RESIDUAL STATUS OF INSECTICIDES IN/ON OKRA FRUITS

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ABSTRACT

The study on the residue status of some insecticides in okra fruits was determined at Department of All India Network Project on Pesticide Residue, Anand Agricultural University, Anand during summer 2010. The initial deposit of ∑ endosulfan residue was 1.217 µgg^{-1} at 0 days (2 hours after application) which was reduced to 0.872 (28.35%) and 0.131 (89.24%) at 1st and 3rd days, respectively and reached to below determination limit (BDL) after 5th day of spray. The initial deposit of cypermethrin residue in a combi product of cypermethrin + chlorpyriphos was 0.268 µgg^{-1} at 0 days which was reduced to 0.183 µgg^{-1} (31.72%) at 1st day and reached to BDL after 3rd days of spray. However, the initial deposit of chlorpyriphos residue was 0.707 µgg^{-1} at 0 days which was reduced to 0.299 µgg^{-1} (62.66%) at 1st day and reached to BDL after 3rd days of spray. The regression equation for endosulfan-I, endosulfan-II and ∑ endosulfan was $y = -0.428x + 2.339$ ($R^2 = 0.860$), $y = -0.308x + 2.791$ ($R^2 = 0.975$) and $y = -0.248x + 3.053$ ($R^2 = 0.952$), respectively. The regression equations for cypermethrin and chlorpyriphos were $y = -0.435x + 2.627$ ($R^2 = 0.925$) and $y = -0.820x + 3.028$($R^2 = 0.965$). The half life of ∑ endosulfan, cypermethrin and chlorpyriphos was 1.214, 0.692 and 0.367 days, respectively. The safe waiting period of 3, 3 and 2 days should be kept after spraying of endosulfan, cypermethrin and chlorpyriphos, respectively.

Key words: Okra, residue, cypermethrin, chlorpyriphos, endosulfan, waiting period

INTRODUCTION

Okra [*Abelmoschus esculentus* (L.) Moench] is an important vegetable crop belonging to family Malvaceae grown in the tropical and sub tropical regions of the world. There are many factors affecting the low productivity of okra. One of them is the losses caused by insect pests. The crop is affected by number of insect pest, mites and nematodes during different growth stages. (Khan and Mukhopadhyay, 2004). Of these, shoot and fruit borer, E*arias vittella* Fabricius is the most destructive pests causing economic damage to the crop at all the growth stages. Yield losses up to 52.33 to 70.75% at Udaipur, Rajasthan (Kanwar and Ameta, 2007) and fruit damage to extent of 91.6% at Anand, Gujarat (Shah *et al*., 2001) was reported due to the infestation of this pest.

The chemical insecticides are used as the front line defense sources against insect pest. Farmers use large quantities of chemical insecticides singly or in combination in order to combat this pest problem and higher yield. Indiscriminate use of pesticides particularly at fruiting stage and non adoption of safe waiting period leads to accumulation of pesticide residues in consumable vegetables. The market samples of various vegetables showed 40-60% contamination across the country (Handa, 1992), among them up to 58 % was in okra (Awasthi, 1998). Thus, there is need of guiding the consumers for observing proper waiting period based on residual status of pesticides. Keeping the above points in view, the present investigations were carried out to study the residual status of some insecticides in/on okra fruits.

MATERIALS AND METHODS

Okra crop was sown during 1st week of February and raised successfully after following standard agronomical practices. Two sprays of respective insecticide [Endosulfan 35 EC, 0.07% (2.0 ml/l water) and Cypermethrin + Chlorpyriphos 55 EC, 0.055% (1.0 ml/l water)] were made at 10 days interval from the fruiting stage.

Sampling:

The representative sample of okra fruits of marketable quality was collected at 0 (2 hours after application), 1, 3, 5 and 7 days after second spray. For this purpose, 100 g okra fruits were picked up randomly from all the three replications under insecticides treated plots as well as untreated control. The samples of okra fruits were collected in a clean polythene bag, tagged with sampling details and immediately brought to the laboratory for residue analysis. The analysis was carried out at the Pesticide Residue Laboratory, All India Network Project on Pesticide Residues, Anand Agricultural University, Anand.

Extraction:

Okra fruits will be subjected to multiresidue analysis by adopting CDFA (California Department of Food and Agriculture)/QuEChERS (Quick, Easy, Cheap, Effective, Rugged and Safe) method for organophosphate, organochlorin and synthetic pyrethroid groups of insecticides. A representative sample of fruits will be cut in to small pieces, homogenized and 50 g sub-sample will be subjected to extraction with acetonitrile (100 ml). The sample will be homogenized at high speed for 3 minutes and the extract will be filtered in a 100 ml capacity glass stoppered cylinder. To this, 15 g NaCL will be added followed by vigorous shaking. The layers will be allowed to separate and two 10 ml aliquots from the upper acetonitrile layer will be collected separately in 15 ml capacity graduated test tubes. Acetonitrile from both the test tubes will be evaporated to near dryness and residues will be reconstituted in 2 ml acetone-hexane (1:1, v/v). The extract in one of the test tube will be directly subjected to estimation of organophosphate group of insecticides by Flame Photometric Detector (FPD) on Gas Liquid Chromatography (GLC) while another extract was subjected to cleanup for estimation of organochlorin and synthetic pyrethroid group of insecticides by Electron Capture Detector (ECD) on GLC.

Clean up:

A glass column (1 cm X 30 cm) will be filled up with 0.5 g of Na_2SO_4 **(**anhydrous), 1 g Florisil and 0.5 g Na_2SO_4 (anhydrous), bottom upward. The column will be washed with 5 ml acetone-hexane (1:9, v/v) followed by 5 ml hexane. When the solvent front reaches the top of the column, the sample extract (2 ml) will be transferred and the test tube will be rinsed twice with 5 ml hexane and the rinsate will be transferred to the column. The pesticides from the column will be eluted by 15 ml acetone-hexane (1:9, v/v) and the elute will be subjected to nitrogen concentrator to make up the final volume to 2 ml.

Estimation:

The different pesticides will be estimated by Gas liquid chromatography with suitable detector.

Calculations involved in residue analysis (Hoskins, 1961)

1) Concentration of Pesticide Residue (ppm) present in/on okra fruits:

$$\text{ppm} = \frac{\text{Conc. of standard X Vol. of standard}\,(\mu\text{l})\,\text{injected X Area/height of Sample peak X Final volume}}{\text{Weight of sample X Vol. of sample}\,(\mu\text{l})\,\text{injected X Area/height of Sample peak}}$$

2) Half life ($T_{0.5}$**)** value based on regression equation

$$T_{0.5}\text{ (days)} = \frac{\text{Log } 2}{b}\, i.e. \frac{0.3010}{b}$$

Where b = Slope of the regression line

3) Waiting period based on regression equation :

y = log (MRL x 100) : Endosulfan

y = log (MRL x 1000) : Cypermethrin + Chlorpyriphos

y = a + bx

Where, x = waiting period (days)

RESULTS AND DISCUSSION

The results obtained from residue levels of insecticides in okra fruits for dissipation study are presented in Table 1 while, regression equation, half life and waiting periods are presented in Table 2. The similar are also exhibited by graphical presentation in Figure 1 to 10 and discussed hereunder.

∑ endosulfan

Data on ∑ endosulfan residue in okra fruits (Table 1) revealed that the initial deposit was 1.217 µg g^{-1} at 0 days (2 hours after application) which reduced to 0.872 and 0.131 at 1st and

3rd days, respectively. The residue was below determination limit (BDL) after 5th day of spray. The reduction in residue within one day after treatment was low (28.35%). The corresponding reduction in residues was 89.24% after 3rd day of spray. Dissipation rate was slow in okra fruits within one day as compared to chlorpyriphos then after, the rate was fast after 3 days.

The results presented in Table 2 indicated that the regression equation for endosulfan-I was $y = -0.428x + 2.339$ ($R^2 = 0.860$) and half life of 0.703 day. The regression equation for endosulfan-II and ∑ endosulfan was $y = -0.308x + 2.791$ ($R^2 = 0.975$) and $y = -0.248x + 3.053$ ($R^2 = 0.952$), respectively. The half life of endosulfan-II and ∑ endosulfan was 0.977 and 1.214 days, respectively. The calculated waiting period for ∑ endosulfan was 3 days.

A higher level of initial deposit of endosulfan residue (1.217 µg g^{-1}) in okra fruits observed in present study corroborates with earlier reports of Shah (1997) who recorded the initial deposit of 7.555 µg g^{-1} of endosulfan in okra fruits. Rajabaskar *et al*. (2001) recorded the initial deposit of endosulfan of 3.18 and 4.39 ppm for lower (700 g a.i./ha) and higher (1400 g a.i./ha) doses, respectively on okra fruits. According to Deivendran *et al*. (2006), residues of endosulfan applied @ 350 g a.i./ha was 3.452 mg/kg after one hour of application. Deen *et al.* (2009) determined the residues of endosulfan applied @ 300 g a.i./ha and found that the initial deposits were 9.25 µg g^{-1}. Shah (1997) reported that endosulfan dissipated at relatively faster rate to 2.353 µg g^{-1} within 24 hrs, which reached to 0.865 µg g^{-1} on the fifth day with half life of 1.81 days in okra. Deen *et al.* (2009) determined the residues of endosulfan applied @ 300 g a.i./ha in okra crop and reported that calculated half-life and safe waiting periods were 3.8 and 8.3 days, respectively. These reports are also more or less corroborating with present findings.

Cypermethrin

The initial deposit of cypermethrin residue in a combi product of cypermethrin + chlorpyriphos (Table 1) in okra fruits was 0.268 µg g^{-1} at 0 days (2 hours after application) which was reduced to 0.183 µg g^{-1} at 1st day. The reduction in residue within one day after treatment was low (31.72%) as compared to chlorpyriphos. The residue was below determination limit (BDL) after 3rd days of spray. The regression equation (Table 2) for cypermethrin was $y = -0.435x + 2.627$ ($R^2 = 0.925$). The half life and waiting period of cypermethrin was 0.692 and 3 day, respectively.

Singh and Udeaan (1989) estimated the dissipation and persistence of cypermethrin in okra fruits and revealed that after second and third sprays, initial residues were 0.76 and 0.65 mg/kg for the 50 g a.i./ha dose and 1.53 and 1.43 mg kg^{-1} for the 100 a.i./ha dose, respectively. According to Singh *et al*. (2004), the initial deposits of cypermethrin applied @ 50 and 100 g a.i./ha were 0.274 and 0.382 mg/kg on okra fruits. Deen *et al.* (2009) determined the residues of cypermethrin, applied @ 60 g a.i./ha in okra fruits and showed that the initial deposits were 0.53 µg/g for cypermethrin. Thus, the reports of Shah *et al*. (1999) and Singh *et al*. (2004) were tally with present findings. In present investigation, cypermethrin residue in okra fruits was reduced to 0.183 µg g^{-1} (31.72%) at 1st day and the residue was below determination limit (BDL) after 3rd days of spray which is confirmed with the earlier reports of Shah *et al*. (1999) who reported that the dissipation of cypermethrin 4% in a combi product Polytrin 44 EC in/on okra fruits after 1, 3 and 5 days of spray were 0.13, 0.09 and 0.07(µg/g). Singh *et al*. (2004) also reported that the residue of cypermethrin applied @ 50 and 100 g a.i./ha dissipated to 0.013 and 0.020 mg/kg in 15 days after third application on okra fruits. Singh *et al*. (2004) reported that the half- life values of cypermethrin applied @ 50 and 100 g a.i./ha were 3.4 and 3.5 days and waiting periods of 2 and 3 days, respectively.

Chlorpyriphos

The data on chlorpyriphos residue in a combi product of cypermethrin + chlorpyriphos in okra fruits (Table 1) revealed that the initial deposit was 0.707 µg g^{-1} at 0 days (2 hours after application) which was reduced to 0.299 µg g^{-1} at 1st day. The reduction in residue within one day after treatment was maximum (62.66%) amongst the insecticides tested. The residue was below determination limit (BDL) after 3rd days of spray. The regression equation (Table 2) for chlorpyriphos was $y = -0.820x + 3.028$ ($R^2 = 0.965$) and half life of 0.367 day. The waiting period for chlorpyriphos was 2 days.

Sardana *et al.* (2005) monitored the chlorpyriphos residues in IPM and non IPM fields of okra and found that the residues was 0.10 µg g^{-1} and 5.75 µg g^{-1} for IPM and non-IPM fields, respectively. Residues of chlorpyriphos solely and in combination with cypermethrin were dissipated to 1.54 and 0.68 µg g^{-1}, respectively at 1 day after spray. Raina and Raina (2008) found that when chlorpyriphos applied @ 500 and 1000 g a.i./ha on cauliflower, an initial deposit was 0.56 and 1.29 ppm, with half-life of 1.4 and 1.6 days for 500 and 1000 g a.i./ha, respectively. Gupta *et al.* (2010) studied the persistence of pre-mix formulations of Action 55 EC (chlorpyriphos 50%+ cypermethrin5%) at recommended (0.8 to 1.0 lit./ha) and double (1.6 to 2.0 lit./ha) dosage and found that residues were persisted beyond 7 days in tomato fruits. Residues of chlorpyriphos dissipated from fruits with half life values of 2.9 to 3.3 days. Thus, above reports are also tally with present findings.

CONCLUSION

The deposit of ∑ endosulfan residue was reduced to 0.131 (89.24%) at 3rd days. The deposit of cypermethrin residue in a combi product of cypermethrin + chlorpyriphos reached to BDL after 3rd days of spray. However, the deposit of chlorpyriphos residue reached to BDL after 3rd days of spray. The half life of ∑ endosulfan, cypermethrin and chlorpyriphos was 1.214, 0.692 and 0.367 days, respectively. The safe waiting period of 3, 3 and 2 days should be kept after spraying of endosulfan, cypermethrin and chlorpyriphos, respectively.

REFERENCES

Awasthi, M. D. (1998). *Pesticide residues in food chain in India- Fruits and vegetables*. In: Pesticides, Man and biosphere, Ed. O. P. Shukla, Omkar and A. K. Kulshrestha, APH Publishing Corporation, New Delhi, pp-121-160.

Deen, M. K., Kumari, B. and Sharma, S. S. (2009). Dissipation and decontamination of residues of three pesticides in okra fruits. *Pesticide Res. J.,* **21**(1): 80-82.

Deivendran, A., Beena Kumari and Yadav, G. S. (2006). Dissipation of endosulfan and dichlorvos residues in/on cauliflower curds. *Environ. Monit. and Assess.,* **116**(1-3): 307-313.

Gupta, S., Gajbhiye, V. T., Sharma, R. K. and Gupta, R. K. (2010). Dissipation of cypermethrin, chlorpyriphos and profenophos in tomato fruits and soil following application of pre-mix formulations. *Environ. Monit. and Assess.,* pp- 25-28.

Handa, P. (1992). *Monitoring of pesticide residues in Indian environment*. In: "Pest Management and Pesticides Indian Scenario". Ed. B. V. David, Namratha Publication, Madras (India), pp- 221-224.

Hoskins, W. M. (1961). Mathematical treatment of the rate of loss of pesticides residues. *FAO Pl. Prot. Bull.*, **9** : 163-168. [Fide : Regupathy, A. and Dhamu, K. P. (2001). *Statistics work book for insecticide toxicology*. Softeck computers, Coimbatore, 77-161].

Kanwar, N. and Ameta, O. P. (2007). Assessment of losses caused by insect pests of okra. *Pestology,* **31**(6): 45-47.

Khan, M. R. and Mukhopadhyay, A. K., 2004, Screening of crop germplasms for resistance against root knot nematode, *Meloidogyne incognita* race 2, *Environment Ecol.*, **22**(spl-3): 445-448.

Raina, A. K. and Raina, M. (2008). Dissipation of chlorpyriphos on cauliflower. *Pesticide Res. J.,* **20**(2): 263-265.

Sardana, H. R., Bambawale, O. M., Singh, D. K. and Kada, L. N. (2005). Monitoring of insecticide residues in IPM and non-IPM fields of okra and brinjal. *Indian J. Pl. Prot.,* **33**(2): 197-201.

Shah, B. H., Shah, P. G., Jhala, R. C. and Vyas, H. N. (1999). Studies on dissipation of some ready-mix insecticides combinations in/on okra fruits. *Pestology,* **23**(6): 3-9.

Shah, B. R. (1997). Bioecology, bioefficacy and residual status of some ready mix insecticides in relation to pest complex of okra [*A. esculentus* (Linnaeus) Moench]. Ph.D Thesis submitted to GAU, Sardar Krushi Nagar.

Shah, B. R., Vyas, H. N. and Jhala, R. C. (2001). Life table of shoot and fruit borer. *Earias vittella* (Fab.) for determining key mortality factors in okra, *Abelmoschus esculentus* (L.) Moench. Paper presented in National Conference on "*Plant Protection - New Horizons in the Millennium*" held at RAU, Udaipur during Feb 23-25, 2001, pp- 4.

Singh, B. and Udeaan, A. S. (1989). Estimation of cypermethrin residues in the fruits of okra, *Abelmoschus esculentus* (Linn.) Moench. *J. Insect Sci.*, **2**(1): 49-52.

Singh, S. P., Kumar, K. S. and Tanwar, R. S. (2004). Dissipation and decontamination of cypermethrin and fluvalinate residues in okra. *Pesticide Res. J.*, **16**(2): 65-67.

Table 1 : Residues of insecticides in okra fruits

Insecticides	Conc. (%)	Residues (µg g^{-1}) Days after treatment				
		0	1	3	5	7
∑ Endosulfan	0.07	1.217±0.749	0.872±0.130 (28.35)	0.131±0.068 (89.24)	BDL	BDL
Cypermethrin	0.005	0.268±0.016	0.183±0.040 (31.72)	BDL	BDL	BDL
Chlorpyriphos	0.05	0.707±0.264	0.299±0.075 (62.66)	BDL	BDL	BDL
Control	-	ND	ND	ND	ND	ND

BDL (∑ Endosulfan = 0.07 µg g^{-1}, Cypermethrin = 0.07 µg g^{-1}, Chlorpyrifos = 0.03 µg g^{-1})

Table 2: Regression equation and half- life of insecticides

Insecticides	Retention time (min)	Half-life (Days)	Regression equations	R^2 Value	Waiting period (Days)
Endosulfan-I	6.55	0.703	y = -0.428x + 2.339	0.860	-
Endosulfan-II	7.32	0.977	y= -0.308x + 2.791	0.975	-
∑ Endosulfan	-	1.214	y = -0.248x + 3.053	0.952	3.03 (3)
Cypermethrin	11.26	0.692	y = -0.435x + 2.627	0.925	3.05 (3)
Chlorpyriphos	5.44	0.367	y = -0.820x + 3.028	0.965	2.11 (2)

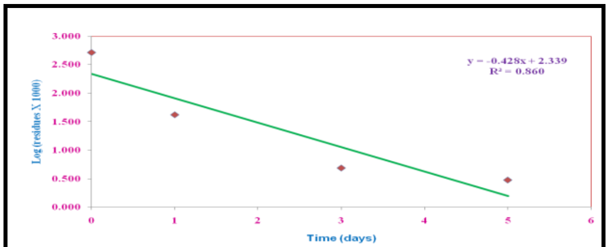

Fig. 1: Dissipation of endosulfan I in/on okra fruits

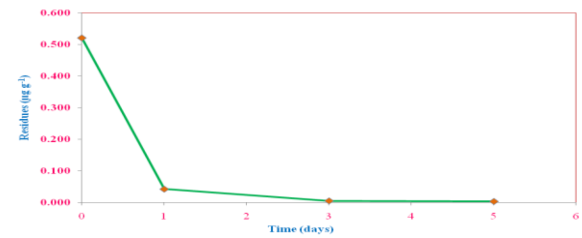

Fig. 2: Chemodynamics of endosulfan I in/on okra fruits

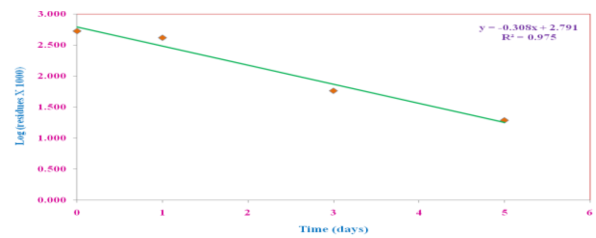

Fig. 3: Dissipation of endosulfan II in/on okra fruits okra fruits

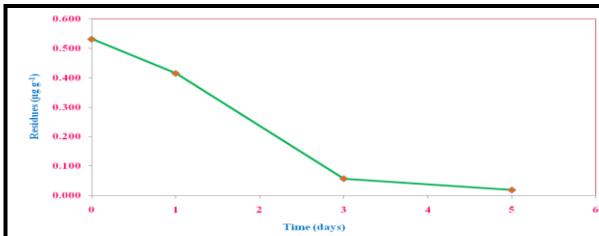

Fig. 4: Chemodynamics of endosulfan II in/on okra fruits

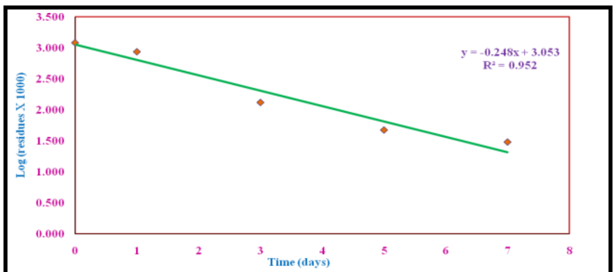

Fig. 5: Dissipation of total endosulfan in/on okra fruits

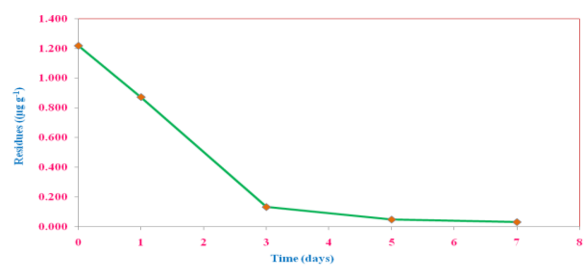

Fig. 6: Chemodynamics of total endosulfan in/on okra fruits

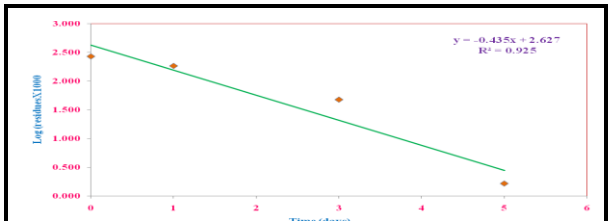

Fig. 7: Dissipation of cypermethrin in /on okra fruits

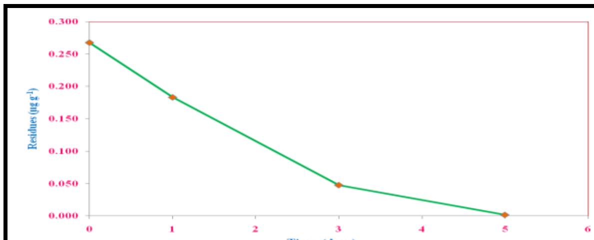

Fig. 8: Chemodynamics of total endosulfan in/on okra fruits

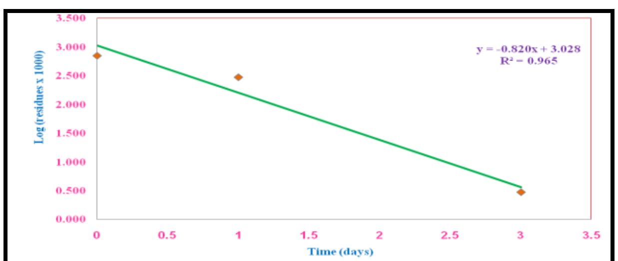

Fig. 9: Dissipation of chlorpyriphos in/on okra fruits

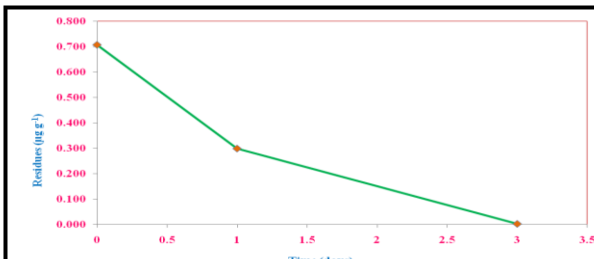

Fig. 10: Chemodynamics of chlorpyrifos in/on okra fruits

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