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Decontamination of λ-cyhalothrin Residues in Okra Fruits

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ABSTRACT: Subjecting fruits and vegetables to effective decontamination methods is crucial for reducing the risks posed by the indiscriminate use of pesticides. In this context, a study was conducted on decontamination of λ-cyhalothrin residues in okra fruits. λ-cyhalothrin was applied in okra crop twice at recommended dose (15 g a.i. ha^{-1}) and double dose (30 g a.i. ha^{-1}). Okra fruit samples were collected from each treatment 0 (2 h), 1 and 3 day(s) after second spray and subjected to various decontamination methods. The residues in the processed fruits were analyzed using Gas Chromatography-Mass Spectrometry. All the decontamination methods were effective in removing λ-cyhalothrin residues from okra fruits, however, washing followed by boiling okra fruits removing maximum residues (53-72%) proved to be the most effective, followed by dipping in 2 per cent salt solution (52-68%) and 2 per cent tamarind solution (48-65%). Conversely, only washing okra fruits under tap water was the least effective method of decontamination.

Keywords: Okra, λ-cyhalothrin, residue, decontamination, GCMS/MS.

INTRODUCTION

Okra (*Abelmoschus esculentus* L. Moench) is an economically important vegetable crop cultivated in tropical and subtropical regions worldwide. Its pods are a rich source of essential minerals like potassium, calcium, phosphorus, and magnesium, while being low in cholesterol and saturated fats (Habtemariam, 2019). One of the major setbacks identified in the production of okra is the increasing incidence of insect pests as more than 72 species of insect pests and mites have been documented to infest the okra crop (Mishra *et al.*, 2002). To combat the problem of insect pests, farmers primarily use chemical pesticides, and they do so at far higher doses than recommended (Kabadad and Gali 2018). A wide range of pesticides including λ-cyhalothrin have been evaluated to minimize losses due to pests. λ-cyhalothrin (S)-a-cyano3-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 (Chauhan *et al.*, 2012). It has been reported very effective against a wide range of chewing and sucking insect pests, particularly lepidopterous, coleopterous and mite pests in fruits, vegetables and other field crops (Dikshit *et al.*, 2000; Mathirajan *et al.*, 2000).

However, use of pesticides presents substantial risks to both the environment and public health (Riyaz *et al.*, 2022; Ahmad *et al.*, 2024). Okra being a fast growing crop is harvested at very short interval of 2-3 days and this increases the chance of fruits having high level of residues which can pose health hazards to the consumers (Mariappan and Kaithamalai 2020). Therefore, before consuming okra fruits, these should be subjected to some culinary process for reducing the pesticide risk. Additionally, common decontamination processes such as washing with tap water or using salt or tamarind solutions have been reported to reduce residue levels significantly (Mariappan and Kaithamalai 2020). Hence, in order to develop effective and affordable risk mitigation methods that can be easily implemented at home, the present study on decontamination of λ-cyhalothrin residues in okra fruits was conducted.

MATERIALS AND METHODS

The present study was conducted at Chaudhary Charan Singh Haryana Agricultural University, Hisar (Haryana), India during 2019.

A. 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 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.

B. Sampling and processing

Okra fruit samples (500 g) were collected 0 (2 h), 1 and 3 day(s) after application of second spray from the treatments including recommended and double the recommended dose of λ-cyhalothrin. For removal of pesticide residues, okra fruits were subjected to various decontamination processes such as only washing under tap water, washing followed by boiling, dipping in lukewarm water, 2 per cent salt (NaCl) solution, 2 per cent tamarind solution, lemon water (1 lemon/1 litre) and 1 per cent vinegar solution for five minutes followed by washing under tap water. Okra fruits not subjected to any decontamination process were treated as control/unwashed. Each treatment was replicated thrice.

C. Extraction and clean-up of residues

After subjecting to various decontamination processes, okra fruit samples were processed by liquid-liquid partitioning method. 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 1litre 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.

D. Residue estimation

GC-MS/MS system based on chromatographic technique was used for estimation of λ-cyhalothrin residues in okra fruits. 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 1µl. 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 1). λ-cyhalothrin residues were calculated as under:

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

Where, A_1 = Peak area of the sample, A_2 = Peak area of the standard, I_1 = Injected volume of standard (µl), I_2 = 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).

E. Method validation

To check the validity and authenticity of the method used for estimation of λ-cyhalothrin residue in okra fruits, a recovery experiment was conducted at different spiking levels. For this purpose, 20 g of crushed okra fruits was 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.

RESULTS AND DISCUSSION

A. Method validation

The average recovery of λ-cyhalothrin residue in okra fruit samples was 89.07, 90.20 and 94.92 per cent when samples were fortified at level of 0.01, 0.05 and 0.10 mg kg^{-1}, respectively (Table 2). Taking into account the satisfactory recovery percentage (70-110%) and RSD (< 20), the method was considered suitable for the assessment of λ-cyhalothrin residues in okra fruits (SANCO 2011). The limit of quantification (LOQ) was found to be 0.01 mg kg^{-1}. Similarly, in another study 87 to 92 per cent and 97 to 101 per cent recovery of lambda-cyhalothrin was reported in tomato fruits when fortified at the level of 0.25 mg kg^{-1} and 0.50 ug g^{-1}, respectively (Chauhan *et al*., 2012).

B. Decontamination of λ-cyhalothrin residues in okra fruits

At recommended dose: Following the application of λ-cyhalothrin at recommended dose (15 g a.i. ha^{-1}), the average initial deposits of 0.079, 0.042 and 0.027 mg kg^{-1} were detected in okra fruit samples collected 0 (2 h), 1 and 3 day(s) after spray, respectively (Table 3). All the decontamination processes were effective in reducing λ-cyhalothrin residues in okra fruits when samples collected 0 (2 h) and 1 day were processed. However, λ-cyhalothrin residues in okra fruits collected 3 days after spraying and subjected to various decontamination processes, were below the limit of quantification in case of all the treatments. Among all, washing followed by boiling of okra fruits was most effective and resulted in maximum removal of residues *i.e.*, 72.15 and 64.29 per cent when samples collected on 0 and 1 day after spray were processed, respectively. Dipping okra fruits in 2 per cent salt solution and then washing under tap water caused 67.09 and 59.52 per cent reduction in residues in okra fruits collected at respective interval and was next in the order of effectiveness, followed by dipping in 2 per cent tamarind solution (65.82 and 57.14 %), lukewarm water (58.23 and 54.76 %), lemon water (59.49 and 52.38 %) and 1 per cent vinegar solution (54.43 and 50 %), and then washing under tap water. In contrast, only washing under tap water removing 51.90 and 42.86 per cent of residues in samples collected on 0 and 1 day after spray, respectively was least effective method of decontamination.

At double the recommended dose: In case of double the recommended dose (30 g a.i. ha^{-1}), the average initial residues of 0.162, 0.089 and 0.058 mg kg^{-1} were detected in okra fruit samples collected 0 (2 h), 1 and 3 day(s) after spray, respectively (Table 4). Likewise, washing followed by boiling of okra fruits was most effective method of decontamination and caused maximum reduction *i.e.*, 72.22, 65.17 and 53.45 per cent in fruits samples collected at respective interval. Dipping okra fruits in 2 per cent salt solution and then washing under tap water removed 67.90, 59.55 and 51.72 per cent of residues at respective interval, and was next effective method, followed by dipping in 2 per cent tamarind solution (64.81, 56.18 and 48.28%), lukewarm water (59.03, 52.81 and 46.55%), lemon water (61.73, 49.44 and 41.33%) and 1 per cent vinegar solution (55.56, 47.19 and 39.66%), and then washing under tap water. Similarly, only washing under tap water was least effective and reduced the residues by only 45.06, 38.20 and 27.59 per cent in fruit samples collected 0 (2 h), 1 and 3 day(s) after spray, respectively.

These results are in close proximity of earlier studies in which also washing followed by boiling was reported to be more effective in removing of λ-cyhalothrin residues than only washing of tomato fruits (Chauhan *et al.*, 2012). Similarly, in case of brinjal, cooking of fruits resulted in maximum removal of λ-cyhalothrin residues, followed by dipping in 2 per cent salt solution, lemon water wash, tap water wash and dipping in 2 per cent tamarind solution (Rao *et al.*, 2014). In contrast, dipping of field bean pods in salt solution (2%) followed by tap water wash was reported to be more effective in removal of λ-cyhalothrin residues than cooking in pressure cooker followed by tap water wash, however, tap water wash as least effective method (Srinivasa *et al.*, 2018). In another studies, processing of fruits with 2 per cent salt solution resulted in 48.02 per cent removal of λ-cyhalothrin residues in tomato (Kelageri *et al.*, 2017) while 42.57 per cent in green chilli (Baby Rani *et al.*, 2019).

Table 1: Parameters of GC-MS/MS used for residue study.

Parameters	Details
Software	GCMS solution version 2.53 SU3
Column	Rtx-5 MS -1(30m × 0.32 mm 1D X 0.25 µm film thickness) of 5 per cent diphenyl + 95 per cent dimethyl polysiloxane
Temperature	
Oven	80^{o} (2 min) ➔ 20^{o}C min^{-1} ➔ 180 (0 min) ➔ 5^{o}C min^{-1} ➔ 300^{o}
Iron source temp.	250^{o}C
Interface temp.	270^{o}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

Table 2: Recovery of λ-cyhalothrin in fortified okra fruits

Fortification level (mg kg^{-1})	Recovery (%) (Mean ± SD)	RSD (%)
0.01	89.07 ± 4.55	5.11
0.05	90.20 ± 4.00	4.43
0.10	94.92 ± 2.68	2.82

Table 3: Efficiency of decontamination methods in reduction of λ-cyhalothrin residues in okra fruits at recommended dose (15 g a.i. ha^{-1}).

Decontamination method	0 D (2 h)		1 D		3 D	
	Residues (mg kg^{-1}) Mean + SD	Reduction (%)	Residues (mg kg^{-1}) Mean + SD	Reduction (%)	Residues (mg kg^{-1}) Mean + SD	Reduction (%)
Unwashed	0.079 + 0.005	—	0.042 + 0.006	—	0.027 + 0.003	—
Tap water washing	0.038 + 0.006	51.90	0.024 + 0.006	42.86	<LOQ	—
Luke warm water	0.033 + 0.005	58.23	0.019 + 0.005	54.76	<LOQ	—
Saline solution (2%)	0.026 + 0.003	67.09	0.017 + 0.004	59.52	<LOQ	—
Tamarind solution (2%)	0.027 + 0.006	65.82	0.018 + 0.005	57.14	<LOQ	—
Lemon water	0.032 + 0.006	59.49	0.020 + 0.006	52.38	<LOQ	—
Vinegar solution (1%)	0.036 + 0.004	54.43	0.021+ 0.006	50.00	<LOQ	—
Washing + Boiling	0.022 + 0.005	72.15	0.015 + 0.003	64.29	<LOQ	—

D = Day(s) after spray

Table 4: Efficiency of decontamination methods in reduction of λ-cyhalothrin residues in okra fruits at double the recommended dose (30 g a.i. ha^{-1}).

Decontamination method	0 D (2 h)		1 D		3 D	
	Residues (mg kg^{-1}) Mean + SD	Reduction (%)	Residues (mg kg^{-1}) Mean + SD	Reduction (%)	Residues (mg kg^{-1}) Mean + SD	Reduction (%)
Unwashed	0.162 + 0.007	—	0.089 + 0.006	—	0.058 + 0.006	—
Tap water washing	0.089 + 0.006	45.06	0.055 + 0.005	38.20	0.042 + 0.006	27.59
Luke warm water	0.066 + 0.006	59.03	0.042 + 0.007	52.81	0.031 + 0.005	46.55
Saline solution (2%)	0.052 + 0.006	67.90	0.036 + 0.005	59.55	0.028 + 0.006	51.72
Tamarind solution (2%)	0.057 + 0.005	64.81	0.039 + 0.005	56.18	0.030 + 0.005	48.28
Lemon water	0.062 + 0.004	61.73	0.045 + 0.006	49.44	0.034 + 0.005	41.33
Vinegar solution (1%)	0.072 + 0.005	55.56	0.047 + 0.005	47.19	0.035 + 0.005	39.66
Washing + Boiling	0.045 + 0.004	72.22	0.031 + 0.004	65.17	0.027 + 0.004	53.45

D = Day(s) after spray

CONCLUSIONS

The indiscriminate and excessive use of pesticides resulted in residues accumulation in the food materials, water, fruits, vegetables and in total diet and leads to the chronic disorders in human body. This risk of residues in vegetables such as okra may be reduced to a greater extent by processing them with some common household decontamination methods like washing followed by boiling of okra fruits, dipping in salt solution (2%) and tamarind solution (2%) which resulted up to 72 per cent reduction in λ-cyhalothrin residues.

FUTURE SCOPE

The similar studies may be conducted for mitigation of pesticides risk in other vegetables as well as fruits. Apart from this, other possible household processes many also be explored for this purpose.

Author contributions. All authors contributed to the study conception and design. Conceptualization of research and designing of the experiments were performed by Bhupender Singh, Ram Karan and Anil Jakhar Bhupender Singh conducted the experiment and collected. Reena Chauhan analyzed the data. The first draft of the manuscript was written by Bhupender Singh and all authors commented on previous versions of the manuscript. All authors read and approved the final manuscript.

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Conflict of Interest. None.

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