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Dissipation of chlorpyriphos on Okra (*Abelmoschus esculentus* L.)

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Abstract

Indiscriminate use of insecticides to combat the insect pests has led to accumulation of residues in okra which are harmful to consumers. In the present study, an attempt has been made to study the dissipation of chlorpyriphos on Okra fruits under agro-climatic conditions of Jammu where no study has been carried out earlier. Field experiments have been conducted for two consecutive years 2004 and 2005 to work out the safe preharvest interval. The average initial deposits of 0.91 mg kg^{-1} and 1.46 mg kg^{-1} have been recorded on okra fruits treated with the recommended (500g a.i. ha^{-1}) and double the recommended dose of chlorpyriphos (1000g a.i. ha^{-1}) respectively, showing percentage dissipation of 97.80 and 98.63% correspondingly. On the basis of dissipation and prescribed MRL of 0.20 mg kg^{-1} of chlorpyriphos for okra, the half-life for the recommended and double the recommended dose has been worked out to be 1.33 and 1.41 days, respectively. The safe waiting period of 2.92 and 4.06 days have been suggested for the recommended and double the recommended dose of chlorpyriphos, respectively.

Keywords*: Chlorpyriphos, Dissipation, Okra, Maximum Residue Limit, Pesticide, Safe waiting period.*

Introduction

Use of pesticides to control pests is unavoidable as pests cause heavy loss to yield and quality of the food items including the vegetables, which forms a very important component of agriculture in India. Various insect pests viz. shoot and fruit borer, leaf rollers, jassids, aphids, moths, mites, fruit flies, caterpillars, weevils and hoppers etc. cause considerable losses to the vegetables which along with fruits and spices have been estimated to be of Rs. 30,000 crores in India only (MOCF, 2002). Several kinds of pesticides are being applied to control the pests, sometime close to the harvest or picking time of vegetables, thereby, leaving little or no time for their adequate dissipation. The presence of pesticide residue or their metabolites is a matter of great concern as it is directly related to the health of the human beings. Use of pesticides cannot be avoided but their quality, quantity and safe waiting period after the spray can be regulated so that minimum level of residues are left on the vegetables at the time of consumption. Supervised trials provide useful information for determining the waiting period which are considered safe interval between the last application of pesticide and harvest of edible part of the vegetables. This also facilitates the growers to adjust their crop harvest intervals accordingly in the interest of vegetable consumers.Okra, commonly called as "Bhendi", is an important vegetable crop grown all round the year and throughout the country for fresh market consumption as well as for preservation, as the fruit is very rich in fiber, proteins, vitamins and minerals (Pawar and Jadhav, 1993). Several studies have indicated the contamination of market samples of okra with different kinds of pesticides including chlorpyriphos.(Dahiya and Chauhan ,1982; Chauhan *et al.*,1983; Saxena *et al.*, 1990; Chahal *et al.*, 1997; Agnihotri, 1999; Chahal *et al.*, 1999; Rao and Rao ,2000; Kole *et al.*, 2002 and Shah *et al*., 2000).Chlorpyriphos (0, 0-diethyl 0-3, 5, 6-trichloro-2-pyridyl phosphorothioate) is a broad-spectrum organophosphorous insecticide used against a number of important arthropod pests in

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the study area. Till date no studies have been conducted on dissipation pattern of chlorpyriphos on okra under agro climatic conditions of J&K. The present studies were, therefore, undertaken to determine the residues of chlorpyriphos on *Abelmoschus esculentus* L.(Variety: Pusa Sawani) following its application at the recommended and double the recommended dosages to find out the desirable waiting period between the last spraying and harvesting of vegetable.

Material and Methods

The field experiments were conducted at village Jessore (32^0 38' N latitude and 74^0 45' E longitude; altitude: 271meters above mean sea level, located in Low- altitude subtropical agro climatic zone characterized by the monsoon concentration of precipitation, hot summers and relatively dry but pronounced winters and preponderance of alluvial soils) located in Tehsil R.S. Pura of District Jammu for the two consecutive years. The okra crop was raised by sowing the certified seeds procured from J & K Agriculture Department, directly into the prepared field in the form of ridges placed at a distance of 60cm with plant to plant spacing of 30cm (recommended by Directorate of Extension Education, SKUAST-Jammu) in starting April 2004 and 2005 and thereafter normal agronomic practices were followed.Chlorpyriphos 20EC, purchased from local market was sprayed twice at the rate of 500g a.i. ha^{-1} (recommended dose) and 1000g a.i. ha^{-1} (double the recommended dose) with Knapsack sprayer. First spray was done at the onset of flowering followed by second spray at an interval of twenty days. The sampling was done on 0(2 hr), 1, 2, 3, 4, 5, 7, 9 and 15 after the second spray for both the years. Samples from each experiment plot consisted of about 8-10 okra fruits and were brought to the laboratory in polythene bags with minimal finger touching for residue analysis.

Extraction and Partitioning: Representative sample of chopped okra fruits (50g) after proper mixing and quartering was blended in an electric grinder and rinsed with Acetone AR. The macerated sample was immersed in 100-150 ml Acetone AR and kept over night. The sample was filtered into 1 liter separating funnel using whatman's filter paper No. 1. After adding 600ml of 5% NaCl solution to the above extract, partitioning was done twice with 100 ml of dichloromethane AR. The lower layer was collected over 30g of anhydrous sodium sulphate and concentrated to about 5ml using rotary vacuum evaporator.

Clean Up: The cleanup of the extract was performed by adsorption column chromatography. Glass column of 2.5 cm (i.d.) x 60 cm (length) was packed with mixture of 20g silica gel (60-120 mesh size) activated at 130°C for 2 hrs and activated animal charcoal (300mg) sandwiched between 5g of anhydrous sodium sulphate layers. The drip tip of the column was plugged with cotton. After pre washing the packed column with 40ml of dichloromethane AR the sample extract was added to it and eluted with 150 ml solvent mixture of Acetone: dichloromethane (2:1). The eluate was evaporated to dryness and the residue was dissolved in 5ml of Acetonitrile (HPLC grade) and the sample was filtered using membrane filter media.

Estimation: The residues of chlorpyriphos was estimated by using High Pressure Liquid Chromatography (Shimadzu HPLC model LC-10A) equipped with dual pump (LC-10 AT) , auto injector (SIL-10 A) , UV detector(SPD-10 A) set at a wavelength of 225 nm. The column used for the separation was Shim pack CLC-ODS (M) 4.6 X 25 cm. long stainless steel tube packed with totally porous, spheric silica particles (5 µ m particle diameter, 100^0A pore diameter).The solvent system used was acetonitrile: water (90:10) at a flow rate of 1ml/min and the retention time of chlorpyriphos was 6.1 minutes. The method offered a sensitivity of 0.5 ng and limit of detection of 0.01mg kg^{-1} (on 50g sample basis).The average recoveries from okra fortified with concentrations ranging from 0.20 to 1.0 mg kg^{-1} were found to be in the range of 83%-93% with an average of 88.42$\pm$2-31%. All the solvents used for extraction and cleanup were glass distilled and chemicals i.e. silica gel, anhydrous sodium sulphate etc. along with glassware were washed with distilled solvents before use. The suitability of all the solvents for residual analysis was ensured by running reagent blanks.

Statistical Analysis: The data was analyzed to work out half life value (RL_{50}) and safe waiting period (T_{tol}) according to Hoskins formula (1961).

Results and Discussion

The quantitative estimates of residues of chlorpyriphos on okra fruits for two consecutive years i.e. 2004 and 2005 at various sampling intervals (0, 1, 2, 3, 4, 5, 7, 9 and 15 days after the second spray) have been presented in the Table 1. The residues of chlorpyriphos on okra fruits collected from control plots has been always found to be below the detectable limits (0.01mg kg^{-1}) during both the years of study period.The average initial deposits of chlorpyriphos on okra fruits after the second spray were found to be 0.86 and 1.80 mg kg^{-1} at the recommended 500g a.i. ha^{-1}) and double the recommended dose (1000g a.i. ha^{-1}), respectively, for the first year (i.e. 2004). About 98% of initial deposits dissipated within 7 days after its application at recommended dose while at double the dose about 99% of the initial deposits get dissipated within 9 days. The time required (T_{tol}) for dissipation of residues below the maximum residue limit (MRL) of 0.2 mg kg^{-1} were found to be 2.82 and 4.45 days, respectively, for lower and higher dosages while the corresponding values for half life (RL_{50}) were found to be 1.3413 and 1.4039 days (Table 1).

Table 1: Dissipation of Chlorpyriphos on Okra fruits during 2004 and 2005

Days after treatment	*Residues of chlorpyriphos (mg kg^{-1})			
	1st Year (2004)		2nd Year (2005)	
	R.D. (500g a.i. ha^{-1})	Double R.D. (1000g a.i ha^{-1})	R.D. (500g a.i. ha^{-})	Double R.D. (1000g a.i ha^{-1})
0 (2hr)	**0.86**	**1.80**	**0.96**	**1.12**
1	**0.51** (40.69)	**0.95** (47.22)	**0.55** (42.71)	**0.80** (28.57)
2	**0.43** (50.0)	**0.59** (67.22)	**0.46** (52.08)	**0.66** (41.07)
3	**0.21** (75.58)	**0.31** (82.78)	**0.22** (77.08)	**0.33** (70.53)
4	**0.12** (86.05)	**0.23** (87.22)	**0.11** (88.54)	**0.24** (78.57)
5	**0.04** (95.35)	**0.07** (96.11)	**0.04** (95.83)	**0.06** (94.64)
7	**0.02** (97.67)	**0.04** (97.78)	**0.02** (96.87)	**0.03** (97.32)
9	BDL	**0.02** (98.89)	BDL	**0.02** (98.21)
15	BDL	BDL	BDL	BDL
RL_{50}(days)	**1.3413**	**1.4039**	**1.3254**	**1.4388**
T_{tol} (days)	**2.820**	**4.450**	**2.999**	**3.576**
Regression Equation	Y=2.8782-0.2244x r = -0.9443	Y=3.1632-0.2144x r = -0.9946	Y=2.9023-0.2271x r = -0.9444	Y= 3.0948-.2092x r = -0.9905

Control Samples showed residue level =BDL; Figures in parenthesis represent %age dissipation. *Average of three replicates; BDL = Below detectable limit (0.01mgkg^{-1}); R.D. = Recommended dos

Similarly for the second year (i.e. 2005) average initial deposits of chlorpyriphos on okra fruits were found to be 0.96 and 1.12 mgkg^{-1} following treatments of recommended and double the recommended dosages. Dissipation of residues was found to be about 97% within 7 days and about 98% within 9 days respectively for lower and higher dosages. The safe waiting periods (T_{tol}) at recommended and double the recommended dosages were found to be 2.999 and 3.576 days, respectively, and for half life (RL_{50}) these values were 1.3254 and 1.4388 days correspondingly (Table 1).On an average (of the two years) initial deposits of 0.91 mg kg^{-1} on okra fruits, treated with the recommended dose (500g a.i. ha^{-1}) of chlorpyriphos, dissipated to 0.53, 0.44, 0.21, 0.11, 0.04 and 0.02 mg kg^{-1} on 1, 2, 3, 4, 5 and 7th day after the spray, respectively showing corresponding percentage dissipation of 41.75, 51.64, 76.92, 87.91, 95.60 and 97.80 percent, whereas average (of the two years) initial deposits of 1.46 mg kg^{-1} recorded on okra fruits treated with double the recommended dose of chlorpyriphos (1000g a.i. ha^{-1}), dissipated to 0.87, 0.62, 0.32, 0.23, 0.06, 0.03 and 0.02 on 1, 2, 3, 4, 5, 7 and 9th day after the spray, respectively showing corresponding percentage dissipation of 40.41, 57.53, 78.08, 84.24, 95.89, 97.94 and 98.63 percent (Table 2).An average, the rate of dissipation on okra fruits has been observed to be rapid during initial days and is almost similar at both the doses of chlorpyriphos which is in close consonance with the works of Hinduja *et al.,* 1979 and Samant *et al.,* 1997 on dissipation of chlorpyriphos.

Table 2: Dissipation of Chlorpyriphos on Okra fruits. (Average of 2004 and 2005).

Days after treatment	Average chlorpyriphos residues (mg kg^{-1})for two years ± S.D.	
	500g a.i. ha^{-1} R.D.	1000g a.i. ha^{-1} Double R.D.
0 (2hr)	0.91±0.07	1.46 ± 0.48
1	0.53±0.02 (41.75)	0.87±0.04 (40.41)
2	0.44±0.02 (51.64)	0.62±0.04 (57.53)
3	0.21±0.007 (76.92)	0.32±0.01 (78.08)
4	0.11±0.007 (87.91)	0.23±0.007 (84.24)
5	0.04±0 (95.60)	0.06±0.007 (95.89)
7	0.02±0 (97.80)	0.03±0.007 (97.94)
9	BDL	0.02±0 (98.63)
15	BDL	BDL
RL_{50}(days)	1.3348	1.4151
T_{tol} (days)	2.918	4.058
Regression Equation	Y = 2.885 - 0.2255x r = -0.9446	Y = 3.1227 – 0.2127x r = -0.9914

Control Samples showed residue level =BDL; Figures in parenthesis represent %age dissipation. *Average of three replicates; BDL= Below detectable limit (0.01mgkg^{-1}); R.D. = Recommended dose.

On the basis of present study, the safe waiting period of 2.92 and 4.06 days have been suggested for the recommended and double the recommendeddosages of chlorpyriphos on okra, respectively. Various workers have reported different safe waiting periods for different pesticides used on okra fruits. Rajabaskar *et al.,* 2001 suggested waiting periods of 2.09 and 4.5 days for okra fruits treated with recommended and double the recommended doses of endosulphan, while Iiango and Devraj, 2003 suggested waiting periods of 3.51 - 5.73 days for okra fruits treated with imidacloprid for different concentrations. Patel *et al.,* 2001 have suggested waiting periods of only

one day for okra fruits treated with lindane while Singh, 1999 recommended waiting periods ranging from 3.93 to 9.22 days for okra fruits sprayed with different doses of monocrotophos. Biswas *et al*., 1991 recommended waiting periods ranging from 4.7 to 6.3 days to be observed between spraying of monocrotophos and harvesting of okra fruits. Thus, the safe waiting periods of 2.92 days (with recommended dose) and 4.06 days (with double the recommended dose) which have been suggested in the present study, are in fair agreement with the findings of some of the workers who have worked on the okra with some other pesticides.

Conclusion

On the basis of dissipation and prescribed Maximum Residue Limit of 0.20 mg kg^{-1} of chlorpyriphos for okra, the safe waiting period of 2.92 and 4.06 days have been suggested for the recommended and double the recommended dosages of chlorpyriphos, respectively.

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