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Field bioefficacy and residue dynamics of chlorantraniliprole (18.50% SC) in okra (*Abelmoschus esculentus)*

SUJAN MAJUMDER[1]*, A T RANI[1,2], PRATAP A DIVEKAR[1], JAYDEEP HALDER[1], K K PANDEY[1] and T K BEHERA[1]

ICAR-Indian Institute of Vegetable Research, Varanasi, Uttar Pradesh 221 305, India

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India is the leading country in okra (*Abelmoschus esculentus* L.) production and it shares 62% of the total okra produced in the world. The annual production of okra is 6.09 million tonnes and its productivity is 12 tonnes/ha (IHD 2018). The shoot and fruit borer, *Earias vittella*, whiteflies, *Bemisia tabaci* Gennadius and leaf hopper, *Amrasca biguttula* are major pests of okra. Okra fruit borer has shown resistance against many conventional insecticides (Satpute *et al.* 2003). To manage the resistant populations, the farmers resort to application of insecticides more frequently which resulted in an undesirable quantity of pesticide residues. Green chemistry novel insecticide molecules offer immense possibility in managing various crop pests. Some new insecticides, viz. chlorantraniliprole, flubendiamide, emamectin benzoate and spinosad have shown promising results in the vegetable insect pests management. Chlorantraniliprole is an effective insecticide with broad spectrum activity against selected pest species in the order Coleoptera, Diptera and Hemiptera (Lahm *et al.* 2009). Microbial origin insecticides, viz. emamectin benzoate and spinosad have shown good efficacy against fruit borers in tomato, okra and brinjal (Chandan *et al.* 2019). However, their residues may remain in the crops and may cause risk to consumers. Also, there are no reports on their efficacy and residues in okra. Therefore, the present study was carried out to study the bioefficacy of these novel insecticides against fruit borer and major sucking pests under field conditions during 2019–20 at research farm of ICAR-Indian Institute of Vegetable Research, Varanasi, Uttar Pradesh. Subsequently, residue dynamics of chlorantraniliprole on okra fruits were also studied.

The experiment consisted of 7 treatments, viz. T_1, Chlorantraniliprole 18.5 SC @30; T_2, Flubendiamide 39.35 SC @48; T_3, Emamectin benzoate 5 SG @8.5; T_4, Spinosad 45 SC @73; T_5, Cypermethrin 25 EC @43; T_6, Nimbecidine 300 ppm @5 ml/litre; and T_7, Control. At first, insecticide spraying was carried out 45 days after sowing (DAS) when pest population reached Economic Threshold Level (ETL). Observations on the sucking pests and fruit borer infestation were recorded one day prior to spray, 5, 10 and 15 days after each spray coinciding with fruit harvest. The per cent fruit damage (PFD) per plant was calculated using the standard method described by Pradhan and Menon (1945). The border row plants were excluded during observation. The per cent reduction (PR) of the pest was calculated for each treatment after each spray. Marketable okra fruit yields were recorded for each treatment at every picking and pooled data was expressed as tonnes/ha. To study the insecticides residues, 250 g of okra fruits were taken from harvested plants at intervals of 0, 1, 3, 5, 7, 10, 15 and 21 days following pesticide application (Majumder *et al.* 2020). Then, 10 ml of ethyl acetate was added to 10 g of the pulverised fruits in a 50 ml centrifuge tube. After adding 10 g of activated sodium sulphate, the sample was vortexed for 2 min. The mixture was once more vortexed for 2 min, then centrifuged at 5000 rpm for 5 min. Dispersive solid-phase extraction (DSPE) with 75 mg of primary secondary amine (PSA) and 225 mg of magnesium sulphate was used to clean an aliquot of the supernatant ethyl acetate layer (1.5 ml). The extract was centrifuged at 10000 rpm for 5 min and filtered through a 0.2 μm membrane filter and 1 μl of the cleaned extract was injected into GC-μECD. Initial testing for the efficiency of extraction and cleanup processes was done in order to standardize the procedure for estimating the amount of chlorantraniliprole residue from okra fruits (Ghosh *et al.* 2021, Majumder *et al.* 2021). Chlorantraniliprole standard solutions of 0.50, 0.25, 0.10, 0.05, 0.025 and 0.0l μg/ml were used to create calibration curve (Fig 1). The optimal approach was thought to recover pesticides between 80–120% with a relative standard deviation under 20%. The LOQ (Limit

[1]ICAR-Indian Institute of Vegetable Research, Varanasi, Uttar Pradesh; [2]ICAR-Indian Institute of Horticultural Research, Central Horticultural Experiment Station, Chettalli, Karnataka. Corresponding author email: sujaniari@gmail.com

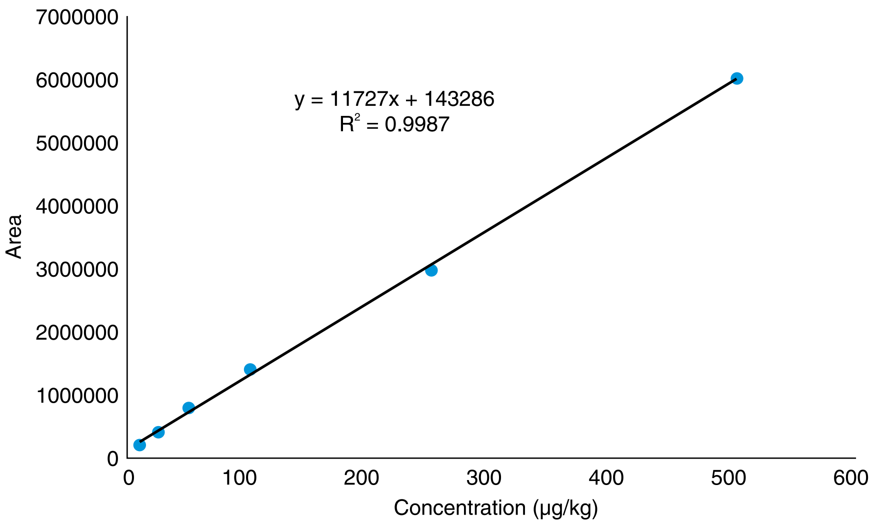

Fig 1 Calibration curve of chlorantraniliprole.

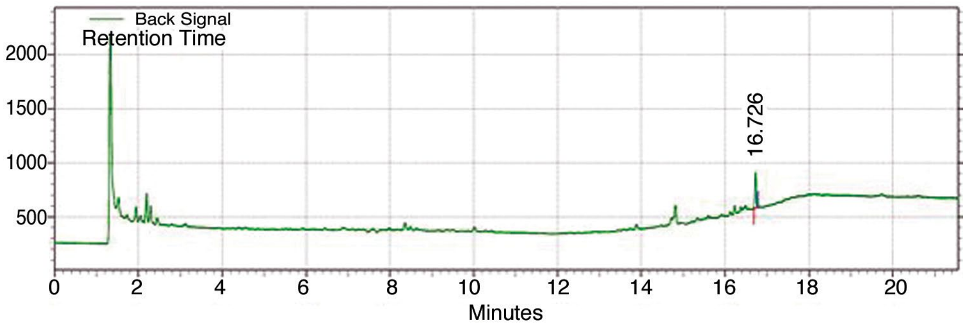

Fig 2 Chromatogram for chlorantraniliprole.

of Quantitation) for okra matrix was determined to be 0.01 µg/ml. The ME was assessed by comparing the peak area response of the solvent standard with that of matrix-matched standard at 0.1 µg/ml (Majumder *et al.* 2022). A 10:1 split ratio of 1 µl of sample extract was injected in GC–µECD. The oven temperature was preheated to 150 ºC for 4 min then ramped up 190ºC at 10ºC/min and held for 4 min before ramping up to 290ºC at 18ºC/min and held for 4 min. Chlorantraniliprole signal appeared at retention time (RT) of 16.72 min (Fig 2). The half-life and Pre-harvest intervals (PHI) on the treated substrate were determined as per the procedure outlined by Majumder *et al.* (2021).

The whitefly population ranged from 3.0–8.0 per three leaves before first spraying. In whitefly population, the highest reduction was observed for chlorantraniliprole 18.5 SC at 30 g a.i./ha (100%), emamectin benzoate 5 SG at 8.5 g a.i/ha (100%) and spinosad 45 SC at 73 g a.i./ha (100%) after third round of spraying. Other insecticides used in the study performed statistically similar to the former (Table 1). Potai *et al.* (2018) demonstrated the satisfactory control of whitefly population with

Table 1 Effect of different novel insecticides on whitefly and leafhoppers of okra

Treatment (g a.i./ha)	Mean number of whiteflies per three leaves							Mean number of leaf hopper per three leaves						
	PTC	1st spray	PROC	2nd spray	PROC	3rd spray	PROC	PTC	1st spray	PROC	2nd spray	PROC	3rd spray	PROC
T_1	8.00	1.31 (1.14)^b	86.44	0.56 (1.00)^b	96.79	0.00 (0.71)^b	100.00	21.93	3.93 (1.98)^b	80.16	5.64 (2.37)^b	82.07	5.42 (2.29)^d	87.81
T_2	7.67	1.93 (1.33)^b	80.00	0.64 (1.05)^b	96.28	0.02 (0.72)^b	99.93	14.93	4.09 (2.01)^b	79.37	6.42 (2.53)^b	79.60	8.89 (2.96)^{bcd}	80.02
T_3	3.00	2.07 (1.39)^b	78.62	0.51 (0.97)^b	97.05	0.00 (0.71)^b	100.00	18.20	4.73 (2.17)^b	76.12	6.80 (2.58)^b	78.41	9.16 (3.02)^{bc}	79.42
T_4	4.67	2.47 (1.57)^b	74.48	0.56 (0.98)^b	96.79	0.00 (0.71)^b	100.00	12.60	4.80 (2.19)^b	75.78	7.60 (2.75)^b	75.86	5.44 (2.32)^d	87.76
T_5	4.00	2.29 (1.51)^b	76.32	1.00 (1.19)^b	94.23	0.04 (0.74)^b	99.87	15.40	4.13 (2.02)^b	79.15	7.73 (2.76)^b	75.44	11.62 (3.40)^b	73.88
T_6	3.00	2.20 (1.46)^b	77.24	0.80 (1.12)^b	95.38	0.04 (0.74)^b	99.87	17.27	4.89 (2.21)^b	75.34	7.22 (2.68)^b	77.06	6.07 (2.46)^{cd}	86.36
T_7	3.67	9.67 (3.10)^a	-	17.33 (4.21)^a	-	33.33 (5.81)^a	-	18.53	19.82 (4.43)^a	-	31.49 (5.61)^a	-	44.49 (6.65)^a	-

PTC, Pre-treatment count; PROC, Per cent reduction over control; Figures in parentheses are X + 0.5 square root transformed values; In column, means followed by same letters are not significantly different at (P = 0.05). Refer to the methodology for treatment details.

Table 2 Effect of different novel insecticides on fruit borer and yield of okra

Treatment (g a.i./ha)	Mean per cent fruit damage (%) on weight basis							Yield (tonnes/ha)
	PTC	1st spray	PROC	2nd spray	PROC	3rd spray	PROC	
T_1	18.07	7.84 (16.16)^{d}	56.43	0.13 (1.93)^{d}	98.91	0.46 (3.10)^{d}	96.04	8.88^{a}
T_2	14.44	12.47 (20.49)^{bc}	30.64	1.39 (6.63)^{c}	88.25	2.18 (8.42)^{cd}	81.24	7.31^{abc}
T_3	13.07	10.84 (18.91)^{cd}	39.74	0.83 (5.10)^{cd}	92.92	3.14 (10.13)^{bcd}	73.03	7.69^{abc}
T_4	16.37	10.71 (19.06)^{cd}	40.46	0.78 (4.73)^{cd}	93.38	1.09 (5.90)^{d}	90.60	8.39^{ab}
T_5	14.46	12.98 (20.89)^{bc}	27.86	6.91 (15.08)^{b}	41.42	7.99 (16.23)^{ab}	31.39	7.28^{abc}
T_6	18.24	15.18 (22.54)^{ab}	15.61	10.57 (18.91)^{a}	10.35	6.46 (14.05)^{abc}	44.51	6.88^{bc}
T_7	16.92	17.99 (25.01)^{a}	-	11.79 (19.97)^{a}	-	11.64 (18.34)^{a}	-	6.28^{c}

PTC, Pre-treatment count; PROC, Per cent reduction over control. Refer to the methodology for treatment details.

chlorantraniliprole in okra under field conditions. Similarly, Lalbabu *et al.* (2017) also stated that spinosad at 60 g a.i./ha was highly effective in reducing the sucking pests incidence on black gram. After third round of insecticide spray, highest decrease in the leafhopper population was recorded in the chlorantraniliprole 18.5 SC at 30 g a.i./ha and spinosad 45 SC at 73 g a.i./ha treated plots (87.81 and 87.76%, respectively) and they were statistically on par with each other (Table 1). Satisfactory control of leafhopper with chlorantraniliprole was demonstrated by Potai *et al.* (2018) in okra under field conditions. Similarly, Babita *et al.* (2018) showed that application of chlorantraniliprole @25 g a.i./ha in okra ecosystem is effective in reducing aphid population after second spray.

The maximum reduction in fruit damage was observed in the plots treated with chlorantraniliprole 18.5 SC at 30 g a.i./ha (96.04%) and spinosad 45 SC at 73 g a.i./ha (90.60%) after 3rd spray and they were statistically on par and found significantly superior over rest of the treatments (Table 2). The results were supported by the findings of Ghuge *et al.* (2020), who reported that chlorantraniliprole (3.30%) recorded minimum percentage of fruit infestation and it was at par with spinetoram (3.81%) and spinosad (4.33%). Similar finding was also reported by Shrivastava *et al.* (2017) that chlorantraniliprole @30 g a.i./ha application on okra was most effective in managing the fruit borer infestation. Similarly, Reddy *et al.* (2019) reported the lowest fruit infestation in okra plots treated with chlorantraniliprole (8.58%). The highest marketable fruit yield of 8.88 tonnes/ha was recorded in the chlorantraniliprole treated plots followed by spinosad with 8.39 tonnes/ha (Table 2).

The average recoveries of the estimation method ranged from 83.67–89.00% with a RSD of 3.53–7.02%. It was discovered that the LOQ was 0.01 mg/kg. The ME evaluated by comparing the peak area response of the solvent standard was found below 20%. The okra fruits initially contained 0.69 mg/kg residue, but it degraded by 9.76% within 24 hours (Table 3), the residue reduction was 29.72% on the third day and after that on the seventh day the fruits were dissipated more quickly (87.78%), and the residue content was 0.08 mg/kg. The reduction in the residues on tenth day was 98.37% in the okra fruits. However, no residue of chlorantraniliprole was found in fifteenth day samples. Half-life and waiting periods, from dissipation kinetics, were computed and its results were 1.72 and 1.10 days respectively. The waiting period was determined using the fixed EU–MRL of 0.60 mg/kg. While the half-life ($t_{1/2}$) was reported to be 16–17 days. Szpyrka *et al*. (2017) found that chlorantraniliprole remained in apples for a longer period of time.

Table 3 Persistence of chlorantraniliprole 18.5% SC residues in okra fruits

Sample collection day	Residues (mg/kg)	Dissipation (%)
(2 hr after spraying)	0.69	
1	0.63	9.76
3	0.49	29.72
5	0.27	60.50
7	0.08	87.78
10	0.01	98.37
15	BDL	
21	BDL	

Bdl, below detection level.

SUMMARY

The okra fruit borer (*Earias vittella*) and sucking pests are a key limiting factor in okra cultivation due to their severe crop destruction behaviour. An experiment was conducted during 2019–20 to assess the bioefficacy of various new insecticides and persistence of chlorantraniliprole 18.50 SC residues in okra fruits at research farm of ICAR-Indian Institute of Vegetable Research, Varanasi, Uttar Pradesh. Among various treatments, chlorantraniliprole tested at 30 g a.i./ha was reported to be effective in reducing fruit damage by *E. vittella* (96%), as well as sucking pests infestation (87.81–1100%) with a significantly higher marketable fruit yield (8.88 t/ha) recorded under open field situations. Ethyl acetate was used to extract the chlorantraniliprole contaminants from the okra fruit matrix,

and PSA and magnesium sulphate were used to clean up the matrix. The estimate for the limit of quantification (LOQ) was 0.01 mg/kg, and the average per cent recoveries ranged from 83.67–89.00. The half-life was estimated 1.72 days. Based on the field bioefficacy against insect pests and residue status in okra fruits, it can be concluded that the chlorantraniliprole 30 g a.i./ha can be utilized in the okra ecosystem to successfully manage the sucking pest complex and fruit borer without posing any health risks to consumers.

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