and 0.50 μ g g⁻¹, respectively [6]. Taking into account the satisfactory recovery percentage (70-110%) and RSD (< 20), the method was considered suitable for the assessment of λ -cyhalothrin in okra and soil [23]. The limit of quantification (LOQ) was found to be 0.01 mg kg⁻¹.

Table 4. Recovery of λ -cyhalothrin in fortified okra fruit and soil samples

Fortification level (mg kg ⁻¹)	Okra fruits		Soil	
	Recovery (%)	RSD	Recovery (%)	RSD
	(Mean \pm SD)	(%)	(Mean \pm SD)	(%)
0.01	89.07 \pm 4.55	5.11	82.03 \pm 3.50	4.27
0.05	90.20 \pm 4.00	4.43	94.97 \pm 3.76	3.95
0.10	94.92 \pm 2.68	2.82	96.97 \pm 1.91	1.97

3.3. Dissipation of λ -cyhalothrin in Okra Fruits

In 2019, the average initial residues of λ -cyhalothrin okra fruits on 0 day (2 h) were 0.079 mg kg⁻¹ when applied at recommended dose (15 g a.i. ha⁻¹). The residues decreased to 0.042 mg kg⁻¹ within 24 hours of application dissipating by 46.84 per cent (Table 5). The residues further declined gradually and reached to 0.027 and 0.017 mg kg⁻¹ in 3 and 5 days of application showing degradation of 65.82 and 78.48 per cent, respectively. After 7 days of application, the residues in okra fruits were below limit of quantification (0.01 mg kg⁻¹). In case of double the recommended dose (30 g a.i. ha⁻¹), the initial residues in okra fruits were 0.162 mg kg⁻¹ which likewise declined gradually and reached 0.089, 0.058, 0.032 and 0.019 mg kg⁻¹ in 1, 3, 5 and 7 day(s) with a reduction of 45.06, 64.20, 80.25 and 88.27 per cent, respectively. Further, the residues were below limit of quantification (0.001 mg kg⁻¹) in fruit samples collected 10 days after spray.

The regression equation was obtained by plotting the graph of log [Residues (mg kg⁻¹) \times 10³] against time (in days) to study the degradation kinetics of residues of λ -cyhalothrin in okra fruits (Fig. 3). The negative value of correlation coefficient indicates that residues declined with lapse of time and followed first order kinetics. Half-life of λ -cyhalothrin was calculated to be 2.43 and 2.39 days with dissipation rate constant of 0.285 and 0.290 per day at recommended and double the recommended dose, respectively.

In 2020, the average initial deposits of 0.087 mg kg⁻¹ were detected in okra fruits samples collected on 0 day (2 h) from the plots sprayed with λ -cyhalothrin at recommended dose (Table 4). Within 24 hours of application, the residues reached to the level of 0.055 mg kg⁻¹ decreasing by 36.78 per cent. Thereafter, the residues of the insecticide declined at much faster rate and reached to the level of 0.029 and 0.017 mg kg⁻¹ in 3 and 5 days with dissipation of 66.67 and 80.46 per cent, respectively. The residues were below limit of quantification in the fruit samples collected 7 days after spray. In case of double the recommended dose, initial residues on 0 day (2 h) were 0.165 mg kg⁻¹ which decreased gradually and reached to the level of 0.107, 0.058, 0.038 and 0.020 mg kg⁻¹ in 1, 3, 5 and 7

days with dissipation of 35.15, 64.85, 76.97 and 87.88 per cent, respectively. However, the residues of λ -cyhalothrin were below limit of quantification in samples collected 10 days after spray. The residues of λ -cyhalothrin dissipated gradually following first order kinetics and reached to its half in 2.17 and 2.41 days at recommended and double the recommended dose, respectively (Fig. 4).

These results are in accordance with the earlier studies in which initial residues of λ -cyhalothrin 5% EC in brinjal were reported to be 1.31 mg kg^{-1} and which reached below detectable limit in 10 days when applied at the rate of $30 \text{ g a.i. ha}^{-1}$ [24]. In another study, initial residues of λ -cyhalothrin in okra fruits were reported to be 0.144 and 0.354 mg kg^{-1} at single ($15 \text{ g a.i. ha}^{-1}$) and double dose ($30 \text{ g a.i. ha}^{-1}$), respectively and reached below detectable limit (0.005 mg kg^{-1}) within 7 days of application [6]. Higher value of initial residues than estimated in present study was might be due to difference in agro climatic conditions prevailed during the respective season. Further, persistence and dissipation of the insecticide residues depend upon a variety of factors such as chemical, plant, soil and environment [25]. On the other hand, initial residues of λ -cyhalothrin (0.004%) in tomato fruits were reported to be 0.550 mg kg^{-1} which reached below detectable limit within 10 days of application with half-life and safety interval of 3.06 and 1.03 days, respectively [25].

3.4. Dissipation of λ -cyhalothrin in Soil

In 2019, average initial residues of λ -cyhalothrin in soil 0 day (2 h) were 0.037 mg kg^{-1} when applied at the recommended dose ($15 \text{ g a.i. ha}^{-1}$) (Table 6). Within 24 hours of application, the residues reached to the level of 0.018 mg kg^{-1} dissipating by 50.89 per cent while 3 days after spray, the residues fell below the limit of quantification (0.01 mg kg^{-1}). In case of double the recommended dose of λ -cyhalothrin, the average initial residues were 0.072 mg kg^{-1} which reached 0.026 mg kg^{-1} within 24 hours of application degrading by 64.19 per cent. Likewise, at double dose also, the residues reached below the limit of quantification within 3 days of application.

In 2020, the average initial residues of λ -cyhalothrin in soil on 0 day (2 h) were 0.046 when applied at recommended dose. The residues decreased gradually and reached 0.029 and 0.018 mg kg^{-1} in 1 and 3 day(s) dissipating by 36.50 and 60.58 per cent, respectively, and fell below the limit of quantification within 5 days of application. At double the recommended dose, the average initial residues of λ -cyhalothrin were 0.083 mg kg^{-1} which reached to the level of 0.051 and 0.021 mg kg^{-1} in 1 and 3 day(s) after application degrading by 38.80 and 74.40 per cent, respectively. Here also, the residues reached below the limit of quantification within 5 days of application. A negative correlation was observed between the time and residue degradation, with dissipation following first-order kinetics.

The present findings are in accordance with Chauhan et al. [6] who reported initial residues of λ -cyhalothrin in soil as 0.011 and 0.021 mg kg^{-1} and reached below detectable limit within 3 and 5 days at single and double dose, respectively. The study on dissipation of combination product *i.e.*, chlorantraniliprole 9.26% + λ -cyhalothrin 4.63% ZC in pigeon pea applying at recommended ($30 \text{ g a.i. ha}^{-1}$) and double the recommended dose ($60 \text{ g a.i. ha}^{-1}$) also indicated initial residues of λ -cyhalothrin as 0.604 and 1.884 mg kg^{-1} and reached below detectable limit (0.013 mg kg^{-1}) by 20 and 30 days of application, respectively. However, no residue of λ -cyhalothrin was detected in soil at initial stage in case of recommended dose while it was 0.124 mg kg^{-1} at double the recommended dose and reached below detectable limit (0.030 mg kg^{-1}) in 30 days of application [22].

Table 5. Dissipation of λ -cyhalothrin in okra fruits

Day(s) after spray	RD (15 g a.i. ha ⁻¹)		DRD (30 g a.i. ha ⁻¹)	
	Residue level (mg kg ⁻¹)	Dissipation	Residue level (mg kg ⁻¹)	Dissipation
	Mean \pm SD	(%)	Mean \pm SD	(%)
<u>2019</u>				
0 (2 h)	0.079 \pm 0.005	--	0.162 \pm 0.007	--
1	0.042 \pm 0.006	46.84	0.089 \pm 0.006	45.06
3	0.027 \pm 0.006	65.82	0.058 \pm 0.006	64.20
5	0.017 \pm 0.004	78.48	0.032 \pm 0.005	80.25
7	<LOQ	--	0.019 \pm 0.004	88.27
10	--	--	<LOQ	--
	Correlation coefficient (r) = -0.974		Correlation coefficient (r) = -0.991	
	Regression equation (y) = 1.826 + (-0.124)x		Regression equation (y) = 2.145 + (-0.126)x	
	Half life value (T _{1/2}) = 2.43 day		Half life value (T _{1/2}) = 2.39 days	
	Degradation constant (k) = 0.285		Degradation constant (k) = 0.290	
<u>2020</u>				
0 (2 h)	0.087 \pm 0.006	--	0.165 \pm 0.006	--
1	0.055 \pm 0.005	36.78	0.107 \pm 0.007	35.15
3	0.029 \pm 0.005	66.67	0.058 \pm 0.007	64.85
5	0.017 \pm 0.005	80.46	0.038 \pm 0.007	76.97
7	<LOQ	--	0.020 \pm 0.005	87.88
10	--	--	<LOQ	--
	Correlation coefficient (r) = -0.995		Correlation coefficient (r) = -0.966	
	Regression equation (y) = 1.907 + (-0.139)x		Regression equation (y) = 2.180 + (-0.125)x	
	Half life value (T _{1/2}) = 2.17 days		Half life value (T _{1/2}) = 2.41 days	
	Degradation constant (k) = 0.320		Degradation constant (k) = 0.288	

Mean of three replications; RD= Recommended dose; DRD = Double the recommended dose; LOQ = Limit of quantification (0.01 mg kg⁻¹)

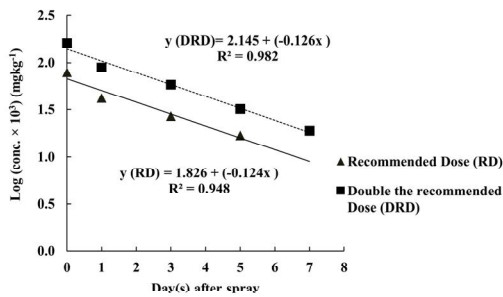


Fig. 3. Dissipation kinetics of λ -cyhalothrin in okra fruits in 2019

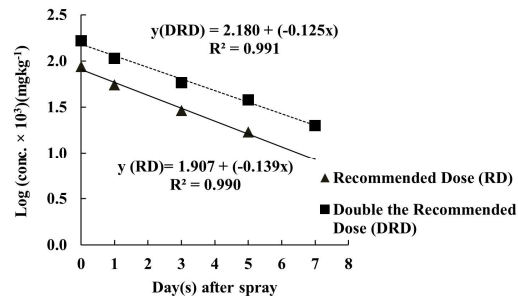


Fig. 4. Dissipation kinetics of λ -cyhalothrin in okra fruits in 2020

Table 6. Dissipation of λ -cyhalothrin in soil

Day(s) after spray	RD (15 g a.i. ha ⁻¹)		DRD (30 g a.i. ha ⁻¹)	
	Residue level (mg kg ⁻¹) Mean \pm SD	Dissipation (%)	Residue level (mg kg ⁻¹) Mean \pm SD	Dissipation (%)
<u>2019</u>				
0 (2 h)	0.037 \pm 0.009	--	0.072 \pm 0.008	--
1	0.018 \pm 0.005	50.89	0.026 \pm 0.007	64.19
3	<LOQ	--	<LOQ	--
<u>2020</u>				
0 (2 h)	0.046 \pm 0.009	--	0.083 \pm 0.009	--
1	0.029 \pm 0.007	36.50	0.051 \pm 0.007	38.80
3	0.018 \pm 0.005	60.58	0.021 \pm 0.006	74.40
5	<LOQ	--	<LOQ	--
		Correlation coefficient (r) = -0.984		
			Correlation coefficient (r) = -1.000	

Mean of three replications; RD = Recommended dose; DRD = Double the recommended dose; LOQ = Limit of quantification (0.01 mg kg⁻¹)

4. CONCLUSIONS

The dissipation of λ -cyhalothrin 5% EC in okra fruits followed first order kinetics and residues reached below limit of quantification (0.01 mg kg⁻¹) within 7 to 10 days of application. In soil, the residues dropped below the limit of quantification within 3 to 5 days. As the residue level of λ -cyhalothrin in okra fruits was below the corresponding EU-MRL (0.2 mg kg⁻¹) even on the same day of spraying, this insecticide can safely be used in okra crop.

DISCLAIMER (ARTIFICIAL INTELLIGENCE)

Authors hereby declare that NO generative AI technologies such as Large Language Models (Chat GPT, COPILOT, etc.) and text-to-image generators have been used during the writing or editing of this manuscript

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