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Dissipation Pattern of Bifenthrin on Dolichos Bean

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A B S T R A C T

An experiment was conducted during *kharif,* 2015 to evaluate the efficacy of eight insecticides *viz.,* fipronil 5% SC, flubendamide 480% SC, chlorantraniliprole 20% SC, bifenthrin 10% EC, profenophos 50% EC, lambda cyhalothrin 5% SC, imidacloprid 17.8 % SL and beta cyfluthrin + imidacloprid 300 OD were studied by spraying thrice replicated thrice in randomized block design on field bean @ 500 g a.i ha^{-1}, 60 g a.i ha^{-1}, 30 g a.i ha^{-1}, 812 g a.i ha^{-1}, 400 g a.i ha^{-1}, 15.63g a.i ha^{-1}, 25 g a.i ha^{-1} and 30 g a.i ha^{-1}, respectively. Against major insect pests of field bean, the dissipation pattern of bifenthrin 10% EC (25 g a.i.ha^{-1}) was studied by collecting samples at regular intervals *i.e.* 0, 1, 3, 5, 7, 10 and 15days after last spray and analyzed. The initial deposits of 2.62 mg kg^{-1} profenophos recorded at 2 hours after last spray dissipated to 1.74, 0.74 and0.22 mg kg^{-1} at 1, 3 and 5 days, after last spray respectively and below determination level (BDL) by 7th day.

Introduction

Pulse crops belong to the family Leguminosae and subfamilies Papilionoideae, Caesalpinoideae and Mimosoideae. They were narrated as the jewels of Indian agriculture, in view of their unique capacity to fill the dietary requirements of protein to thirsty population of rural India, besides their sustainable character through replenishing soil fertility.

The average protein content varies from 18 to 26 per cent. *Lablab purpureus* (L.) Sweet. Popularly known as field bean, hyacinth bean, dolichos bean, country bean, butter bean, and Indian bean which is an important pulse cum vegetable crop in India and is cultivated extensively in recent past for its fresh tender

pods, leaves and seeds and as cattle feed. The fresh and dried seeds constitute major vegetarian source of proteins in the human diet of Indians.

The field bean fresh pods are acceptable and liked by all, especially during winter season under South Indian conditions and it is rich in nutritive value as it is a rich source of carbohydrates, minerals, vitamins, such as vitamin A, vitamin C, fat and fiber. The protein content of field bean is quite high varying from 20.0 to 28.0 per cent (Schaaffhausen, 1963). However, some of these insecticides leave residues on pods and these residues may persist up to harvest. Presence of pesticide residues in the harvested

beans was posing problem at the time of export and in recent times importing countries have rejected few consignments. Pesticide use has increased rapidly over the last two decades at the rate of 12 per cent per year. The extensive and irrational use of pesticides resulted in the presence of residues of insecticides on beans is likely to be associated with severe effects on human health. Hence, great significance has to be given to estimate pesticide residues in beans.

Materials and Methods

The experiment was laid out in a Randomized Block Design (RBD) with 9 treatments including untreated control replicated thrice with individual plot size of 20 m^2 (5mx4 m) and the insecticides *viz.,* fipronil 5% SC, flubendamide 480% SC, chlorantraniliprole 20% SC, bifenthrin 10% EC, profenophos 50% EC, lambda cyhalothrin 5% SC, imidacloprid 17.8 % SL and beta cyfluthrin + imidacloprid 300 OD were studied by spraying thrice replicated thrice in randomized block design on field bean @ 500 g a.i ha^{-1}, 60 g a.i ha^{-1}, 30 g a.i ha^{-1}, 812 g a.i ha^{-1}, 400 g a.i ha^{-1}, 15.63g a.i ha^{-1}, 25 g a.i ha^{-1} and 30 g a.i ha^{-1}, respectively on field bean first at50% flowering and the second and third spray ten days later to evaluate the efficacy against major insect pests and the dissipation studies were conducted for the same by collecting samples at regular intervals *i.e.* 0, 1, 3, 5, 7, 10 and 15 days after last spray in polythene bags and brought to the laboratory immediately for further sample processing in the laboratory as detailed here under.

Fortification and recovery results of bifenthrin on beans

Field bean samples fortified with bifenthrin at 0.05 mg kg^{-1}, 0.25 mg kg^{-1} and 0.50 mg kg^{-1} were analyzed under GC-ECD and the mean recovery of the residues using the method was

84.78, 84.36 and 92.52 per cent, respectively (Table 2). The results shown that the method was suitable for the analysis of bifenthrin residues up to 0.05 mg kg^{-1}, and the limit of quantification (LOQ) was 0.05 mg kg^{-1}.

Hence, the method described above is suitable for the analysis of samples collected from the field sprayed with bifenthrin residues to study the residue dynamics/dissipation pattern. Samples of beans were collected from profenophos @ 400 g a.i./ha sprayed plots at regular intervals i.e. 0, 1, 3, 5, 7, 10 and 15 days after last spray, and analysed for residues following the validated methods. Residues (mg kg^{-1}) were calculated using the formula given below.

$$Residues (mg\ kg^{-1}) = \frac{Sample\ peak\ area\ X\ conc\ of\ std\ (ppm)\ X\ \mu l\ std.\ injected\ X\ Final\ volume\ of\ the\ Sample\ (2\ ml)}{Standard\ Peak\ area\ X\ weight\ of\ sample\ Analysed\ (2\ g)\ X\ \mu l\ of\ sample\ injected} \times recovery\ factor$$

The following parameters were calculated to know the dissipation pattern of the insecticides on cabbage.

Dissipation percentage

$$Per\ cent\ dissipation = \frac{Initial\ deposit - Residues\ at\ given\ time}{Initial\ deposit} \times 100$$

Waiting period

Waiting period (T_{tol}) is defined as the minimum number of days to lapse before the insecticide reaches the tolerance limit. The waiting periods were calculated by the following formula.

$$Ttol = \frac{[a - Log\ tol]}{b}$$

Where,

T_{tol} = Minimum time (in days) required for the pesticideresi due to reach below the tolerance limit.

a = Log of apparent initial deposits obtained in the regression equation ($Y = a + bX$)

tol = Tolerance limit of the insecticide (MRL)

b = Slope of the regression line

Results and Discussion

The field bean crop after attaining of 50 per cent flowering with 10 days sequential interval of three sprayings were sprayed with bifenthrin 10% EC @ 812 g a.i ha^{-1}and the pod samples were collected at regular intervals of zero (2 hours after spray), 1, 3, 5, 7, 10 and 15 days after third spray on field bean. The samples were processed and estimated for residues of fipronil on Gas Chromotography (GC-ECD) (Table 1). The dissipation pattern were presented in table 3 and depicted in figure 1. Initial deposit of bifenthrin was recorded as 2.62 mg kg^{-1}. The residues recorded at 1, 3 and 5th day after third spraying were found to be 1.74, 0.74 and0.22 mg kg^{-1}, respectively and showing a dissipation per cent of 33.59, 71.76 and 91.60, respectively.

The residues were below detectable level (BDL) after 7 days showing 100 per cent dissipation. The regression equation was Y= 2.3841 + (-0.4685) X with R^2 value of 0.9486.

There were no MRLs suggested for bifenthrinin field bean by either Codex Alimentarius Commission (CAC) or by Food Safety and Standards Authority of India

(FSSAI), hence the day at which the bifenthrin residues reached below detectable level (7th day) was considered as safe waiting period.

Since sufficient literature was not available on the dissipation pattern of bifenthrin on field beans, the information available on other crops was also taken into account for discussion.

Table.1 Gas chromatograph parameters

Gas Chromatograph	Gas Chromatography- AGILENT- 7890B
Column	VF-5ms Capillary Column 30 m length, 0.25 mm Internal Diameter, 0.25 ▢ m film thickness; 1% methyl siloxane
Column Oven (^{0}C)	Bifenthrin - Initial 200^{0}C for 6 min - increase @ 20^{0}C/min upto 280^{0}C – hold for 10 min.
Detectors	Electron Capture Detector (ECD)
Detector Temperature (^{0}C)	300
Injector Temperature (^{0}C)	280
Injector Status	Split Ratio: 1:2
Carrier Gas	Nitrogen, Iolar II, Purity 99.999%
Carrier Gas Flow (ml min^{-1})	2
Make-up Flow (ml min^{-1})	25
Retention time (min)	Bifenthrin 11.94

Table.2 Recovery bifenthrin residues in field bean

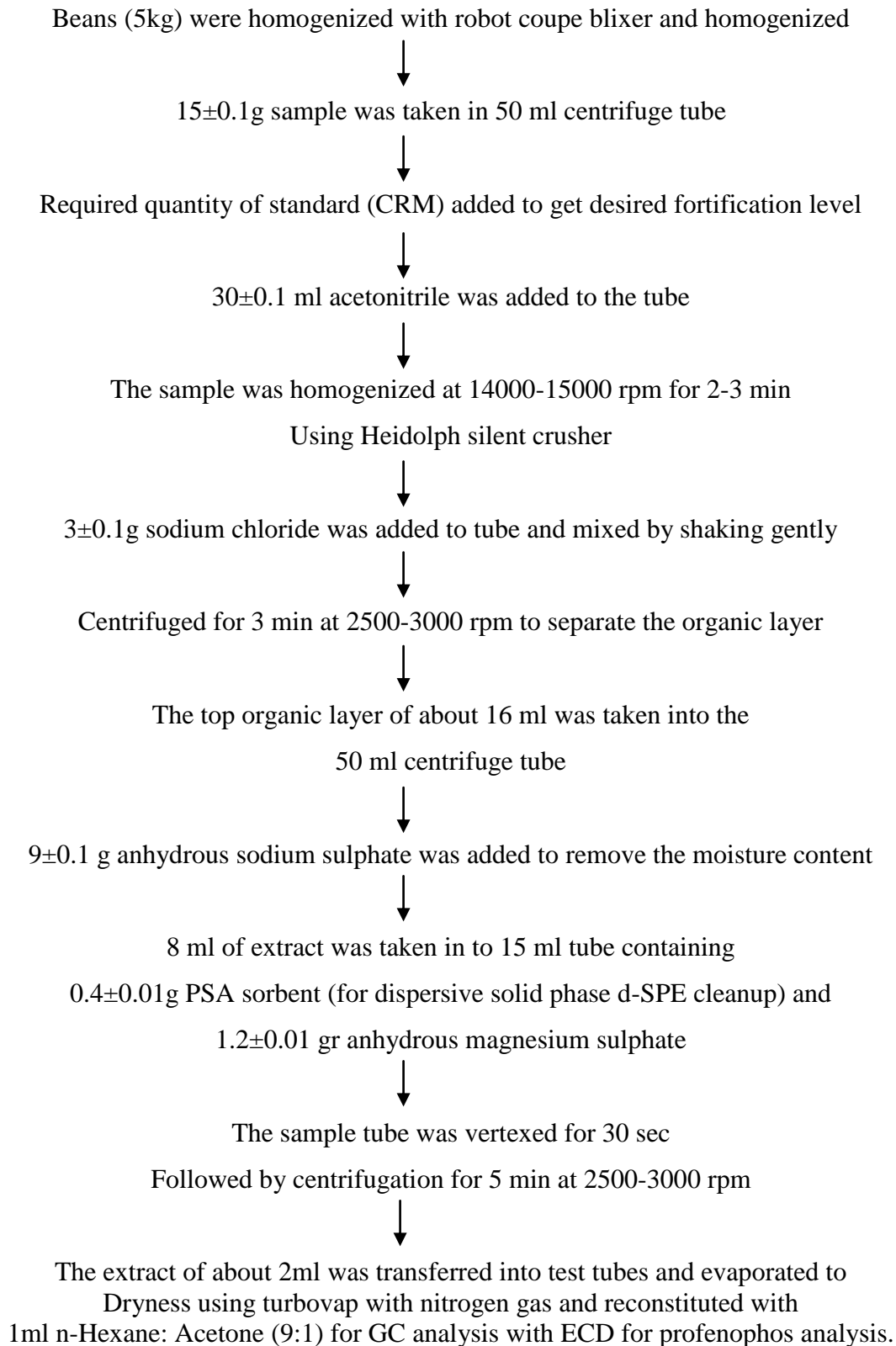| Details	Recoveries of bifenthrin from fortified field bean samples					
	Fortified level					
	0.05mg kg^{-1}		0.25mg kg^{-1}		0.50mg kg^{-1}	
	Residues recovered (mg kg^{-1})	Recovery %	Residues recovered (mg kg-1)	Recovery %	Residues recovered (mg kg-1)	Recovery %
R1	0.0427	85.46	0.211	84.68	0.471	94.17
R2	0.0420	84.09	0.210	84.03	0.454	90.85
R3	0.0427	85.43	0.210	84.27	0.470	94.13
Mean		84.78		84.36		92.52
SD		0.963		0.459		2.35
RSD		1.14		0.54		2.54

Table.3 Dissipation of bifenthrin 10% EC (812 g a.i. ha-1) in field bean after three sprays

	Residues of bifenthrin (mg kg^{-1})				
Daysafter last spray	R1	R2	R3	Average	Dissipation %
0	2.64	2.59	2.65	2.62	--
1	1.75	1.76	1.72	1.74	33.59
3	0.69	0.70	0.85	0.74	71.76
5	0.25	0.23	0.19	0.22	91.60
7	BDL	BDL	BDL	BDL	100
10	BDL	BDL	BDL	BDL	100
15	BDL	BDL	BDL	BDL	100
Regression equation	Y= 2.3841+ (-0.4685) X				
R^2	0.9486				
MRL	NA				
Safe waiting period	7 days				
BDL- Below Determination Level NA- Not Available					

Fig.1 Dissipation of bifenthrin in field bean after three sprays

Extraction and Clean –Up

Beans (5kg) were homogenized with robot coupe blixer and homogenized

↓

15±0.1g sample was taken in 50 ml centrifuge tube

↓

Required quantity of standard (CRM) added to get desired fortification level

↓

30±0.1 ml acetonitrile was added to the tube

↓

The sample was homogenized at 14000-15000 rpm for 2-3 min

Using Heidolph silent crusher

↓

3±0.1g sodium chloride was added to tube and mixed by shaking gently

↓

Centrifuged for 3 min at 2500-3000 rpm to separate the organic layer

↓

The top organic layer of about 16 ml was taken into the

50 ml centrifuge tube

↓

9±0.1 g anhydrous sodium sulphate was added to remove the moisture content

↓

8 ml of extract was taken in to 15 ml tube containing

0.4±0.01g PSA sorbent (for dispersive solid phase d-SPE cleanup) and

1.2±0.01 gr anhydrous magnesium sulphate

↓

The sample tube was vertexed for 30 sec

Followed by centrifugation for 5 min at 2500-3000 rpm

↓

The extract of about 2ml was transferred into test tubes and evaporated to
Dryness using turbovap with nitrogen gas and reconstituted with
1ml n-Hexane: Acetone (9:1) for GC analysis with ECD for profenophos analysis.

The present investigation findings were differed from the findings of Patyal *et al.,* (2010) who reported that persistence of bifenthrin at 60 g a.i.ha^{-1} and 120 g a.i.ha^{-1} on apple fruit and found the residues of bifenthrin initially in the range of 0.669-1.062 mg kg^{-1} at 60 g a.i.ha^{-1} dose and 1.348-1.784 mg kg^{-1} at 120 g a.i.ha^{-1} dose which dissipated to half in 4.85-5.22 and 4.38-6.66 days, respectively and suggested safe waiting period of 2.1 to 5.4 and 6.7 to 11.3 days.

Katroju *et al.,* (2014) reported that the dissipation pattern of bifenthrin 10 EC when applied at the rate of 100 g a.i. ha^{-1} with two sprays of insecticide 10 days interval starting from fruit initiation stage. The initial deposit of bifenthrin was 0.85 mg kg^{-1} which dissipated to 0.39, 0.15 mg kg^{-1} by 1st and 3rd day after last spray, respectively and to BDL by 5th day.

Pre-harvest interval of 3 days was suggested for bifenthrin in tomato. The variation of initial deposits in the present investigation may be due to variation in climatic conditions, geographical location, matrix and dosage applied (Khay *et al.,* 2008). Reddy *et al.,* (2012) reported the dissipation pattern of bifenthrin 10 EC when sprayed @ 50 g a.i. ha^{-1} at head formation stage of cabbage variety Varun followed first order degradation kinetics.

They observed that the initial deposit (1.03 mg kg^{-1}) was dissipated below detectable level at 7 days after third spray. The waiting period suggested for safe harvest of cabbage heads after three sprays for bifenthrin was 5.16 days. Reddy *et al.,* (2014) reported the residue dynamics of bifenthrin 10 EC in cabbage when applied at the rate of 100 g a.i.ha^{-1}. The initial deposits of 2.24 mg kg^{-1}bifenthrin recorded at 2 hours after last spray while, below determination level (BDL) by 10th day. Based on the dissipation pattern a safe waiting period of 3 days was recommended for bifenthrin in cabbage.

The variation of initial deposits in the present investigation may be due to variation in dosage applied, climatic conditions and also matrix. Rama rao and Raja goud (2016) reported that the dissipation pattern of initial deposit of bifenthrin on tomato was 0.24 mg kg^{-1} which dissipated to below detectable level (BDL) by 10th day after first spray in harvested tomato fruits.

The change in initial deposits in the present findings may be due to variation in climatic conditions and matrix (Khay *et al.,* 2008).

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