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Persistence of Chlorpyriphos and Deltamethrin Residues in Cabbage

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Abstract

The experiment was conducted during *kharif* -2007 with cabbage variety Varun. Three sprays of chlorpyriphos @ 0.05% and deltamethrin @ 0.01% was sprayed at head formation stage. The dissipation pattern of chlorpyriphos revealed that initial deposit of chlorpyriphos (2.75 mg kg^{-1}) was dissipated below detectable residues at 15 days after third spray. While, in case of deltamethrin, that the initial deposit of cypermethrin (1.21 mg kg^{-1}) was dissipated below detectable level at 7 days after third spray. The maximum residue limit for chlorpyriphos and deltamethrin was found to be 0.5 and 0.2 mg kg^{-1}, respectively. The waiting period for safe harvest of cabbage heads after three sprays of chlorpyriphos (0.05%) and deltamethrin (0.01%) at head formation stage was 6.53 and 1.39 days, respectively. The half life values for chlorpyriphos and deltamethrin was 1.76 and 1.34 days, respectively.

1. Introduction

Cabbage is the fourth most widely grown vegetable crop of our country. In India, cabbage production is about 5.7 mt from 0.27 mha area with average yield of 21.11 t ha^{-1}. In Andhra Pradesh state, cabbage production is about 0.3883 mt (0.68% of Indian cabbage production) from 0.00155 mha (0.57% Indian cabbage cultivated area) with an average yield of 25 t ha^{-1} (CMIE, 2009). However, insect pests are major limiting factor in productivity of these vegetables. In India, it is estimated that at least 52% loss in marketable yield occurred due to diamond back moth attack alone and loss could be more than 80% when attack is severe (Chellaiah and Srinivasan, 1986). Alternate use of chlorpyriphos and deltamethrin would be profitable and most effective method of control of diamond back moth (Nathuram et al., 2001). Even though, these are the most commonly used economical insecticides, the information on their persistence in cabbage is not available. Hence, dissipation of chlorpyriphos and deltamethrin residues were studied in detail.

2. Materials and Methods

Field experiment was conducted during *kharif*-2007 with cabbage variety, Varun. Each treatment was replicated thrice in a randomized block design. Three sprays of chlorpyriphos (0.05%) and cypermethrin (0.01%) were applied, while first spray was initiated at head formation stage and subsequent sprays were given ten days after first spray with knap sack sprayer. The representative cabbage samples of three heads were collected from each plot at 0 (2 h), 1, 3, 5, 7, 10 and 15 days after three sprays.

2.1. Chlorpyriphos extraction and clean-up

The cabbage heads were chopped, blended and a representative sample of fifty grams was blended with 350 ml of acetone/water (65:35 v/v) for 2 m at high speed. The extract was filtered and the sample extract was transferred to one liter separating funnel to extract the residues with 200 ml mixture of hexane: dichloromethane (1:1 v/v) by vigorous shaking for one minute. The lower aqueous phase is transferred to another one liter separating funnel. The organic phase of the first separating funnel is dried by passing through approximately 1.5" sodium sulphate supported on pre-washed cotton in 4" funnel. To the separating funnel containing aqueous phase, 10 ml saturated sodium chloride solution was added and shaken vigorously for 30s. To this, 100 ml dichloromethane was added and shaken vigorously and the lower organic phase dried while passing through the same sodium sulphate. The extraction is repeated once more with 100 ml dichloromethane and dried

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as above. The extract is then concentrated by using vacuum rotary evaporator. Concentration step was repeated in hexane to remove all the traces of dichloromethane and dissolved the extract in 10 ml acetone for GC analysis. The extraction and clean-up method of chlorpyriphos residues in soil samples was followed as that of cabbage samples.

2.2. Deltamethrin extraction and clean-up

The cabbage heads were chopped, blended and a representative sample of 25 gm of finely chopped cabbage was taken in mixer grinder using 100 ml n-hexane and acetone (1:1 v/v) and filtered through Buckner funnel using Filter Paper No. 1. The extracted flask and Buckner funnel was rinsed with mixture of n-hexane: acetone. The entire fraction was pooled together and concentrated.

The concentrated extract was transferred into one liter separating funnel, to this extract 100 ml each of n-hexane and sodium chloride solution was added. The contents were shaken and kept for separation of layers. The upper organic n-haxane layer was collected over sodium sulphate and lower aqueous layer is re-extracted twice with 50 ml of n-hexane and finally aqueous layer was discarded. The n-hexane fractions were pooled and concentrated and cleaned-up with florisil column. The column was eluted with n-hexane:acetone (9:1) and the elutent was concentrated and analyzed on GC-ECD. The extraction and clean-up method of deltamethrin residues in soil samples was followed same as that of cabbage samples.

2.3. Estimation of chlorpyriphos and cypermethrin

The residues of chlorpyriphos and deltamethrin were determined using Varian 3800 Gas chromatograph equipped with electron capture detector and capillary column VF-1 MS, 15 m, 0.25 mm, id 0.25 mm film thickness. The operating temperatures were detector 300°C, injector 280 °C, column oven programmed at 70°C for 1 m, increased @ 20°C m^{-1} to 150°C for 5 m, increased @ 2°C m^{-1} to 240°C for 15 m (total time 76 m). The carrier gas (nitrogen flow) was 1.0 ml m^{-1} and make up flow was 15 ml m^{-1}. The retention time for chlorpyriphos and deltamethrin was 23.91 and 72.48 m, respectively. The residue data was subjected to regression analysis and waiting periods (T_{tol}), and half life (RL_{50}) were calculated as suggested by Gunther and Blinn (1955), and Hoskins (1961).

3. Results and Discussion

The recovery test was carried out at 0.1 and 0.01 mg kg^{-1} levels for fortification of chlorpyriphos and deltamethrin on cabbage and soil. The percent recovery of chlorpyriphos was 83.75 at 0.01 ppm and 86.12 at 0.1 ppm level of fortification in cabbage (Table 1), while in the soil it was 85.25 and 87.38%, respectively at 0.01 and 0.1 ppm level of fortification. The percent recovery of deltamethrin was 86.39 at 0.01 ppm and 89.26 at 0.1 ppm level of fortification in cabbage (Table 1), while in the soil it was 86.61 and 88.47%, respectively at 0.01 and 0.1 ppm level of fortification.

Chlorpyriphos and deltamethrin residues in cabbage and soil are presented in the Table 2. The initial deposit of chlorpyriphos (2.75 mg kg^{-1}) was dissipated to below detectable level at 15 days after third spray with corresponding dissipation of 28.00, 59.27, 72.36, 89.45 and 97% at 1, 3, 5, 7 and 10 days after third spray. The initial deposits obtained in the present study was in conformity with the studies conducted on tomato by Peter et al. (2001) who reported that the initial deposits of chlorpyriphos in tomato was 3.01 mg kg^{-1}. Based on the first order kinetics, the half life of chlorpyriphos was 1.76 days. The half life values in the present study was in conformity with Raina and Raina (2008) who reported that the half life

Table 1: Percent recovery of chlorpyriphos and deltamethrin in cabbage and soil

Fortification level (mg kg^{-1})	% recovery of chlorpyriphos		% recovery of deltamethrin	
	Cabbage	Soil	Cabbage	Soil
0.10	86.12	87.38	89.26	88.47
0.01	83.75	85.25	86.39	86.61

Table 2: Dissipation of chlorpyriphos (0.05%) and deltamethrin (0.07%) residues in cabbage

Day after third spray	Chlorpyriphos		Deltamethrin	
	Residues (mg kg^{-1})	Dissipated (%)	Residues (mg kg^{-1})	Dissipated (%)
0	2.75	-	1.21	-
1	1.98	28.00	0.64	47.10
3	1.12	59.27	0.13	89.26
5	0.76	72.36	0.04	96.69
7	0.29	89.45	BDL	BDL
10	0.08	97.00	BDL	BDL
15	BDL	BDL	BDL	BDL
Soil	BDL	BDL	BDL	BDL
MRL (mg kg^{-1})	0.50		0.20	
T_{tol} (days)	6.53		1.39	
$T_{1/2}$ (days)	1.76		1.34	
Regression equation	Y=2.273 + (-0.256) X9705344949		Y=1.011 + (-0.224) X	

BDL=Below detectable level (0.01 mg kg^{-1}); MRL= Maximum residue limits

values of chlorpyriphos ranged from 1.4 to 1.5 mg kg^{-1} when chlorpyriphos was sprayed at 500 g ai ha^{-1} in cauliflower. The maximum residue limit of chlorpyriphos in cabbage was 0.5 mg kg^{-1} (Codex alimentarius, 1998). The waiting period for safe harvest of cabbage heads was 6.53 days after three sprays of application of chlorpyriphos @ 0.01% for safe consumption. The findings of Kaushik (2002) were in conformity with the present results who reported that the safe waiting period for grape was 6.14 days after three sprays of chlorpyriphos application.

The initial deposits of deltamethrin was 1.21 mg kg^{-1} that dissipated to below detectable level on 7th day after third spray with corresponding dissipation of 47.10, 89.26 and 96.69% at 1, 3 and 5 days after last spray, respectively. Based on the first order kinetics, the half life of deltamethrin was 1.34 days. The maximum residue limit for deltamethrin in cabbage head is 0.2 mg kg^{-1} (Codex alimentarius, 1998). The waiting period for safe harvest of cabbage heads was 1.39 days after three sprays of application of deltamethrin @ 0.01% for safe consumption. Babu et al. (2001) reported that safe waiting period for cabbage heads was three days after application of cypermethrin in cabbage. Singh et al. (2003) reported that betacyfluthrin on cabbage dissipated to below detectable levels at 20 days after second spray when betacyfluthrin sprayed at 25.0 g ai ha^{-1}, while Ahuja et al. (2006) reported that deltamethrin on brinjal dissipated to below detectable level at 10 days after second spray . The variation of the results of the dissipation pattern of deltamethrin in cabbage may be due to variation in the chemical and dosage of the chemical.

4. Conclusion

From the foregoing discussion, it is understood that within 6.53 and 1.39 days (safe period) after spraying of chlorpyriphos 0.05% and deltamethrin 0.01%, respectively, thrice also, the cabbage is safe for consumption. Because, the initial deposits of chlorpyriphos and deltamethrin when sprayed thrice at 0.05% and 0.01% during head formation stage were 2.75 and 1.21 mg kg^{-1}, respectively which were dissipated to below detectable levels.

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