Persistence of Fipronil and Bifenthrin Residues in Cabbage

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Abstract

The experiment was conducted during *kharif* 2007 with cabbage variety Varun. Three sprays of fipronil @ 100 and bifenthrin @ 50 g a.i. ha^{-1} respectively were sprayed at head formation stage. The dissipation pattern of fipronil revealed that initial deposit of fipronil (1.69 mg kg^{-1}) was dissipated to below detectable residues at 10 days after third spray. While, in case of bifenthrin, the initial deposit (1.03 mg kg^{-1}) was dissipated below detectable level at 7 days after third spray. The maximum residue limit for fipronil and bifenthrin was found to be 0.2 and 0.1 mg kg^{-1}, respectively. The waiting period for safe harvest of cabbage heads after three sprays of fipronil and bifenthrin @ 100 and 50 g a.i. ha^{-1} at head formation stage was 5.19 and 5.16 days, respectively. The half life values for fipronil and bifenthrin was 3.13 and 3.38 days, respectively.

1. Introduction

Cabbage is the fourth most widely grown vegetable crop of our country. However, insect pests are major limiting factor in productivity of the cabbage. 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, cabbage production is about 0.38883 mt (0.68% of Indian cabbage production) from 0.0155 mha (0.57% Indian cabbage cultivated area) with an average yield of 25 t ha^{-1} (CMIE, 2009). Insect pests and diseases are important biotic constraints in vegetable production in India, causing considerable losses. Farmers use a number of pest control methods to mitigate these losses. Chemical pest control being the preferred strategy in practice, fruits and vegetables are important recipients of chemical pesticides in India. Fipronil belongs to phenylpyrazole group of insecticides and has been found effective for the management of DBM on cauliflower (Bharadwaj et al., 2005). 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). Even though, these are the most commonly used economical insecticides, the information on their persistence in cabbage is not available. Hence, dissipation of fipronil and bifenthrin residues was studied in detail.

2. Materials and Methods

Field experiment was conducted during *kharif*-2007 with cabbage variety, Varun replicated thrice in a randomized block design at students' farm, College of Agriculture, Rajendranagar, Hyderabad, Andhra Pradesh, India. Three sprays of fipronil 120 and bifenthrin 80 g a.i. ha^{-1} were applied; first spraying was done 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. Fipronil extraction and clean-up

Extraction and cleanup of fipronil residues in cabbage leaf and soil samples were done by the method suggested by the Jimenez et al., (2007) where twenty five grams of the cabbage leaf and soil samples were taken and final volume was concentrated to 5 ml.

2.2. Bifenthrin 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 entire fraction was pooled together, concentrated and

transferred to a one liter separating funnel. 100 ml each of n-hexane and sodium chloride solution was added, 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 bifenthrin residues in soil samples was followed same as that of cabbage samples.

2.3. Estimation of fipronil and bifenthrin

The residues of fipronil and bifenthrin was determined using SHIMADZU 2010 A Gas chromatograph equipped with electron capture detector and capillary column DB 1 capillary column, 30 mts, 0.25 mm ID, 0.25 µm film thickness. The operating temperatures were detector 300^{o}C, injector 260 ^{o}C, column oven programmed at 180^{0}C-5 min-3^{0}C-210^{0}C-5-min-20^{0}C-240^{0}C-10min (total time 31.5 m). The carrier gas (nitrogen flow) was 1.0 ml m^{-1} and make-up flow was 25 ml m^{-1}. The retention times for fipronil and bifenthrin was 15.30 and 28.27 m. 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

Prior to sample analysis, a recovery test was conducted. For the recovery test, 25 grams of the cabbage sample and soil collected from control samples were fortified at two levels separately i.e. 0.01 ppm and 0.1 ppm of fipronil and bifenthrin from the standards prepared. The contents were mixed thoroughly and the samples extracted and cleaned up as per the procedure described above and recoveries of fipronil and bifenthrin were presented in Table 1.

The mean percent recovery of fipronil in cabbage leaf and soil samples were 82.76 and 85.13 at 0.01 ppm, respectively and 84.74 and 86.75 at 0.1 ppm level of fortification respectively. The mean percent recovery of bifenthrin in cabbage leaf and soil samples were 87.28 and 85.91 at 0.01 ppm, respectively and 89.17 and 88.15 at 0.1 ppm level of fortification respectively. Reddy and Reddy (2011) reported that percent recovery of chlorpyriphos and deltamethrin is above 80%, hence the present investigation is in concurrence with the earlier workers.

Fipronil and bifenthrin residues in cabbage and soil are presented in the Table 2. The initial deposit of fipronil (1.69 mg kg^{-1}) was dissipated to below detectable level at 10 days after third spray with corresponding dissipation of 26.35, 47.92, 78.70 and 96.45% at 1, 3, 5 and 7th day after third spray of fipronil @ 100 g a.i. ha^{-1}. The initial deposits obtained in the present study was in conformity with the studies conducted on tomato by Lakra and Daahiya (2008) who reported that the initial deposits of fipronil in bhendi was 1.79 mg kg^{-1}. Based on the first order kinetics, the half life of fipronil was 3.13 days. The half life values in the present study was in conformity with Rupal and Dethe (2009) who reported that the half life values of fipronil ranged was 2.75 days when fipronil was sprayed at 300 g a.i. ha^{-1} in tomato. The maximum residue limit of fipronil in cabbage was 0.02 mg kg^{-1} (Codex alimentarius, 1998). The waiting period for safe harvest of cabbage heads was 5.19 days after three sprays of application of fipronil @ 250 g a.i. ha^{-1} for safe consumption. The findings of Dahiya and Lakra (2008) were in conformity with the present results who reported that the safe waiting period for tomato was 5.99 days after three sprays of fipronil application. The firpronil residues were not detected in the soil collected at 15 days after second spray.

Table 1: Percent recovery of fipronil and bifenthrin in cabbage and soil

Fortification level	Fipronil		Bifenthrin	
	Cabbage	Soil	Cabbage	Soil
0.01 ppm	82.76	85.13	87.28	85.91
0.10 ppm	84.74	86.75	89.17	88.15

Table 2: Dissipation of fipronil and bifenthrin residues in cabbage

Day after third spray	Fipronil		Bifenthrin	
	Residues (mg kg^{-1})	Dissipated (%)	Residues (mg kg^{-1})	Dissipated (%)
0	1.69	-	1.03	-
1	1.25	26.35	0.87	15.53
3	0.88	47.92	0.48	53.39
5	0.36	78.70	0.13	87.37
7	0.06	96.45	BDL	--
10	BDL	--	BDL	--
15	BDL	--	BDL	--
Soil	BDL	--	BDL	--
MRL (mg kg^{-1})	0.2		0.10	
T_{tol} (days)	5.19		5.16	
T1/2 (days)	3.13		3.38	
Regression equation	Y=0.913 + (-0.169) X		Y=0.194 + (-0.786) X	

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

The initial deposit of bifenthrin (1.03 mg kg^{-1}) was dissipated to below detectable level at 7 days after third spray with corresponding dissipation of 15.53, 53.39 and 87.37%, respectively at 1, 3 and 5 days after third spray of bifenthrin @ 50 g a.i. ha^{-1} at head formation stage (Table 2). Based on the first order kinetics, the half life of bifenthrin was 3.38 days. The maximum residue limit of bifenthrin in cabbage was 0.10 mg kg^{-1} (Codex alimentarius, 1998). The waiting period for safe harvest of cabbage heads was 5.16 days after two sprays of application of bifenthrin @ 50 g a.i. ha^{-1} for safe consumption. Dhrumarajan et al., 2009 reported that beta cyfluthrin persisted for 7 days after second spray and half life and waiting periods were 2.7 and 2.33 days respectively. The variation in the present study with other workers may be due to variation in the insecticide and crop. The bifenthrin residues were not detected in the soil collected at 15 days after second spray.

4. Conclusion

The fipronil sprayed thrice @ 100 g a.i. ha^{-1} at head formation stage, the initial deposit of fipronil (1.69 mg kg^{-1}) was dissipated to below detectable level at 10 days after last spray. While, in case of bifenthrin when sprayed @50 g a.i. ha^{-1} at head formation stage, the initial deposit (1.03 mg kg^{-1}) was dissipated to below detectable level at 7 days after third spray. The waiting period for safe harvest of cabbage heads after three sprays of fipronil and bifenthrin @ 100 and 50 g a.i. ha^{-1} respectively was 5.19 and 5.16 days.

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