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RESEARCH ARTICLE

Dissipation kinetics and decontamination of chlorantraniliprole residues in okra fruits and soil

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Abstract

Information on the persistence behavior of a pesticide in agricultural produce and soil is crucial before its recommendation for agricultural use. A study was carried out to investigate dissipation kinetics and decontamination of chlorantraniliprole residues in okra fruits and soil, applying twice at the recommended dose (25 g a.i. ha^{-1}) and double dose (50 g a.i. ha^{-1}). Residues were estimated using Gas Chromatography-Mass Spectrometry. In 2019, the initial level of chlorantraniliprole residues in okra fruits was 0.099 and 0.201 mg kg^{-1}, while in 2020, it was 0.071 and 0.135 mg kg^{-1} at the respective doses. In both years, residues declined below the limit of quantification (0.01 mg kg^{-1}) within 7 and 10 days at recommended and double doses, with corresponding half-lives of 2.28 and 2.95 days in 2019 and 3.27 and 3.20 days in 2020. The initial residues in soil were 0.031 and 0.055 mg kg^{-1} in 2019, while 0.045 and 0.073 mg kg^{-1} in 2020 at the corresponding doses. Residues dropped below the limit of quantification within 3-5 days with a half-life of 1.58 days for both doses in 2019, while 1.51 and 1.65 days in 2020 at the respective doses. Washing okra fruits under tap water and then boiling was most effective in removing residues (58-72 %), followed by dipping in a 2 % salt solution (55-68 %) and subsequently tap water washing. Initial residues in okra fruits well below the EU MRL (0.3 mg kg^{-1}) even at double dose indicated that chlorantraniliprole is safe for okra cultivation; however, employing decontamination methods can further minimise the residue risk.

Keywords: chlorantraniliprole; decontamination, dissipation; half-life; residues; okra

Introduction

Okra, *Abelmoschus esculentus* (L.) Moench, originating in Ethiopia, is cultivated widely in tropical and subtropical regions worldwide. Rich in water and nutrients, okra fruits are a good source of vitamins and minerals (1). It is consumed both fresh and cooked and used as an ingredient in soups, salads and stews (2). In India, okra is grown in an area of 0.546 M ha with a total production of 6.7 MMT and a productivity of 12.27 MT (3). Okra is highly susceptible to damage by insect pests as it is attacked by approximately 72 species of insect and mite pests (4). To manage these pests, farmers frequently rely on several chemical pesticides, including chlorantraniliprole (CAP) {3-bromo-N-[4-chloro-2-methyl-6-[(methylamino)carbonylphenyl]-1-(3-chloro-2-pyridinyl)-1H-pyrazole-5-carboxamide}, a novel insecticide belonging to the anthranilic diamide class. This insecticide was developed by DuPont Crop Protection in 2007 (5). It is the first commercially available anthranilic diamide insecticide that targets ryanodine receptors, triggering an uncontrolled release of calcium ions from stores in the sarcoplasmic reticulum, leading to disrupted muscle contraction (6, 7).

Chlorantraniliprole at the prescribed dosage has demonstrated effectiveness against insect pests (8), especially lepidopteran pests, causing a substantial reduction in fruit damage (9). Additionally, it has been recognised as a viable alternative to synthetic pyrethroids in vegetable crops (10). However, the misuse or excessive application of pesticides by farmers is a growing concern as it can result in pesticide residues in harvested produce, which may persist for a longer time, potentially posing health risks to consumers (11, 12). Climatic conditions play a vital role in the dissipation of pesticides from crops and soil. Temperature, humidity, sunlight intensity and rainfall significantly influence the rate of degradation, volatilisation, leaching and photodecomposition. High temperatures and intense solar radiation enhance volatilisation and photolysis, thereby accelerating pesticide breakdown, whereas low temperatures and dry conditions slow down microbial and chemical degradation, leading to longer persistence. Similarly, rainfall can either enhance dissipation through leaching and runoff or cause redistribution of residues on plant surfaces (13-15). Therefore, it is crucial to study the dissipation behaviour of the pesticides in various environmental components, such as food and soil, before recommending them for field application. Additionally, several simple household techniques have been proposed to reduce pesticide residues to a greater extent (16, 17) and offer a practical approach to minimise the presence of these residues in food. In this context, the present study was undertaken to investigate the dissipation pattern of chlorantraniliprole in okra fruits and soil, as well as to evaluate decontamination techniques for mitigating the risks associated with its residues.

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. However, the decontamination study was conducted exclusively in 2019.

Field experiment

The field experiment was conducted in a randomised block design with a plot size of 5.4 × 4.2 m. During both seasons, okra (variety *Hisar Naveen*) was sown at a spacing of 60 cm and 30 cm between the rows and plants, respectively, adhering to recommended agronomic practices (18). The soil type of the research area is sandy loam. Chlorantraniliprole 18.5 SC was applied in okra at the recommended dose (25 g a.i. ha^{-1}) and double the recommended dose (50 g a.i. ha^{-1}) twice, first at fruit initiation, followed by a second spray at 15 days intervals. One treatment without any insecticidal application was maintained as a control. Each treatment was replicated three times. The trial was conducted in a semi-arid region and the climatic conditions prevailing during the study are presented in Table 1.

Dissipation study

Reagents and chemicals

The Certified Reference Material (CRM) of chlorantraniliprole was procured from Sigma Aldrich with a purity of 99.2 %, while its formulation [Coragen 18.5 SC] was obtained from the local market. A 10 mg quantity of CAP pesticide was dissolved in 100 ml of acetonitrile to prepare a stock solution. Working solutions ranging from 0.01 to 1.0 ppm for calibration and recovery experiments were then prepared from this stock solution using serial dilution. All solvents used were of analytical grade and reagent blanks were run before sample analysis to ensure the purity of chemicals and organic solvents. Glasswares used in the experiment were made of borosilicate and were thoroughly cleaned with acetone before each use.

Sampling and processing

For investigating the dissipation pattern of chlorantraniliprole, samples of okra fruits (500 g) were collected in polybags at 0 (2 h after application), 1, 3, 5, 7 and 10 day(s) after the second spray from five randomly selected plants in each treatment. Fruit samples were processed on the same day of collection by the standard method (19). For the extraction of pesticide residues from okra fruits, 20 g of the macerated sample was taken in a flask with 100 mL of acetone, shaken for one hour and filtered. The filtrate was transferred to a separatory funnel, mixed with 450 mL of brine solution (10 % NaCl) and partitioned twice with 100 mL and 50 mL of each dichloromethane and hexane. After each extraction, the organic phase was collected, passed through anhydrous sodium sulfate to remove moisture and treated with 0.3 mg of activated charcoal for four hours. The extract was then filtered, concentrated nearly to dryness using a rotary vacuum evaporator and adjusted to a final volume of 3 mL with n-hexane. For cleanup, the residue was passed through a glass column (60 cm × 2.2 mm i.d.) packed with Florisil and activated charcoal (3:0.3 w/w) between two layers of anhydrous sodium sulfate. The extract was eluted with 100 mL of hexane: acetone (9:1 v/v), concentrated again to near dryness and reconstituted to a final volume of 3 mL with n-hexane. Chlorantraniliprole residues were quantified using GC-MS/MS.

Similarly, soil samples (1 kg) were collected at the same interval to a depth of 15 cm beneath the treated okra plants, air -dried in the shade at room temperature and processed by the previously described method (20). A 15 g dried sample was treated with 0.5 mL ammonia for 1 hr, then layered on a glass column prepared with sodium sulfate and a Florisil-activated charcoal (3:0.3 w/w) mixture. The column was eluted with 100 mL hexane: acetone (9:1 v/v) and the eluate was concentrated to near dryness, reconstituted to 3 mL with n-hexane and transferred to vials for analysis.

Residue estimation

A GC-MS/MS system based on a chromatographic technique was employed for the estimation of chlorantraniliprole residues in okra fruits and soil. Before residue estimation, the system was standardised. The GC analysis was carried out using GC-MS Solution software version 2.53 SU3. An Rtx-5 column (30 m length, 0.25 µm film thickness) was used for the separation. Helium served as the carrier gas at a flow rate of 21 mL min^{-1} and the injection volume was 1 µL. The retention time recorded for chlorantraniliprole was 20.602 minutes. The limit of detection (LOD) and limit of quantification (LOQ) were 0.005 mg

Table 1. Meteorological parameters during the period of study

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

Table 2. GC-MS/MS parameters

Software	GCMS solution version 2.53 SU3
Column	Rtx-5 MS -1(30m ´ 0.32 mm 1D × 0.25 µm film thickness) of 5 % diphenyl ± 95 % dimethyl polysiloxane
Temperature	
Oven	80° (2 min) → 20°C min^{-1} → 180 (0 min) → 5 °C min^{-1} → 300°
Iron source temperature	250 °C
Interface temperature	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	Chlorantraniliprole: 20.602

kg^{-1} and 0.01 mg kg^{-1}, respectively (Table 2).

Method validation

A recovery experiment was carried out at various spiking levels to validate the method used for estimating chlorantraniliprole residues in okra fruits and soil. To achieve this, 20 g of macerated okra fruits and 15 g of soil from control plots were fortified with chlorantraniliprole Certified Reference Material (CRM) at three concentration levels: 0.01, 0.05 and 0.10 mg kg^{-1}. At each fortification level, the % recovery was estimated following the prescribed method of processing and analysis.

Linearity study

A calibration curve for chlorantraniliprole was constructed, plotting concentrations ranging from 0.01 to 1.00 ppm against peak area recorded for each concentration, revealing a linear relationship between the two (Table 3 and Fig. 1). The retention time (Rt) recorded for chlorantraniliprole was 20.602 minutes, as illustrated in a typical chromatogram (Fig. 2).

Table 3. Standard curve data for chlorantraniliprole

Concentration (mg kg^{-1})	Area
0.010	37416
0.025	74833
0.050	149666
0.100	299332
0.250	599664
0.500	1189929
1.000	2394659

Efficiency of the method

The mean recovery of chlorantraniliprole in okra fruits was 81.20, 82.13 and 90.07 % at fortification levels of 0.01, 0.05 and 0.10 mg kg^{-1}, respectively. In soil, the corresponding recoveries were 80.00, 83.20 and 89.32 % (Table 4). Given the acceptable recovery range (70-110 %) and relative standard deviation (<20), the method was deemed suitable for estimating chlorantraniliprole residues in okra fruits and soil (21-23).

Decontamination study

Okra fruit samples (500 g) were collected 0 (2 hr after application), 1 and 3 days (s) after application of the second spray from the plots treated with the recommended and double the recommended dose of chlorantraniliprole. For the removal of pesticide residues, okra fruits were subjected to various household processing methods, such as only washing under tap water, washing followed by boiling, dipping in lukewarm water, 2 % salt (NaCl) solution, 2 % tamarind solution, lemon water (1 lemon/1 L) and 1 % vinegar solution. Okra fruits were dipped in lukewarm water and different solutions for five minutes, followed by washing under tap water. Okra fruits not subjected to any processing method were treated as control/unwashed. Each treatment was replicated three times.

Table 4. Recovery of chlorantraniliprole in spiked okra fruit and soil samples

Fortification level (mg kg^{-1})	Okra fruits		Soil	
	Recovery (%) (Mean ± SD)	RSD (%)	Recovery (%) (Mean ± SD)	RSD (%)
0.01	81.20 ± 3.40	4.19	80.00 ± 3.16	3.94
0.05	82.13 ± 4.65	5.66	83.20 ± 4.56	5.48
0.10	90.07 ± 4.48	4.97	89.32 ± 3.83	4.29

Data analysis

Data analysis was performed using Microsoft Excel 2010. Mean insecticide residues, standard deviation (SD), regression equation, coefficient of determination (R^2), half-life values and waiting period were computed. The dissipation behaviour of insecticides was analysed using a first-order kinetic model. A linear relationship between the natural logarithm of residue concentrations and time was plotted to derive the regression equation. Half-life of chlorantraniliprole residues was calculated using the following formula (21, 22).

$$\text{Half-life } (t_{1/2}) = \ln 2/k \qquad \text{(Eqn. 1)}$$

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

Results and Discussion

Dissipation of chlorantraniliprole in okra fruits and soil

Okra fruits

In 2019, the mean residues of chlorantraniliprole in okra fruits on day 0 (2 hr after application) were 0.099 mg kg^{-1} at the recommended dose of 25 g a.i. ha^{-1} (Table 5). The residue level

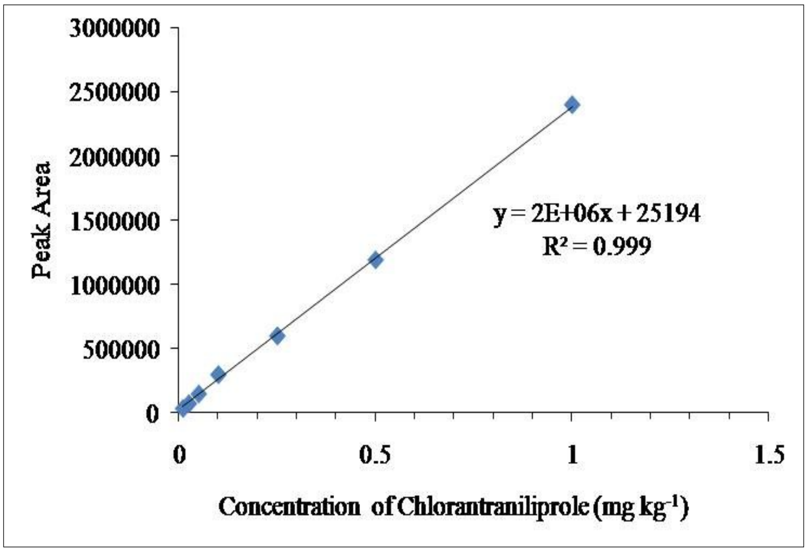

Fig 1. Standard curve of chlorantraniliprole

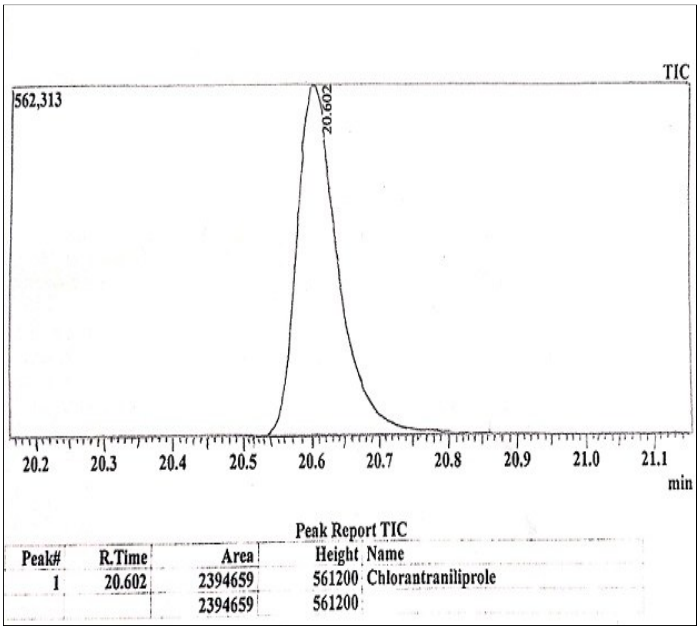

Fig 2. Chromatogram of standard chlorantraniliprole on GC-MS/MS

Table 5. Dissipation of chlorantraniliprole in okra fruits

Day(s) after spray	RD (25 g a.i. ha^{-1})		DRD (50 g a.i. ha^{-1})	
	Residue level (mg kg^{-1}) *Mean ± SD	Dissipation (%)	Residue level (mg kg^{-1}) Mean ± SD	Dissipation (%)
2019				
0 (2 h)	0.099 ± 0.007	--	0.201 ± 0.005	--
1	0.061 ± 0.005	38.38	0.126 ± 0.004	37.29
3	0.047 ± 0.005	52.53	0.098 ± 0.006	51.07
5	0.019 ± 0.004	80.81	0.078± 0.003	61.12
7	<LOQ	--	0.030 ± 0.004	85.09
10	--	--	<LOQ	--
	Correlation coefficient (r) = -0.975		Correlation coefficient (r) = -0.960	
	Regression equation (y) = 1.980 ± (- 0.132)x		Regression equation (y) = 2.280 ± (- 0.102)x	
	Half life value ($T_{1/2}$) = 2.28 days		Half life value ($T_{1/2}$) = 2.95 days	
	Degradation constant (k) = 0.305		Degradation constant (k) = 0.236	
2020				
0 (2 h)	0.071 ± 0.005	--	0.135 ± 0.007	--
1	0.059 ± 0.006	16.90	0.108 ± 0.006	20.00
3	0.037 ± 0.006	47.89	0.085 ± 0.004	37.01
5	0.025 ± 0.004	64.78	0.059 ± 0.006	56.30
7	<LOQ	--	0.026 ± 0.005	80.74
10	--	--	<LOQ	--
	Correlation coefficient (r) = -0.999		Correlation coefficient (r) = -0.970	
	Regression equation (y) = 1.854 ± (-0.092)x		Regression equation (y) = 2.159 ± (-0.094x)	
	Half life value ($T_{1/2}$) = 3.27 days		Half life value ($T_{1/2}$) = 3.20 days	
	Degradation constant (k) = 0.212		Degradation constant (k) = 0.216	

*Mean of three replications; RD = Recommended dose, DRD = Double the recommended dose; LOQ = Limit of quantification (0.01 mg kg^{-1})

dropped to 0.061 mg kg^{-1} when assessed one day after insecticidal application, representing a reduction of 38.38 %. The residues declined progressively and reached 0.047 mg kg^{-1} and 0.019 mg kg^{-1} in 3 and 5 days with degradation of 52.53 and 80.81 %, respectively. By the 7th day, residues fell below LOQ (0.01 mg kg^{-1}). In comparison, okra fruit samples collected from the plots treated with double dose (50 g a.i. ha^{-1}) contained initial deposits of 0.201 mg kg^{-1} (on day 0), which decreased to 0.126 mg kg^{-1} within 24 h, representing 37.29 % degradation. Residue level further declined to 0.098, 0.078 and 0.030 mg kg^{-1} in 3, 5 and 7 days of application, indicating 51.07, 61.12 and 85.09 % reduction, respectively. By the 10th day of spray, residues dropped below LOQ.

A negative correlation was observed between the residue level of chlorantraniliprole and time. The dissipation followed first-order kinetics, with degradation rate constants of 0.305 and 0.236 per day, leading to residues falling below the LOQ within 7 and 10 days after application, with half-life values of 2.28 and 2.95 days at the recommended and double dose, respectively (Fig. 3). In 2020 as well, dissipation of chlorantraniliprole closely followed the trend that was observed during the previous year. At the recommended dose, the residue level in okra fruits was 0.071 mg kg^{-1}on day 0, which declined to 0.059 mg kg^{-1} within 24 h, representing a 16.90 % reduction. The dissipation rate then accelerated with residue level dropping to 0.037 mg kg^{-1} and 0.025 mg kg^{-1} in 3 and 5 days, representing a reduction of 47.89 and 64.78 %, respectively. By the 7th day, residues were below LOQ. However, at double dose, the initial residue concentration was 0.135 mg kg^{-1}on day 0, which declined to 0.108, 0.085, 0.059 and 0.026 mg kg^{-1}in 1, 3, 5 and 7 days with dissipation of 20.00, 37.01, 56.30 and 80.76 %, respectively. By the 10th day, residues were below the LOQ. Likewise, residue levels declined progressively over time, following first-order kinetics, with

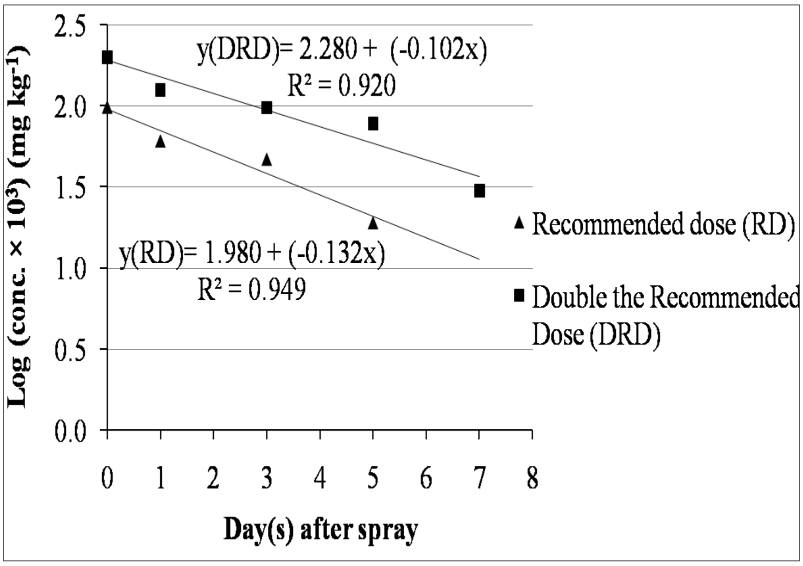

Fig 3. Dissipation kinetics of chlorantraniliprole residues in okra fruits in 2019

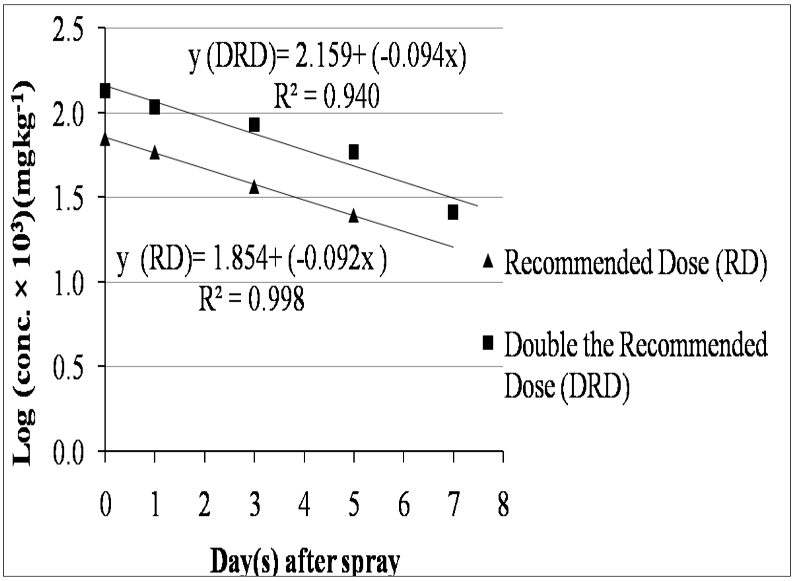

Fig 4. Dissipation kinetics of chlorantraniliprole residues in okra fruits in 2020

dissipation rate constants of 0.212 and 0.216 per day and reached below LOQ within 7 and 10 days after application with half-life values of 3.27 and 3.20 days at the recommended and double dose, respectively (Fig. 4).

Soil

In 2019, the average residues of chlorantraniliprole detected in soil samples on day 0 were 0.031 mg kg^{-1} when applied at the recommended dose of 25 g a.i. ha^{-1} (Table 6). Within 24 h, the residue level decreased to 0.020 mg kg^{-1}, indicating a degradation of 35.48 %. By the 3rd day of spraying, residues were below LOQ (0.01 mg kg^{-1}). At double the recommended dose (50 g a.i. ha^{-1}), the initial residue concentration was 0.055 mg kg^{-1}, which reduced to 0.039 mg kg^{-1} and 0.015 mg kg^{-1} in 1 and 3 days, degrading by 29.09 and 72.73 %, respectively. Within 5 days of application, the residues fell below LOQ. A negative correlation was observed between residue degradation and time with chlorantraniliprole dissipation following first-order kinetics (Fig. 5). The residues degraded gradually with a degradation constant of 0.437 per day. The half-life of chlorantraniliprole residues was calculated to be 1.58 days for both doses.

In 2020, the average initial residues of chlorantraniliprole were higher compared to the previous year. At the recommended dose, the initial residue level was 0.045 mg kg^{-1} on day 0, which decreased to 0.025 mg kg^{-1} and 0.011 mg kg^{-1} within 1 and 3 days, with degradation of 44.58 and 75.58 %, respectively. By the 5th day of spraying, residues in the soil were below LOQ. At double dose, the initial residues on day 0 were 0.073 mg kg^{-1} and declined to 0.051 mg kg^{-1} and 0.021 mg kg^{-1} in 1 and 3 days, resulting in degradation of 29.97 and 71.18 %, respectively. Here also, a similar trend was observed with respect to the dissipation of chlorantraniliprole residues, which dropped below LOQ in 5 days, exhibited negative correlation with time and followed first-order kinetics (Fig. 6). Residues of chlorantraniliprole decreased gradually with a degradation constant of 0.460 and 0.419 per day at respective doses. Half-life of chlorantraniliprole residues in soil was calculated to be 1.51 and 1.65 days at the recommended and double dose, respectively.

Decontamination of chlorantraniliprole residues in okra fruits

Okra fruit samples collected 0 (2 hrs after application), 1 and 3 day(s) after application of chlorantraniliprole at the recommended dose (25 g a.i. ha^{-1}) contained initial residues of 0.099, 0.061 and 0.047 mg kg^{-1}, respectively (Table 7). All household processing methods resulted in considerable removal of chlorantraniliprole residues from the okra fruits. Washing under tap water followed by boiling was most effective in removing 71.72, 65.57 and 63.83 % of residues from okra fruit samples collected 0, 1 and 3 day(s) after application,

Table 6. Dissipation of chlorantraniliprole in soil

Day(s) after spray	RD (25 g a.i. ha^{-1}) Residue level (mg kg^{-1}) *Mean ± SD	RD Dissipation (%)	DRD (50 g a.i. ha^{-1}) Residue level (mg kg^{-1}) Mean ± SD	DRD Dissipation(%)
2019				
0 (2 h)	0.031 ± 0.003	--	0.055 ± 0.003	--
1	0.020 ± 0.003	35.48	0.039 ± 0.005	29.09
3	<LOQ	--	0.015 ± 0.004	72.73
5	--	--	<LOQ	--
	Correlation coefficient (r) = -1.000		Correlation coefficient (r) = -0.997	
	Regression equation (y) = 1.757 ± (- 0.190)x		Regression equation (y) = 1.491 ± (- 0.190)x	
	Half-life value ($T_{1/2}$) = 1.58 days		Half-life value ($T_{1/2}$) = 1.58 days	
	Degradation constant (k) = 0.437		Degradation constant (k) = 0.437	
2020				
0 (2 h)	0.045 ± 0.004	--	0.073 ± 0.002	--
1	0.025 ± 0.003	44.58	0.051 ± 0.003	29.97
3	0.011 ± 0.002	75.58	0.021 ± 0.003	71.18
5	<LOQ	--	<LOQ	--
	Correlation coefficient (r) = -0.996		Correlation coefficient (r) = -0.999	
	Regression equation (y) = 1.631 ± (-0.200x)		Regression equation (y) = 1.874 ± (-0.182x)	
	Half-life value ($T_{1/2}$) = 1.51 days		Half-life value ($T_{1/2}$) = 1.65 days	
	Degradation constant (k) = 0.460		Degradation constant (k) = 0.419	

*Mean of three replications; RD = Recommended dose, DRD = Double the recommended dose; LOQ = Limit of quantification (0.01 mg kg^{-1})

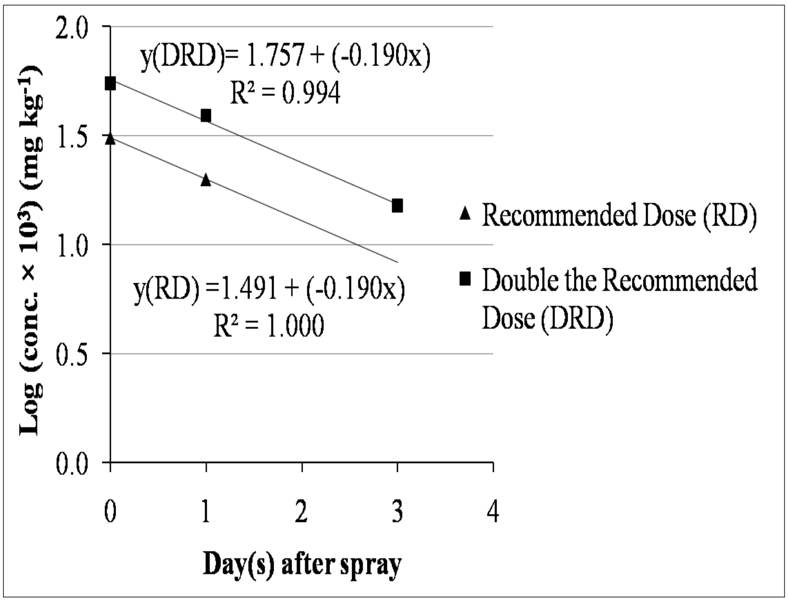

Fig 5. Dissipation kinetics of chlorantraniliprole residues in soil in 2019

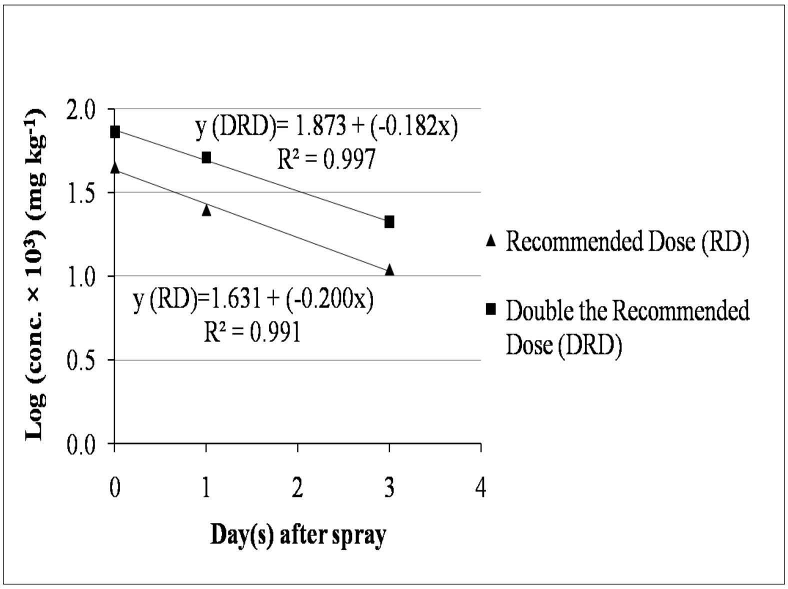

Fig 6. Dissipation kinetics of chlorantraniliprole residues in soil in 2020

Table 7. Effect of household processing method on reduction of chlorantraniliprole residues in okra fruits when applied at recommended dose (25 g a.i. ha^{-1})

Household Processing 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	0.099 ± 0.007	--	0.061 ± 0.005	--	0.047 ± 0.005	--
Washing under tap water	0.045 ± 0.006	54.55	0.030 ± 0.006	50.82	0.025 ± 0.006	46.81
Luke warm water	0.039 ± 0.005	60.61	0.029 ± 0.006	52.46	0.025 ± 0.006	46.81
Salt solution (2 %)	0.035 ± 0.006	64.65	0.025 ± 0.005	59.02	0.021 ± 0.005	55.32
Tamarind solution (2 %)	0.041 ± 0.007	58.59	0.027 ± 0.005	55.74	0.023 ± 0.006	51.06
Lemon water	0.040 ± 0.006	59.60	0.028 ± 0.006	54.10	0.024 ± 0.005	48.94
Vinegar solution (1 %)	0.038 ± 0.007	61.62	0.026 ± 0.005	57.38	0.022 ± 0.005	53.19
Washing ± Boiling	0.028 ± 0.006	71.72	0.021 ± 0.004	65.57	0.017 ± 0.004	63.83

D = Days after spray; *okra fruits were dipped in lukewarm water and different solutions for 5 minutes, followed by washing under tap water.

respectively. Dipping okra fruits in 2 % salt solution followed by washing under tap water was the next effective method, removing 64.65, 59.02 and 55.32 % of residues from fruit samples collected at the respective time intervals. Following to this, dipping okra fruits in 1 % vinegar solution and then washing under tap water removed 61.62, 57.38 and 53.19 % of residues while dipping in 2 % tamarind solution followed by washing under tap water resulted in 58.59, 55.74 and 51.06 % removal of residues from okra fruit samples collected 0, 1 and 3 day(s) after spraying, respectively. Dipping okra fruits in lemon water followed by washing under tap water removed 59.60, 54.10 and 48.94 % of residues, while dipping in lukewarm water followed by washing under tap water resulted in 60.61, 52.46 and 46.81 % removal of chlorantraniliprole residues from samples collected at the corresponding time interval. However, washing under tap water alone proved to be the least effective method, removing only 54.55, 50.82 and 46.81 % of residues from samples collected 0, 1 and 3 day(s) after spraying, respectively.

At double the recommended dose of chlorantraniliprole (50 g a.i. ha^{-1}), mean initial residue level in okra fruits collected 0 (2 hrs after application), 1 and 3 day(s)after spraying were 0.201, 0.126 and 0.098 mg kg^{-1}, respectively (Table 8). Likewise, washing the okra fruits under tap water followed by boiling proved to be most effective in removing the residues to the tune of 72.26, 64.46 and 58.16 % in samples collected at the respective time intervals. Next to this, dipping okra fruits in 2 % salt solution followed by washing under tap water removed 67.77, 62.86 and 57.14 % of residues, while dipping in 1 % vinegar solution followed by washing under tap water resulted in 65.28, 61.27 and 53.06 % removal of residues from samples collected 0, 1 and 3 days after spraying, respectively. Dipping okra fruits in 2 % tamarind solution removed 65.78, 58.89 and 51.02 % residues, while dipping in lemon water followed by washing under tap water resulted in 62.79, 57.29 and 47.96 % removal of residues from the samples collected 0, 1 and 3 day(s) after spraying, respectively. Dipping okra fruits in lukewarm water followed by washing under tap water was comparatively less effective, resulting in 60.80, 54.11 and 43.88 % removal of residues at the corresponding time intervals. However, washing okra fruits under tap water alone remained the least effective method, removing only 54.82, 49.34 and 38.78 % residues from samples collected 0, 1 and 3 days after spraying, respectively.

The persistence and dissipation of insecticide residues depend on various factors such as chemical composition, formulation, dose, plant growth, plant part position, soil conditions and other related factors (24). In the present study, findings on dissipation of chlorantraniliprole 18.5 % SC in okra fruits treated with the recommended (25 g a.i. ha^{-1}) and double the recommended dose (50 g a.i. ha^{-1}) indicated that the residues were below MRL even on the day of application itself, which is desirable considering the short harvesting interval of okra fruits. Under field conditions in Haryana, residues of chlorantraniliprole in okra fruits declined below the LOQ within 7 and 10 days after application, with half-life ranging from 2.28–3.27 days and 2.95–3.07 days at the recommended and double doses, respectively. In soil, residues dropped below the LOQ within 5 days at both doses, with corresponding half-lives of 1.51–1.58 days and 1.58-1.65 days, respectively. These results are confirmed by the earlier studies, revealing that chlorantraniliprole residues in okra fruits dropped below the detection limit within the same period, with a half-life of 2.27 and 2.45 days at the recommended dose and double dose, respectively. In soil, residues reached below the detection limit within 15 days at the recommended dose (25). The half-life of chlorantraniliprole in soil was also reported to be 1.94-1.70 days (26).

Present findings are also in alignment with those of an earlier investigation conducted in Tamil Nadu, where residues of

Table 8. Effect of household processing methods on reduction of chlorantraniliprole residues in okra fruits when applied at double the recommended dose (50 g a.i. ha^{-1})

Household processing methods*	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	0.201 ± 0.006	----	0.126 ± 0.006	----	0.098 ± 0.005	----
Washing under tap water	0.091 ± 0.005	54.82	0.064 ± 0.008	49.34	0.060 ± 0.007	38.78
Luke warm water	0.079 ± 0.005	60.80	0.058 ± 0.005	54.11	0.055 ± 0.004	43.88
Salt solution (2 %)	0.065 ± 0.005	67.77	0.047 ± 0.005	62.86	0.042 ± 0.004	57.14
Tamarind solution (2 %)	0.069 ± 0.004	65.78	0.052 ± 0.006	58.89	0.048 ± 0.005	51.02
Lemon water	0.075 ± 0.007	62.79	0.055 ± 0.005	57.29	0.051 ± 0.005	47.96
Vinegar solution (1 %)	0.070 ± 0.005	65.28	0.049 ± 0.004	61.27	0.046 ± 0.006	53.06
Washing ± Boiling	0.056 ± 0.006	72.26	0.045 ± 0.004	64.46	0.041± 0.004	58.16

D = Days after spray; *okra fruits were dipped in lukewarm water and different solutions for 5 minutes, followed by washing under tap water.

chlorantraniliprole (25 g a.i. ha^{-1}) in okra fruits and soil were reported to fall below the detection limit (0.01 mg kg^{-1}) within 10 and 5 days with corresponding half-life of 2.21 and 2.16 days in fruits and soil, respectively (27). Research also indicates that the residues of chlorantraniliprole declined below the detection limit (0.01 mg kg^{-1}) within 10 days of application at 30 and 60 g a.i. ha^{-1}. The corresponding half-lives were reported as 1.60 and 1.70 days, respectively (25). Variations between the two studies may be attributed to differences in agro-climatic conditions (28, 29, 30) and the applied doses, as Kerala, being a coastal region, experiences higher humidity levels compared to Hisar, Haryana, which lies in a semi-arid zone. Similarly, several researchers have reported that chlorantraniliprole residues declined below the LOQ within 5-10 days in tomato, 15 days in okra, 10 days in cabbage and brinjal (26, 28, 31-33). The variation in the time required to reach the LOQ can be attributed to differences in the dissipation behaviour of chlorantraniliprole 18.5 % SC, which depends on factors such as environmental conditions, application rate and the plant species treated (34, 35).

Research indicates that washing followed by cooking is more effective in removing pesticide residues than washing alone (36). Research also indicates that dipping okra fruits in a 2 % vinegar solution removed over 80 % of chlorantraniliprole residues, making it more effective than 2 % saline or tamarind solutions (28). The variation in results observed in the present study may be attributed to the lower concentration of vinegar (1 %) used. Conversely, earlier work demonstrated that dipping okra fruits in 2 % lemon water was more effective than saline or tamarind solutions in residue removal, which may again relate to differences in the concentration of the lemon juice used. Consistent with previous reports, washing under tap water was found to be the least effective method for decontamination of chlorantraniliprole residues (27). Similarly, in other vegetables, boiling cauliflower and cabbage completely removed chlorantraniliprole residues, proving more effective than washing under tap water (37). Likewise, washing chilli fruits with a 5 % saline solution was reported to be more efficient in residue removal than washing with hot or tap water (38).

Conclusion

The initial residue level of chlorantraniliprole in okra fruits was well below the European Union maximum residual limit (0.3 mg kg^{-1}) on the day of application itself, even at double the recommended dose. Moreover, the residues declined rapidly, dropping below the limit of quantification (0.01 mg kg^{-1}) within 7 and 10 days in okra fruits and 3 and 5 days in soil at recommended and double doses, respectively. This indicates that chlorantraniliprole is safe for use in okra cultivation. To further minimise the potential risk of residues, employing simple household decontamination methods such as boiling of okra fruits or dipping in salt solution before cooking is recommended to ensure consumer safety. It is also suggested that the dissipation behaviour of other commonly used pesticides in okra and related crops be investigated under diverse climatic conditions to enhance food safety assessment.

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Authors' contributions

All authors contributed to the study conception and design. BS, RKG and AJ conceptualised the research and designing of the experiments were designed. BS experimented and collected data. RC helped in the analysis of the data. All authors read and approved the final manuscript.

Compliance with ethical standards

Conflict of interest: Authors do not have any conflict of interests to declare.

Ethical issues: None

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