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Study the persistence of lambda-cyhalothrin residues in the soil of okra field

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Abstract
A field experiment was conducted at Rajasthan Agricultural Research Institute, Durgapura, Jaipur during Summer, 2022 to study the persistence of lambda-cyhalothrin residues in the soil of okra field, when sprayed at its recommended dose (lambda-cyhalothrin 15 g. ha^{-1}) and double of the recommended dose (lambda-cyhalothrin 30 g. ha^{-1}). The samples were extracted and cleaned up using a modified QuEChERS method and the residues were analyzed by GC-ECD. The residues level of lambda-cyhalothrin in okra field soil collected at harvest time of okra crop were below the detectable level (BDL) at the recommended dose and double of the recommended dose, respectively.

Keywords: Dissipation, residues and lambda-cyhalothrin

1. Introduction
Okra (*Abelmoschus esculentus* (L.) Moench), also known as Bhindi or lady's finger, is an important crop that is grown in India throughout the year and belongs to the family Malvaceae. Okra is one of the vegetable crops that are farmed in India. It is cultivated in a great number of tropical and subtropical regions around the world, including India. Okra's young, tender fruits can be eaten fresh or prepared as a vegetable, or they can be cut and dried for use in cooking. As a result of its high mucilage content, it is also used for the purpose of thickening sauces and soups. According to Chauhan (1972) [5], the roots and stems of okra are utilised in the process of cleansing cane juice. It is an excellent source of proteins, vitamins, minerals, carbs, iron, potassium, and acids such as rhamnose (22%), galacturonic acid (27%), and amino acid (11%), and it is also the best supplier of iodine and calcium. Pesticide residues can still be found in food even when "good agricultural practices," or GAP, are used. These residues can also be found in the soil. Not only does the excessive and careless use of insecticides on agricultural products pose a threat to human health, but it also endangers the lives of all other forms of life. Pesticide residues are associated with both acute and chronic health issues. According to Kelageri *et al*. (2017) [10], persistent pesticide exposure has been linked to an increased risk of developing cancer, chronic renal diseases, a suppressed immune system, sterility in both males and females, endocrine problems, neurological and behavioural disorders, and notably in children. These risks are especially prevalent among youngsters.
Degradation of pesticides affects the diversity of microbes in the soil, as well as metabolic processes and enzyme activity (Hussain *et al*., 2009; Munoz-Leoz *et al*., 2011) [8, 12]. Pesticides that enter the soil can change soil microbial diversity and biomass. Any change in the activity of soil microorganisms caused by pesticides results in soil ecosystem disruption and loss of soil fertility (Handa *et al*., 1999) [7]. Insecticides used on the crop eventually made their way into the soil. Pesticides in the soil have an impact on soil microflora and fauna, beneficial microorganisms, natural enemies, soil texture, and eventually crop productivity. So the study looked at the durability of these suggested insecticides in soil.

2. Materials and Methods
2.1. Reagents and Instruments
Certified Reference Material (CRM) as procured from accu standard and all the solvents used were HPLC grade. The chemicals (Na_2SO_4, primary secondary amine (PSA) and $mgSO_4$ were used analytical regent grade and activated by heating at 30 °C for 12 hrs and kept in desiccators. GC Shimadzu-2010, Analytical balance, Mixer, Centrifuge and Turbovap.

2.2. Pesticides and application rate
Commercial formulations, of lambda-cyhalothrin (5% EC) at recommended dose @ 15 g. ha^{-1} and double of recommended dose @ 30 g. ha^{-}.[1]

2.3. Field experimental design
The field experiment was conducted at Rajasthan Agricultural Research Institute, Durgapura, Jaipur during Kharif, 2022. The soil of the experimental field was sandy loam with pH 8.1, EC 0.18 dSm^{-1} , organic carbon 2.1 g kg^{-1}, available N 178 kg ha^{-1}, P_2O_5 21.8 kg ha^{-1} and K_2O 193.4 kg ha^{-1}. The trial included three treatments: control, the indicated dose of lambda-cyhalothrin 5% EC (15 g/ha), and spiromesifen 5% EC (30 g/ha) with four replications. Every agronomic practice was followed. During the testing period, there was no rain. The first spray of insecticide was applied during the fruit development stage, and the second spray was applied at 10-day intervals at the prescribed dose (15 g/ha) and double the recommended dose (30 g/ha), respectively, while one plot was kept untreated and used for fruit sampling as a control in each treatment. At the harvest of the okra crop, approximately 1 kilogramme of soil was collected randomly and independently from the control and treated plots for each treatment.

2.4. Sampling
At harvest time, soil samples (1 kg) from the sprayed okra field were collected from each replication for examination. Soil samples were taken from depths ranging from 0 to 15 cm during sampling. The samples were placed in plastic containers and left to dry in the laboratory in the shade at room temperature. The air-dried samples were manually desegregated using a pestle and marble mortar, then passed through a 20-mm brass soil sieve and properly combined to obtain homogeneity.

2.5. Extraction (QuEChERS)
According to Asensio Ramos *et al.* (2010) [1], 10 g of a representative soil sample and 20 ml of acetonitrile were extracted out of a 50 ml centrifuge tube. Four grammes of magnesium sulphate and one gramme of sodium chloride were added after a minute of vigorous shaking. Citrate buffered media (0.5 g of disodium hydrogen citrate sesquihydrate and 1 g of trisodium citrate dehydrate) was added to increase the recovery values. Centrifuge for five minutes at 3,300 rpm. 250 mg of PSA and 1.5 g of magnesium sulphate were contained in a 15 ml centrifuge tube with 10 ml of the supernatant. After briefly shaking the contents and one minute of sonication, the tube was centrifuged for ten minutes at 4,400 rpm. Four millilitres of the supernatant were removed from the aforesaid centrifuge tube and dried using rotavapour at 40 degrees Celsius. One millilitre of hexane was used to resurrect the dry residue. A small amount of anhydrous sodium sulphate was added and filtered through PTFE filters in the event that an aqueous phase was detected.

2.6. Standards
For quantification, the reference standard lambda-cyhalothrin was obtained from the Pesticide Residues Laboratory, Division of Entomology, RARI, Durgapura, Jaipur, Rajasthan.

A. Standard stock solution
To make a 1000 mg kg^{-1} standard stock solution, 98.2% pure lambda-cyhalothrin was dissolved in a 100 ml volumetric flask with hexane.

B. Intermediates stock solution:
To prepare a homogenous intermediate stock solution of 10 mg kg^{-1}, 1 ml of the standard stock solution was transferred to a 100 ml volumetric flask, the volume was adjusted, and the flask was shaken thoroughly. The standard stock solution was brought to room temperature. This was used to strengthen the samples.

C. Working standard
We made working standards of 0.01 to 1 mg kg^{-1} from the intermediate stock solution by stepwise dilution and labelled graduated test tubs after letting them cool to room temperature. The working standards were used to find out how long these chemicals stayed in the samples and how much of them were left over.

2.7. Recovery studies
The soil samples were made richer at 0.05, 0.25, and 0.50 mg kg^{-1} for lambda-cyhalothrin by adding the right amount of 10 mg kg^{-1} intermediates stock solution to figure out the recovery percentage.

2.8. Instruments parameters
A Shimadzu-2010 gas chromatograph with a capillary column DB 5 (30 m x 0.25 mm ID, 0.25 m film thickness) was used to measure the amount of lambda-cyhalothrin residues.
The following were the operating parameters.

Column temperature °C	Rate (°C/minute)	Temperature (°C)	Hold time (minute)
	------	160.0	1.00
	7.0	280.0	5.00
Injector temperature °C	280		
Detector temperature °C	300		
Gas flow rate (ml min^{-1})			
Total flow (Detector)		12.0	
Column flow		1.50	
Aliquot injected		1µl	
Split ratio		5	

2.9. Analysis of lambda-cyhalothrin residues
The gas chromatograph (GC) and the Shimadzu-2010 instrumental parameters were utilized for the purpose of detecting and quantifying lambda-cyhalothrin residue in the soil. Standard solutions containing various amounts of pesticides were created and injected into the instrument before the sample extract could be injected. This was done before the sample could be injected. The insecticide chemical was qualitatively identified by contrasting the retention duration of peaks and quantitatively calculated by comparing the area of chromatograms obtained in each test sample to that of the analytical standard. Both of these steps were performed in parallel. The results of the sample were given in milligrams per kilogramme. The actual amount of pesticide residue that was present in the sample was determined by using the following formula, which was based on the value that was obtained from the sample.

$$\text{Residues } (\mu g/g^{-1}) = \frac{\text{Peak area (Sample) x Conc. std (ppm) x µL std injected x Final volume of the sample (1 mL)}}{\text{Peak area (Std) x weight of the sample (2 g) xµL of sample injected}}$$

$$\text{Wt. of sample Analyzed (g)} = \frac{\text{Sample wt. (10 g) X Aliquot taken (4 mL)}}{\text{Volume of extract (20 ml)}} = 2 \text{ g}$$

$$\% \text{ Recovery} = \frac{\text{Sample peak area}}{\text{Standard peak area}} \times 100$$

2.10. Statistical analysis
Statistical analysis was performed on Microsoft Excel-2016 (Microsoft Corporation, USA). All analysis was performed in triplicate and the results were expressed as mean ± SD.

3. Results and Discussion
3.1 Recovery
To ensure the reliability of the results, a recovery study was also conducted for lambda-cyhalothrin in soil. The soil samples were spiked with lambda-cyhalothrin at 0.05, 0.25 and 0.50 mg kg^{-1} fortification levels and analysed as per the methodology described earlier. The results of the recovery study are presented in Table 1. The recovery study of lambda-cyhalothrin was carried out at the fortification level of 0.05, 0.25 and 0.50 mgkg^{-1} in soil. The mean recovery of lambda-cyhalothrin at 0.05, 0.25 and 0.50 mg kg^{-1} fortification levels was 88.0, 91.4 and 88.1 percent in soil, respectively. A similar trend of recoveries has been reported by Mao *et al*. (2010) [11] who reported the average recovery of lambda cyhalothrin as 91.36–96.64 percent from fortified tobacco field soil at different fortification levels between 0.05 and 0.25 mg/kg. Beevi *et al*. (2014) [3] recovered 99.70 and 104.80 percent lambda-cyhalothrin from cardamom field soil at fortification levels of 0.05 and 0.25 mg/kg, respectively, which strongly support the present finding. According to the Sante (2015) [16] guidelines, any analytical method that records mean recovery in the range of 70–120 percent is accurate and precise. Hence, the method employed in the present study for the extraction of lambda-cyhalothrin from soil was accurate and precise.

3.2 Residues
The gas chromatograph (GC) and the Shimadzu-2010 instrumental parameters were utilised for the purpose of detecting and quantifying lambda-cyhalothrin residue in the soil. Before the injection, Under the cover of an okra crop and under favourable climatic conditions such as high temperature, it indicates that relatively low dosages of lambda-cyhalothrin (15 g. and 30 g. a.i. ha^{-1}, respectively) may play a role in the faster dissipation/degradation of lambda-cyhalothrin. It is reasonable to infer that evaporation, leaching, and the uptake of these pesticides by crops each play a part in the process that leads to their rapid dissipation.
The persistence and dissipation of lambda-cyhalothrin residues in soil under cover of the okra crop have been researched (obtained from two treatments, namely the recommended dose (15 g. a.i. ha^{-1}) and double of the required dose (30 g. a.i. ha^{-1}; the results of these two treatments are shown in Table 2). At the time of harvesting the okra crop, soil samples were taken for analysis. In the case of soil samples, the residues at the time of harvesting the okra crop were not found in either the recommended dose (15 g. a.i. ha^{-1}) or double of the necessary dose (30 g. a.i. ha^{-1}). The control soil samples did not, in the same way, exhibit any of the residues. The findings of Reddy and Reddy (2013) [15], who evaluated the persistence and dissipation of lambda cyhalothrin (30 g a.i./ ha^{-1}) on chilli as well as in soil, lend support to the conclusions of the current research. Reddy and Reddy (2013) [15] discovered that after 15 days of spraying, there was no residue found in the soil where they conducted their study. The present findings are also in accordance with those of Barik *et al*. (2010) [2] who reported that no residue of lambda-cyhalothrin was detected in the harvested paddy, straw, grain, and soil samples when applied at 66 g a.i. ha^{-1} and Teló *et al.* (2015) [17] investigated the dissipation and persistence of insecticides (lambda-cyhalothrin and thiamethoxam) in irrigation water, soil, rice plant, panicle, and rice grain; however, pesticide residues were not detected in soil. These results are also in accordance with Nidhi (2018) [13], Gupta R. (2018) [6], and Bhartiya S. (2018) [4], who reported that the lambda-cyhalothrin residues were below the level of determination (0.05 mg/kg^{-1}) in field soil samples of cauliflower, pea, and cabbage crops on the third day after application when lambda-cyhalothrin was applied at 15 and 30 g a.i. ha^{-1}
Above findings was also in accordance with Kansara *et al*. (2021) [9] who found that residues of insecticides combination (Chlorantraniliprole 9.26% + lambda-cyhalothrin 4.63% ZC) measured from soil at 30th day and harvest time were below the limit of quantification (LOQ) in pigeonpea. Furthermore, Pathan *et al*. (2023) [14] who studied persistence and dissipation kinetics of novaluron 9.45% + lambda-cyhalothrin 1.9% ZC insecticides in tomato crop and revealed that in soil samples, the residue levels were at below the determination level (0.05 mg/kg) for lambda-cyhalothrin at recommended dose @ 43.31 g a.i. ha^{-1} and at double of the recommended dose @ 86.62 g a.i. ha^{-1} support the present findings.

3.3 Limit of quantitation and Limit of detection
In the soil, the limits of quantification (LOQ) and detection (LOD) for lambda-cyhalothrin were 0.05 mg kg^{-1} and 0.01 mg kg^{-1} respectively. The levels of lambda-cyhalothrin residue that were found in the field soil that had been collected at the time of harvesting okra crops were below the detectable level (BDL) at both the authorised dose and double the recommended dose.

Table 1: Percent recovery of lambda-cyhalothrin in soil at different fortification levels.

Level of Fortification (mg kg^{-1})	Replications	Soil	
		µg recovered	Recovery (%)
0.05	R_1	0.043	86.0
	R_2	0.041	82.0
	R_3	0.045	90.0
	R_4	0.047	94.0
Mean ± SD		88.0±5.163	
0.25	R_1	0.217	86.8
	R_2	0.222	88.8
	R_3	0.246	98.4
	R_4	0.229	91.6
Mean ± SD		91.4±5.064	
0.50	R_1	0.423	84.6
	R_2	0.437	87.4
	R_3	0.456	91.2
	R_4	0.446	89.2
Mean ± SD		88.1±2.802	

Table 2: Residues (mg kg^{-1}) of lambda-cyhalothrin in soil under okra crop at recommended dose (15 g. a.i. ha^{-1}) and double of the recommended dose (30 g. a.i. ha^{-1}).

Days	Replications	Recommended dose (15 g a. i. ha^{-1})		Double of the recommended dose (30 g a. i. ha^{-1})	
		Average* Residues ± SD	% Dissipation	Average* Residues ± SD	% Dissipation
Soil Control	R_1	ND	-	ND	-
	R_2	ND	-	ND	-
	R_3	ND	-	ND	-
	R_4	ND	-	ND	-
Soil at Harvest time	R_1	ND	-	ND	-
	R_2	ND	-	ND	-
	R_3	ND	-	ND	-
	R_4	ND	-	ND	-

* Average of four replications
ND-Not Detected

4. Conclusion

The field trial for this investigation on the dissipation and residues of lambda-cyhalothrin insecticide in soil was carried out as part of this research project. The QuEChERS method was utilized, and it resulted in the effective extraction of lambda-cyhalothrin residue from soil samples. The methodology that was created exhibited levels of accuracy and precision that were deemed acceptable, and it was successfully used to the study of the dissipation kinetics of lambda-cyhalothrin in soil. At the time when the okra crop was harvested, the current result indicated that the soil did not contain any pesticide residues including lambda-cyhalothrin. This conclusion would be helpful to the government in providing guidelines on the appropriate and safe use of lambda-cyhalothrin, as well as in determining local Maximum Residue Levels (MRL) for the chemical.

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