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Dissipation of insecticides in okra fruits

S. A. PANDEY, [1]P. MISHRA AND V. K. KOSHTA

Department of Entomology, College of Agriculture, IGKVV, Raipur -492012, Chhattisgarh
[1]College of Agriculture, JNKVV, Powarkheda-461110, Hoshangabad, Madhya Pradesh

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ABSTRACT

Dissipation behavior of newer insecticide molecules (emamectin benzoate, flubendiamide, indoxacarb, bifenthrin, deltamethrin and lambda-cyhalothrin) applied at different doses in okra crop during Rabi season in year 2014-15 was studied at 0,1,3,5,7,10,14 and 15 days after treatment. Dissipation was maximum on 10^{th} day in emamectin benzoate (99.97%) followed by bifenthrin 10^{th} day (97.73%), indoxacarb 7^{th} day (96.66), deltamethrin 14^{th} day (96.15%), flubendiamide 10^{th} day (95.00%) and lambda-cyhalothrin 10^{th} day (89.79%). Half life ($t_{1/2}$) values for the above insecticides were 0.6, 2.18, 1.32 to 1.58, 5.9 to 17, 5.5 and 5.0, respectively.

***Keywords*:** Dissipation, gas chromatography, half-life period, insecticides, mass spectrometry and okra

Okra (*Abelmoschus esculentus*) is popularly known as bhindi, lady's finger *etc*. It is the only vegetable crop of significance in the Malvaceae family and is very popular in the Indo-Pak subcontinent. In India, it ranks number one in its consumption but its original home is Ethiopia and Sudan, and North-eastern African countries. Medicinal plants are the nature's gift to human being to have disease-free healthy life. It is used in the treatment of catarrhal infections, dysuria and gonorrhoea. (Okra) is an important medicinal plant of tropical and subtropical India. Its medicinal usage has been reported in the traditional systems of medicine such as Ayurveda, Siddha and Unani (Kumar *et al.,* 2013). Okra dry seed contains good edible oil (13-22%) and protein (20-24%). The oil is used in soap, cosmetic industry and as vanaspati while protein is used for fortified feed preparations. High iodine content of fruits helps to control goitre while leaves are used in inflammation and dysentery. (Mishra, 2001). There are several constraints in the cultivation of okra. Many of the pests occurring on cotton are also found on okra crop. As high as, 72 species of insects have been recorded on okra (Srinivasa Rao and Rajendran, 2002), of which, the sucking pests *viz*. Aphids, *Aphis gossypii* (Glover); leafhopper, *Amrasca biguttula biguttula* (Ishida); whitefly, *Bemisia tabaci* (Gennadius); shoot and fruit borer, *Earias vittella* and mite *Tetranychus cinnabarinus* (Boisduval) causes significant damage to the crop.

MATERIALS AND METHODS

Field study was carried out in the experimental field of Department of Horticulture, College of Agriculture, Indira Gandhi Krishi Vishwavidyalaya (IGKV), Raipur, Chhattisgarh (C.G.) during *kharif* 2014-15. The research field located in the south eastern part of Chattisgarh and lies at 21- 16^{0}N latitude and 81.36^{0}E longitude with an altitude of 298 meter above mean sea level. The experiment was laid out in a Randomized Block Design with three replications having a plot size 4.0 x 3.0m each with 0.5 meter pathway between plots. Before spraying, okra fruits in all plots or replicates were tagged and sprayed with recommended doses of insecticides and spark at 1L ha^{-1} to runoff stage.

Suitable okra fruit samples of marketable size from all the treatments of emamectin benzoate 5SG, flubendiamide 48SC, indoxacarb 14.5SC, bifenthrin 10EC, deltamethrin 2.8EC, lambda-cyhalothrin 5EC and untreated control were collected at 0,1,3,5,7,10,14 and 15 days after spraying. The treatment wise samples were properly mixed and used for sample preparation.

A representative 50g of chopped and macerated okra fruits was placed overnight into a mixture of 50 ml of methanol, 50 ml of acetonitrile, 50 ml of methanolic solution of 1% ammonium acetate and 1 ml of triethanol amine in an Erlenmayer flask. The extract was filtered through glass wool plugged in filtering funnel into one litre capacity of seperatory funnel. The residual material was rinsed with methanol and transferred to the same seperatory funnel. The contents of seperatory funnel were diluted with 600 ml of brine solution and partitioned thrice into 100,75 and 75 ml of dichloromethane and was dried over anhydrous sodium sulphate. The organic layer was collected into a 500 ml beaker and dried over anhydrous sodium sulphate. The extracts were cleaned up by using silica gel as an adsorbent. Before use, silica gel was activated at 110^{0} C for 2 hours. A glass column (60 x 1.5 cm i.d.) was packed with activated silica gel (10-12g) in between the two small layers of anhydrous sodium sulphate supported on glass wool. The column was pre-washed with dichloromethane, following which the concentrated extract was poured over it. The glass beaker was rinsed with dichloromethane and the extract was transferred to the column. The extract was eluted with a freshly prepared solvent mixture of

Email: sagaranand687@gmail.com

dichloromethane and ethyl acetate (2:1, v/v). The received filterate was concentrated to near dryness on a rotator evaporator under vacumm at 35^{0}C to remove completely the traces of dichloromethane and the final volume was reconstituted to 5 ml with HPLC-grade methanol.

The cleaned extracts were analysed on gas chromatography mass spectromectry (GCMS) equipped with capillary columns using electron capture detector (ECD) and nitrogen-phosphorous detector (NPD). Operating conditions were as per details for following insecticides *viz*., emamectin benzoate, flubendiamide, indoxacarb, bifenthrin, deltamethrin and lambda-cyhalothrin. Column temperature (25^{0}C, 30^{0}C, 260^{0}C, 220^{0}C, 260^{0}C and 260^{0}C, respectively) Pump flow (0.5, 0.5, 2.0, 2, 40 and 40 ml min^{-1}, respectively) wavelength (245, 210, 310, 230, 280 and 270-290 nm, respectively) Injection port (20, 10, 300, 20, 300 and 300 micro litre, respectively) Detector (293, 240, 310, 300, 300 and 300^{0}C, respectively) and retention time (11.0, 10.10, 5.72, 6.10, 3.65 and 19.30 minutes, respectively).

RESULTS AND DISCUSSION

The dissipation patterns of different insecticides are presented in the table 1. The average initial spraying deposit of emamectin benzoate 5SG @ 12g *a.i.* ha^{-1}, observed as 0.09ppm for samples collected at 0 days (2 hours) after application. The emamectin benzoate 5SG @ 12g *a.i.* ha^{-1} gradually declined its dissipation in okra fruits to the level of 0.03, 0.0025, 0.000625, 0.00015 and 0.000019 ppm with the reduction of 66.66, 97.22, 99.30, 99.83 and 99.97 per cent after the spray of 1,3,5,7 and 10 days, respectively. However, no residues in okra fruits were detected on 14th and 15th day sample. The half-life value for emamectin benzoate was 0.6 days @ 12g *a.i.* ha^{-1} below the permissible maximum residue limit (MRL) of 0.02ppm.

Table 1: Dissipation of insecticides in/on okra fruit after different dates of spraying

Treatments	Insecticide residues (ppm)								Half life
	0 Day	1 Day	3 Days	5 Days	7 Days	10 Days	14 Days	15 Days	(Days)
Emamectin benzoate @ 12 g *a.i.* ha^{-1}	0.09	0.03 (66.66)	0.0025 (97.22)	0.000625 (99.30)	0.00015 (99.83)	0.000019 (99.97)	BDL	BDL	0.6
Flubendiamide @ 55 g *a.i.* ha^{-1}	6.00	4.50 (25.00)	1.30 (78.33)	1.10 (81.66)	0.7 (88.33)	0.30 (95.00)	BDL	BDL	5.5
Indoxacarb @ 50 g *a.i.* ha^{-1}	1.50	0.76 (49.33)	0.28 (81.33)	0.09 (94.00)	0.05 (96.66)	BDL	BDL	BDL	1.32 to 1.58
Bifenthrin @ 25 g *a.i.* ha^{-1}	0.53	0.32 (39.62)	0.20 (62.26)	0.09 (83.01)	0.03 (94.33)	0.012 (97.73)	BDL	BDL	1.58 to 2.18
Deltamethrin @ 15 g *a.i.* ha^{-1}	0.52	0.28 (46.15)	0.18 (65.38)	0.12 (76.92)	0.10 (80.76)	0.06 (88.46)	0.02 (96.15)	BDL	5.9 to 17
Lambda-cyhalothrin @ 15 g *a.i.* ha^{-1}	2.45	1.23 (49.75)	0.66 (73.06)	0.50 (79.59)	0.38 (84.48)	0.25 (89.79)	BDL	BDL	5.0
SEm (±)	**3.22**	**2.12**	**2.30**	**3.61**	**4.01**	**5.37**	**2.46**	**1.17**	**1.32**
LSD (0.05)	**8.88**	**6.77**	**7.23**	**9.00**	**6.49**	**11.89**	**4.19**	**3.38**	**3.31**

Note: MRLvalues for emamectin benzoate – 0.02ppm, flubendiamide – 4ppm, indoxacarb – 0.5ppm, bifenthrin – 0.4ppm, deltamethrin – 0.2ppm, lambda-cyhalothrin – 1ppm; MRL value of rynaxypyr insecticide could not be traced out presently in the available literature by the student. Hence, dissipation analysis has not been undertaken. BDL – Below Detectable Limit

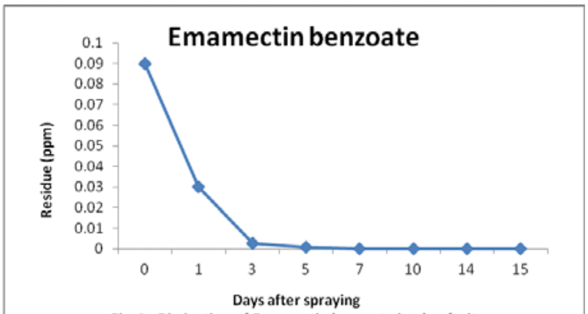

Fig. 1: Dissipation of Emamectin benzoate in okra fruits

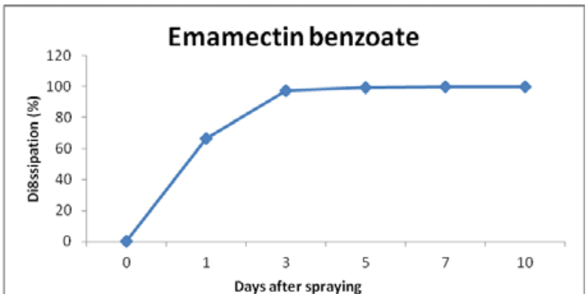

Fig. 2: Percentage dissipation of Emamectin benzoate in okra fruits

The initial spraying deposit of flubendiamide 48SC @ 55g *a.i.* ha^{-1}, evaluated as 6ppm for samples collected at 0 days (2 hours) after application. The flubendiamide 48SC @ 55g *a.i.* ha^{-1} gradually declined its dissipation in okra fruits to the level of 4.5, 1.3, 1.1, 0.7 and 0.3 ppm with the reduction of 25.00, 78.33, 81.66, 88.33 and 95.00 per cent after the spray of 1,3,5,7 and 10 days, respectively. However, no residues in okra fruits were detected on 14th and 15th day sample. The half-life value for flubendiamide was 5.5 days @ 55g *a.i.* ha^{-1} below the permissible maximum residue limit (MRL) of 4ppm.

Average deposition of indoxacarb in its initial spraying at 14.5SC @ 50g *a.i.* ha^{-1}, which was evaluated as 1.5 ppm for samples collected at 0 days (2 hours) after application. Dissipation of indoxacarb 14.5SC @ 50g *a.i.* ha^{-1} gradually declined in okra fruits to the level 0.76,0.28, 0.09 and 0.05 ppm with the reduction of 49.33, 81.33, 94.00 and 96.66 per cent after the spray of 1, 3, 5 and 7 days, respectively. There were no detection of residues in okra fruits on 10th, 14th and 15th day sample. The half-life value for indoxacarb was 1.32 to 1.58 days @ 50g *a.i.* ha^{-1} below the permissible maximum residue limit (MRL) of 0.5ppm.

Gradualy declined of bifenthrin 10EC @ 25g *a.i.* ha^{-1} dissipation in okra fruits to the level of 0.32, 0.20, 0.09, 0.03 and 0.012 ppm. After the spray of 1,3,5,7 and 10 days the reduction in its dissipation occurs 39.62, 62.26, 83.01, 94.33 and 97.73 per cent after the spray of 1, 3, 5, 7 and 10 days, respectively. Samples taken on 14th and 15th days shows no residues in okra fruits were detected. Bifenthrin half-life value was 1.58 to 2.18 days for @ 25g *a.i.* ha^{-1} and maximum residue limit (MRL) of 0.4 ppm. Is below the permissible limit.

For sample collected at 0 days (2 hours) after application 0.52 ppm was evaluated as the average initial spraying deposit of deltamethrin 2.8EC @ 15g *a.i.* ha^{-1}. In okra fruits deltamethrinresidues gradual declined its dissipation to the level 0.28, 0.18, 0.12, 0.1, 0.06 and

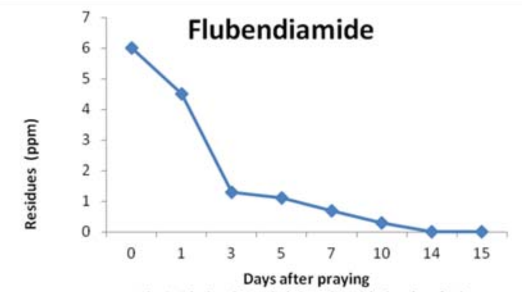

Fig.3: Dissipation of Flubendiamide in okra fruits

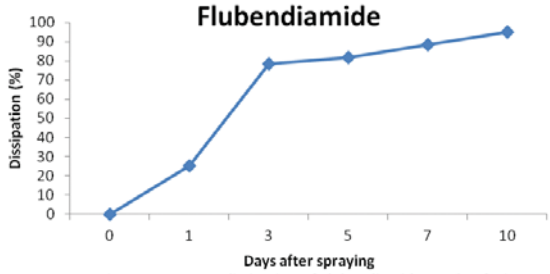

Fig. 4: Percentage dissipation of Flubendiamide on okra fruits

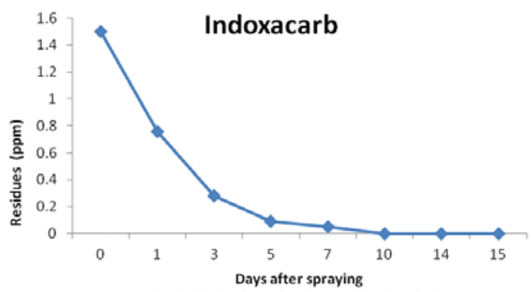

Fig. 5: Dissipation of Indoxacarb in okra fruits

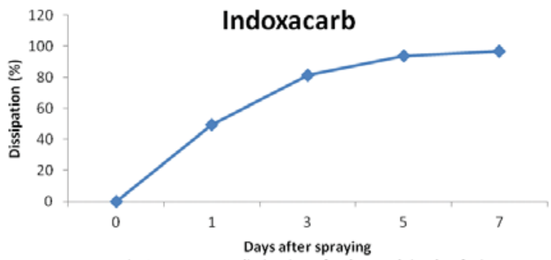

Fig.6: Percentage dissipation of Indoxacarb in okra fruits

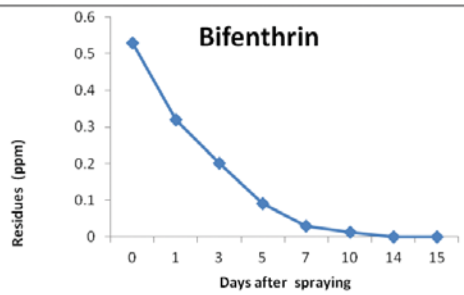

Fig.7: Dissipation of Bifenthrin in okra fruits

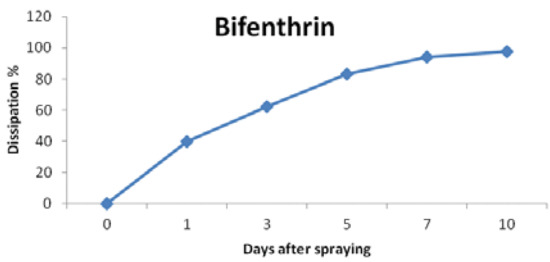

Fig.8: Percentage dissipation of Bifenthrin in okra fruits

0.02 ppm with the reduction of 46.15, 65.38, 76.92, 80.76, 88.46 and 96.15 per cent after the spray of 1, 3, 5, 7, 10 and 14 days, respectively. On 15th day sample no residues were detected in okra fruits. The half-life value for deltamethrin was 5.9 to 17 days for 15g *a.i.* ha^{-1} below the permissible maximum residue limit (MRL) of 0.2ppm.

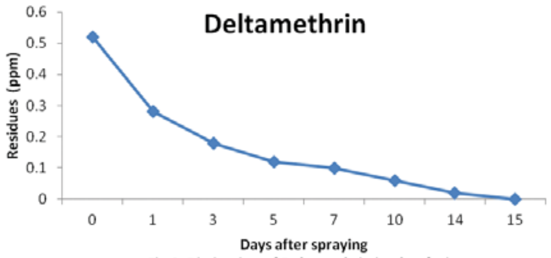

Fig.9: Dissipation of Deltamethrin in okra fruits

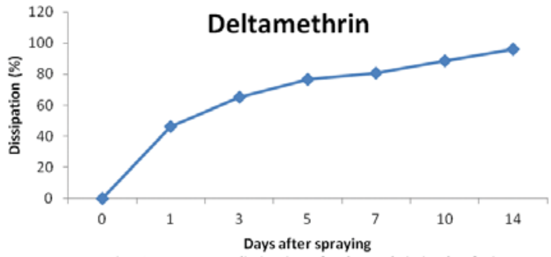

Fig.10: Percentage dissipation of Deltamethrin in okra fruits

After application of lambda-cyhalothrin 5EC @ 15g *a.i.* ha^{-1}, for samples collected, were the average initial spraying deposit was analysed as 2.45ppm at at 0 days (2 hours). In okra fruits the residues of lambda-cyhalothrin 5EC @ 15g *a.i.* ha^{-1}, gradually declined its dissipation to the level 1.26, 0.66, 0.5, 0.38 and 0.25 ppm with the reduction of 49.75, 73.06, 79.59, 84.48 and 89.79 per cent after the spray of 1, 3, 5, 7 and 10 days, respectively. In okra fruits there were no residues detected on 14th and 15th day sample. Lambda-cyhalothrin half-life value was 5.0 days @ 15g *a.i.* ha^{-1} which was below the permissible maximum residue limit (MRL) of 1.0ppm.

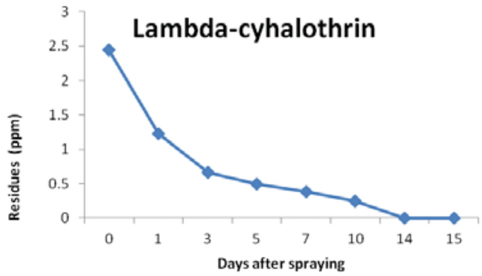

Fig.11: Dissipation of Lambda-cyhalothrin in okra fruits

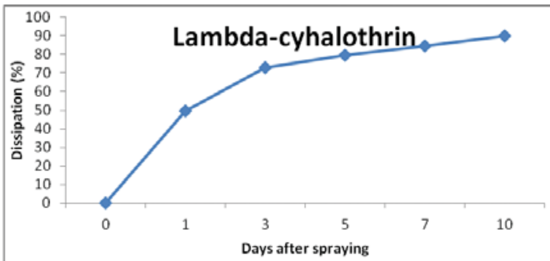

Fig.12: Percentage dissipation of Lambda-cyhalothrin in okra fruits

As evident from the table 1 and fig. (1-12), dissipation of insecticides residues followed a monophasic first order kinetics showing degradation of insecticides like, flubendiamide 48SC @ 55g *a.i.* ha^{-1} gradually declined its dissipation in okra fruits to the level of 4.5, 1.3, 1.1, 0.7 and 0.3 ppm with the reduction of 25.00, 78.33, 81.66, 88.33 and 95.00 per cent after the spray of 1,3,5,7 and 10 days, respectively and half-life value for Flubendiamide was 5.5 days @ 55g *a.i.* ha^{-1} below the permissible maximum residue limit (MRL) of 4ppm. Similar behavior were observed by Das et.al. (2012) with half-life period of 4.7 to 5.1 days at standard and double dose, respectively. The half-life value for Indoxacarb was 1.32 to 1.58 days @ 50 g *a.i.* ha^{-1} below the permissible maximum residue limit (MRL) of 0.5ppm. Similar findings were observed by Sharma and Mahopatra (2005) with half-life period of 1.1 to 1.5 days. Gupta *et al.* (2009) reported half-life of 0.58–1.02 days days for Indoxacarb. The half-life value for Bifenthrin was 1.58 to 2.18 days for @ 25g *a.i.* ha^{-1}. Below the permissible maximum residue limit (MRL) of 0.4ppm. Similar behavior were observed by Gupta *et al*. (2009) with half-life of 1.32– 1.58 days for bifenthrin. Thus our results are in conformity with the earlier reports.

It is clearly indicated that emamectin benzoate 5SG @ 12g *a.i.* ha^{-1} insecticide was quite effective for reducing the incidence of okra shoot and fruit borer *Earias vittella* (Fab.) and dissipated in the okra crop upto ten (10) days.

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