Bull Environ Contam Toxicol (2009) 82:243–247
DOI 10.1007/s00128-008-9581-8

Persistence of New Insecticides and Their Efficacy Against Insect Pests of Okra

Suman Gupta · R. K. Sharma · R. K. Gupta · S. R. Sinha · Rai Singh · V. T. Gajbhiye

Received: 25 January 2008 / Accepted: 8 October 2008 / Published online: 23 October 2008

Abstract Persistence and efficacy of bifenthrin (25 and 50 g ai ha^{-1}), fipronil (50 and 100 g ai ha^{-1}) and indoxacarb (70 and 140 g ai ha^{-1}) has been studied in okra fruits. The initial deposits varied from 0.259–0.382 µg g^{-1} at low and 0.461–0.688 µg g^{-1} at high rate of application. The residues persisted upto 10 days with half-life of 1.32–1.58 days for bifenthrin, 0.65–1.12 days for fipronil and 0.58–1.02 days for indoxacarb. Based on ADI, the suggested waiting period was 1 day for bifenthrin and indoxacarb and 3 days for fipronil. All the insecticides were found effective against leafhopper and shoot and fruit borer.

Keywords Bifenthrin · Fipronil · Indoxacarb · Okra · Persistence · Bioefficacy

Okra *Abelmoschus esculantus* (L.) (Moench) is a major economically important vegetable crop. It alone accounts for 21% of the exchange earnings from export of vegetables from India. Insect pests pose major constraint to the production of this important export oriented crop. The major pests are leafhopper *Amrasca biguttula biguttula* (Ishida), whiteflies *Bemisia tabaci* (Gennadius) (vector of vein clearing disease) and shoot and fruit borer, *Earias vittella* (Fabricius). The shoot and fruit borer is the main pest of maturing fruits. The larva bores into shoot or fruit and start eating on internal contents causing withering of plant and reduction in marketable value of the fruit. Various insecticides like cypermethrin, endosulfan, quinalphos, imidacloprid, etc. have been recommended for the management of these pests in okra (Prasad et al. 1993; Kumar and Singh 2001; Bhargava and Bhatnagar 2001). Vegetable growers by and large undertake many sprays of chemical pesticides to counter the problem of insect pests. Indiscriminate use of pesticides leads to undesirable load of pesticide residues in marketable vegetables (Kumari et al. 2002). Therefore, there is a need for use of pesticides requiring low dose and less number of sprays.

Bifenthrin, fipronil and indoxacarb (Fig. 1) are relatively new introductions and have shown promise in the management of insect pests of vegetables. Fipronil belongs to phenylpyrazole group of insecticides and has been found effective for the management of DBM on cauliflower (Bharadwaj et al. 2005), shoot and fruit borer in brinjal (Sahu et al. 2004) and thrips and mites in chillies (Reddy et al. 2005). Indoxacarb belongs to oxadiazine group of insecticides and has been used to control DBM in cabbage (Murthy et al. 2006), leaf folder and fruit borer in chillies (Singh et al. 2005b) and also tomato fruit borer (Singh et al. 2005a). Bifenthrin is a third generation synthetic pyrethroid having applications both in agriculture and in public health control. It has shown good bioefficacy against insect pests of brinjal (Sudhakar et al. 1998) and tomato (Rushtapakornchai and Petchwichit 1996). However, there are no reports on their efficacy and residues in okra. Therefore, present investigation was undertaken to study the persistence of bifenthrin, fipronil and indoxacarb in okra and their efficacy against leafhopper and shoot and fruit borer.

S. Gupta (✉) · R. K. Gupta · V. T. Gajbhiye
Division of Agricultural Chemicals, Indian Agricultural Research Institute, New Delhi 110012, India
e-mail: drsumangupta2002@yahoo.com

R. K. Sharma · S. R. Sinha · R. Singh
Division of Entomology, Indian Agricultural Research Institute, New Delhi 110012, India

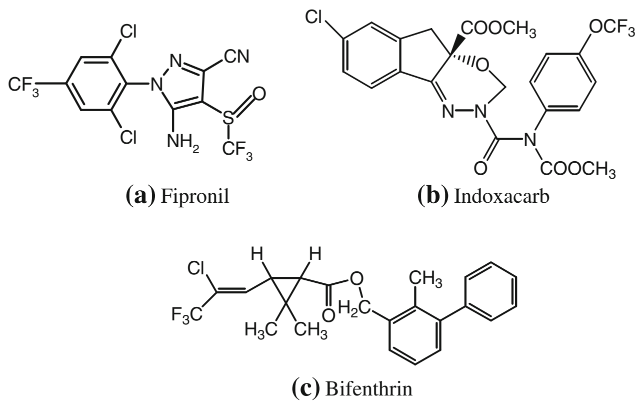

Fig. 1 Chemical structures of (**a**) fipronil (**b**) indoxacarb and (**c**) bifenthrin

Materials and Methods

Investigations were conducted in *kharif,* 2006 (July–October) in the vegetable farm of Division of Entomology, IARI, New Delhi. The okra crop (variety A4) was raised in randomized block design with seven treatments replicated thrice in 20 m^2 plot size. Appropriate agronomic practices were followed for raising the okra crop. The insecticides used were fipronil (Regent 5 SC, @ 50 and 100 g ai ha^{-1}), bifenthrin (Talstar 10 EC, @ 25 and 50 g ai ha^{-1}) and indoxacarb (Avaunt 14.5 SC, @ 70 and 140 g ai ha^{-1}). Two foliar sprays were given at fortnightly interval beginning at flowering/fruiting stage. A control with only water spray was maintained for comparison.

For residue analysis, the samples of fruits were drawn on 0, 1, 3, 7, 10 and 15 days after the second insecticide application. The fruits were cut into small pieces and a representative 50 g sample was drawn. The samples were transferred to the jar of the mixer grinder and 100 mL distilled acetone was added. The contents were macerated for 3 min and filtered through Whatman No. 1 filter paper using buchner funnel. The solid residues were transferred back to the jar and process of extraction was repeated two more times using fresh 50 mL acetone. The acetone extracts were combined and concentrated using rotary evaporator. The concentrated extract was quantitatively transferred to separatory funnel, diluted with 100 mL 10% aqueous NaCl solution and partitioned thrice with dichloromethane (50 mL each). The dichloromethane phases were combined and concentrated to 1–2 mL using rotary evaporator.

The extracts were cleaned by adsorption column chromatography using silica gel/neutral alumina as adsorbents. For fipronil and bifenthrin samples, silica gel (10 g) was wet packed in between two 2 cm layers of anhydrous sodium sulfate and column was washed with 50 mL distilled hexane. The dichloromethane extract was concentrated to dryness, residues dissolved in 1–2 mL hexane and loaded on to the column. To ensure complete transfer of residues, flask was washed two more times using small portions of hexane and the washings were transferred to the column. The column was eluted with a mixture of hexane and acetone (9:1) for bifenthrin and with a mixture of hexane and acetone (4:1) for fipronil. For cleanup of indoxacarb samples, 10 g neutral alumina was wet packed in between two 2 cm layers of anhydrous sodium sulfate and column was washed with 50 mL distilled hexane. Sample dissolved in 2–3 mL hexane was transferred to the column and the column was further washed with 10% acetone:hexane mixture (50 mL). All the washings were discarded and finally the column was eluted with 100% acetone (100 mL).

Residues of bifenthrin, fipronil and indoxacarb were analysed on Varian CP 3800 gas chromatograph (GLC) equipped with electron capture detector and CP-Sil 5 CB (25 m × 0.25 mm × 0.25 μm) column. The operating temperatures were: detector 300°C, injector 275°C and column programmed as 220°C for 6 min, increased @ 20°C/min to 260 and hold for 2 min for bifenthrin, 200°C for 6 min, increased @ 20°C/min to 260 and hold for 2 min for fipronil and 260°C for 10 min, increased @ 20°C/min to 270 and hold for 2 min for indoxacarb. The carrier gas was high purity nitrogen at a flow rate of 2 mL per min. Under these conditions, the retention time was 6.02 min for bifenthrin, 3.81 min for fipronil and 5.64 min for indoxacarb (Fig. 2). Limit of detection of fipronil, bifenthrin and indoxacarb was 0.010, 0.015 and 0.020 ng, respectively.

The cleaned extracts were concentrated to dryness and re-dissolved in 5 mL of hexane. Samples along with standard solutions were injected and retention time and peak area was recorded.

For bioefficacy evaluation, population of leafhoppers was recorded from five plants per plot, one leaf each from top, middle and bottom. The observations of leafhoppers were taken 1 day before and 1st, 7th and 14th day after second spray. Population reduction over control (PROC) was calculated by the formula (Flemming and Retnakaran 1985): PROC (%) $= 100 \times 1 - (T_a \times C_b)/(T_b \times C_a)$, where $T_a =$ Population in treatment after spray; $T_b =$ Population in treatment before spray; $C_a =$ Population in control after spray; $C_b =$ Population in control before spray. For assessing fruit borer infestation, picking of fruits was undertaken at 3 days interval. Observations of healthy and damaged fruits were recorded on number and weight basis and percent damage were calculated.

The residue data was subjected to regression analysis (linearised form of first order kinetics equation) and half-life values were calculated. The bioefficacy data was statistically analysed using AGRES software after angular transformation of the percentage data.

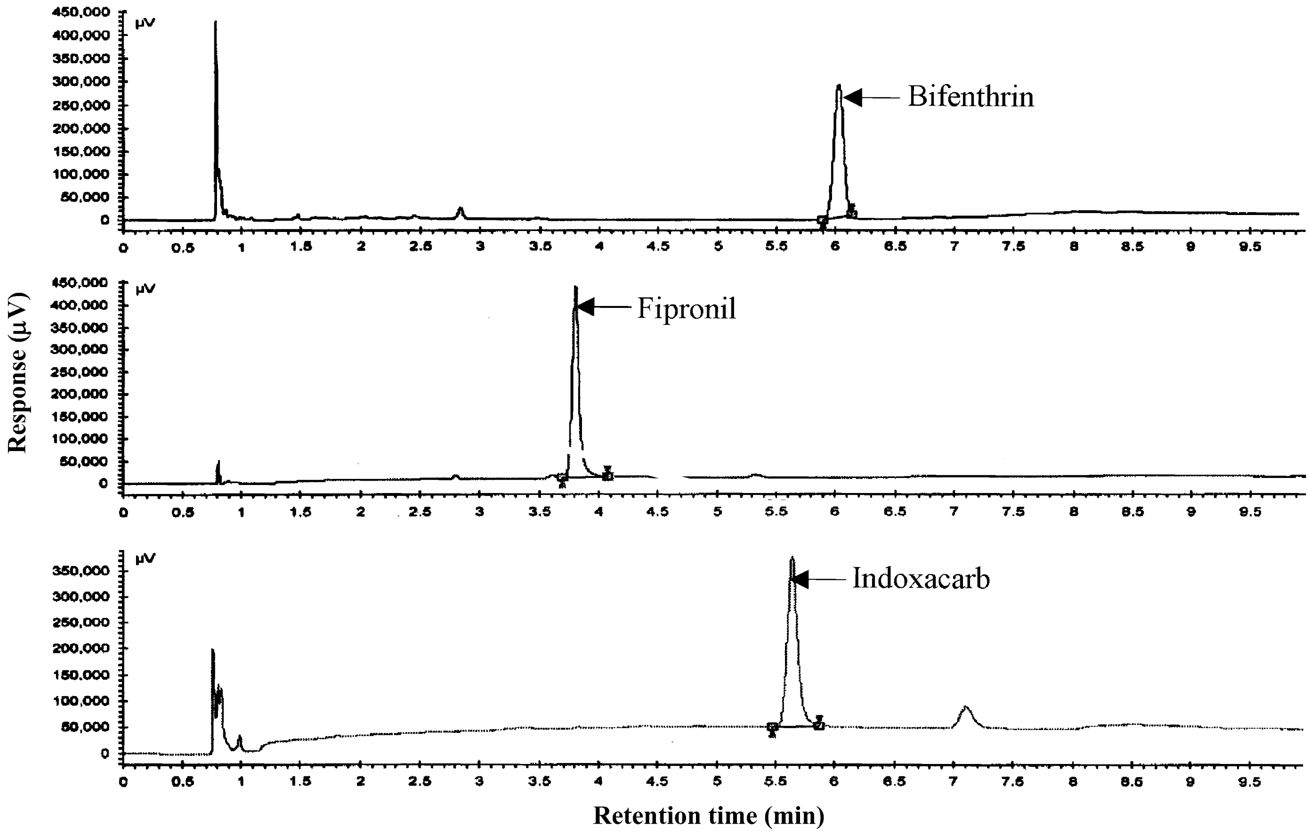

Fig. 2 GLC chromatogram of bifenthrin, fipronil and indoxacarb

Results and Discussion

The recovery of bifenthrin, fipronil and indoxacarb from okra fruits fortified at 0.5 μg g^{-1} varied from 83% to 91%, 86% to 89% and 88% to 92%, respectively. The residue data is presented in Table 1.

Following application of insecticides at recommended and double the recommended dosages on okra crop, the initial deposits (2 h after application) of bifenthrin on brinjal fruits were 0.382 and 0.688 μg g^{-1} (Table 1). The residues dissipated with time and non-detectable (ND) and 0.007 μg g^{-1} residues were detected on 10th day, accounting for the loss of 99%–100%. However, on 15th day no residues were detected in the marketable fruits. Similarly, the initial deposits of fipronil were 0.275 and 0.461 μg g^{-1} at low and high rate of application, which dissipated to non-detectable (ND) and 0.007 μg g^{-1} on 7th day amounting to the loss of 98.5%–100% (Fig. 3). The initial deposits of indoxacarb were 0.259 and 0.674 μg g^{-1} following application at recommended and double the recommended dosages, which dissipated to non-detectable level and 0.005 μg g^{-1} on 7th day amounting to the loss of 99%–100%. No residues of fipronil and indoxacarb were detected in okra fruits 10 day onwards (Table 1). The dissipation of all the pesticides was faster probably due to dilution effect of faster weight gain in okra fruits.

Table 1 Residues of insecticides in okra fruits

Insecticides	Days dosage (g ai ha^{-1})	Residue (μg g^{-1})					
		0	1	3	7	10	15
Bifenthrin	25	0.382	0.134	0.052	0.008	ND	ND
Bifenthrin	50	0.688	0.159	0.083	0.011	0.007	ND
Fipronil	50	0.275	0.131	0.012	ND	ND	ND
Fipronil	100	0.461	0.286	0.021	0.007	ND	ND
Indoxacarb	70	0.259	0.115	0.008	ND	ND	ND
Indoxacarb	140	0.674	0.238	0.084	0.005	ND	ND

The dissipation of residues of all the insecticides followed first order kinetics with correlation coefficient (*r*) values more than 0.9 (Table 2). The half-life values for low and high doses were 1.32 and 1.58 days for bifenthrin, 0.65 and 1.12 days for fipronil, 0.58 and 1.02 days for indoxacarb. As the insecticides are new introductions, maximum residue limit (MRL) values are not available. Therefore, the waiting period has been calculated based on acceptable daily intake values (ADI) taken from the literature (Anonymous 2007). Based on the calculations (Table 3), a waiting period of 1 day is suggested for bifenthrin and indoxacarb as the initial deposits itself were safe. For

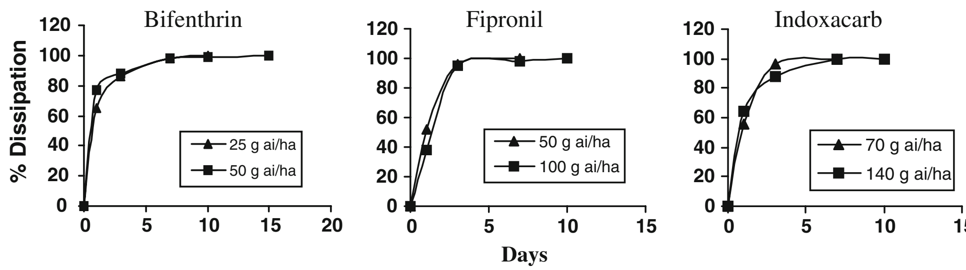

Fig. 3 Dissipation of bifenthrin, fipronil and indoxacarb in okra

Table 2 Regression equation for first order dissipation of insecticides on okra fruits

Insecticides	Dose (g ai ha$^{-1)}$	Regression equation	Correlation coefficient	Half-life (days)
Bifenthrin	25	Y = −0.5408 − 0.2280*X*	0.989	1.32
	50	Y = −0.4333 − 0.1899*X*	0.966	1.58
Fipronil	50	Y = −0.5044 − 0.4628*X*	0.994	0.65
	100	Y = −0.4415 − 0.2679*X*	0.944	1.12
Indoxacarb	70	Y = −0.5221 − 0.5142*X*	0.994	0.58
	140	Y = −0.2311 − 0.2952*X*	0.997	1.02

Table 3 Maximum permissible intake (MPI) and theoretical maximum residue contribution (TMRC) values for insecticides on okra

Insecticide	ADI	MPI	Dose (g ha^{-1})	TMRC					Waiting period (days)
				0 day	1 day	3 days	7 days	10 days	
Bifenthrin	0.02	1.00	25	0.096	0.034	0.013	0.002	–	1
			50	0.172	0.040	0.021	0.003	0.002	1
Fipronil	0.0002	0.01	50	0.069	0.033	0.003	–	–	3
			100	0.115	0.072	0.005	0.002	–	3
Indoxacarb	0.01	0.50	187	0.065	0.029	0.002	–	–	1
			375	0.169	0.060	0.021	0.001	–	1

MPI (mg per person per day) = ADI × Average body weight (50 kg); TMRC (mg per person per day) = Residues × Average daily consumption (250 g); residues safe when TMRC < MPI

fipronil, a waiting period of 3 days is suggested as harvesting of okra fruits is normally carried out at 3 days interval.

Perusal of data revealed that after II spray the population reduction over control (PROC) of leafhoppers (*Amrasca biguttula biguttula* Ishida) varied from 9.9% to 100% at one day and 66.7% to 100% on 7th day after spraying (Table 4). PROC of different treatments on 14th DAS varied between 30.7% and 66.7%. Bifenthrin reduced the leafhopper population by 76.1% and 52.2% on 1st day following application @ 25 (T_1) and 50 g ai ha^{-1} (T_2), respectively. The reduction further increased to 100% on 7th day. Population reduction was 64.1% in both the treatments of bifenthrin on 14th day. Both doses of fipronil (T_3 50 and T_4 100 g ai ha^{-1}) showed 100% leafhopper reduction on 1st and 7th day, while it was 30.7% and 64.8% on 14th day with low and high rate of application, respectively. Indoxacarb at both doses (T_5 70 and T_6 140 g ai ha^{-1}) exhibited PROC of 16.7% and 9.9% on 1st day, 76.1% and 66.7% on 7th day and 38.5% and 66.4% on 14th day, respectively. Bifenthrin at both dosages was effective against leafhopper. Effectiveness of bifenthrin against *A. biguttula biguttula* has already been reported on brinjal (Sudhakar et al. 1998) and cotton (Saleem et al. 2001). Foliar sprayings of indoxacarb has also been found effective in managing leafhopper (Bharpoda et al. 2003).

The impact of damage due to shoot and fruit borer (*Earias vittella* Fab.) in various treatments is also summarized in Table 4. The infestation (on weight basis) in various treatments ranged from 10.6 to 21.3, while it was 28.0% in control.

There are no reports pertaining to the efficacy of fipronil on *E. vittella.* However, it is considered for use against major lepidopterous and orthopterous on a wide range of field and horticultural crop (Anonymous 1996). Bifenthrin has been reported to be effective against *E. vittella* (Afzal et al. 1995). Bharpoda et al. (2003) reported that foliar

Table 4 Impact of insecticides on population of leafhopper, *Amrasca biguttula biguttula* Ishida and infestation by shoot and fruit borer, *Earias vittella* Fab

Treatment	Dose (g ai ha^{-1})	% Reduction of leafhopper population over control (PROC)			Damage due to shoot borer (%)	
		1 DAS	7 DAS	14 DAS	(Weight)	(Number)
Bifenthrin	25	76.1	100.0	64.1	19.1 (25.9)	16.1 (23.5)
Bifenthrin	50	52.2	100.0	64.1	[a]	[a]
Fipronil	50	100.0	100.0	30.7	10.6 (19.0)	12.8 (20.8)
Fipronil	100	100.0	100.0	64.8	[a]	[a]
Indoxacarb	70	16.7	76.1	38.5	21.3 (27.4)	20.5 (27.0)
Indoxacarb	140	9.9	66.7	66.7	14.8 (22.6)	16.2 (23.6)
Control	–	–	–	–	28.0 (31.8)	32.9 (35.0)
CD(0.05)					5.6	5.8

[a] Denotes observation not taken, dose used for residue purpose only

sprays of indoxacarb at 75 g ai ha^{-1} gave significantly superior control over other evaluated insecticides.

The study revealed that bifenthrin, fipronil and indoxacarb could be used for the management of leafhopper and fruit borer in okra. From toxic residue point of view all the treatment were found safe with waiting period of 1–3 days.

References

Afzal M, Rasheed A, Ullah MS (1995) The comparative efficacy of some latest spray-schedules against the bollworms of FH-682 cotton. Pak Entomol 17:87–89

Anonymous (1996) 'Fipronil' Worldwide technical bulletin. Rhone-Poulenc, Research Triangle Park, p 20

Anonymous (2007) Codex alimentarius: notes on Pesticides: bifenthrin—http://www.codexalimentarius.net/mrls/pestdes/pest_ref/prfe_178.htm; fipronil—http://www.codexalimentarius.net/mrls/pestdes/pest_ref/prfe_202.htm; indoxacarb—www.who.int/entity/ipcs/food/jmpr/summary_2005.pdf

Bharadwaj V, Devi N, Raj D (2005) Effect of insecticides/bio-pesticides on the diamondback moth, *Plutella xylostella* (Linn.), and its parasitoid complex. Pest Manag Econ Zool 13(2):231–234

Bhargava KK, Bhatnagar A (2001) Bioefficacy of imidacloprid as a seed dresser against sucking pests of okra. Pest Manag Econ Zool 9(1):31–34

Bharpoda TM, Sisodiya DB, Patel JR (2003) Effectiveness of indoxacarb 15% SC in controlling insect-pests of 'H 6' upland cotton (*Gossypium hirsutum*). Indian J Agric Sci 73(4):203–205

Flemming R, Retnakaran A (1985) Evaluating single treatment data using Abbot's formula with reference to insecticides. J Econ Entomol 78:1179–1181

Kumar M, Singh AK (2001) Bioefficacy of insecticides against the cotton jassid, *Amrasca biguttula biguttula* (Ishida) on okra. Pest Manag Econ Zool 9(1):55–58

Kumari B, Madan VK, Kumar R, Kathpal TS (2002) Monitoring of seasonal vegetables for pesticide residues. Earth Environ Sci 74(3):263–270

Murthy MS, Jagadish PS, Sannaveerappanavar VT (2006) Efficacy of plant products and new insecticides against diamondback moth, *Plutella xylostella* (L.) (Yponomeutidae: Lepidoptera) on cabbage. J Plant Prot Environ 3(2):1–5

Prasad RT, Reddy GPV, Murthy MMK, Prasad VD (1993) Efficacy and residues of certain newer insecticides on okra. Indian J Plant Prot 21(1):47–50

Reddy AV, Sreehari G, Kumar AK (2005) Evaluation of certain new insecticides against chilli thrips (*Scirtothrips dorsalis*) and mites (*Polyphagotarsonemus latus*). Res Crops 6(3):625–626

Rushtapakornchai W, Petchwichit P (1996) Efficiency of some insecticides for controlling tobacco whitefly *Bemisia tabaci* and leaf miner *Liriomyza trifolii* on tomato. Kaen Kaset Khon Kaen Agric J 24(4):184–189

Sahu BB, Senapati B, Mohapatra LN (2004) Bioefficacy of certain insecticides against brinjal shoot and fruit border *Leucinodes orbonalis* Guen. Orissa J Hortic 32(2):81–86

Saleem M, Ahmad AS, Khan AH, Mustafa K, Husain R (2001) Effect of different insecticides against insect pests of CIM–443, cotton. Pak Entomol 23:87–89

Singh S, Choudhary DP, Mathure YS (2005a) Efficacy of some newer insecticides against fruit borer, *Helicoverpa armigera* (Hubner) on tomato. Indian J Entomol 67(4):339–341

Singh V, Thakur BS, Chandraker MK (2005b) Bio-efficacy of insecticides against insect pests of chilli. Environ Ecol 23S(Special 3):600–604

Sudhakar K, Punnaiah KC, Krishnayya PV (1998) Efficacy of certain selected insecticides on the sucking pest complex of brinjal. Indian J Entomol 60(3):241–244