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Dissipation Pattern of Imidacoprid + Beta Cyfluthrin (Solomon 300 OD) on Dolichos Bean

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ABSTRACT: Three applications of beta cyfluthrin + imidacloprid (Solomon 300 OD) @ 30 g a.i ha^{-1} were made to Dolichos bean *viz*., the first application was made at the 50% blooming stage, and the second and third applications were made at intervals of 10 days. For the purpose of analysing the dissipation pattern, green pod samples were regularly taken at intervals of two hours, one day, three days, five days, seven days, and fifteen days following the third spray. After the third spray on the field bean, the initial deposits of imidacloprid, which were 1.11 mg kg^{-1}, were reduced to 0.06 mg kg^{-1} by the fifth day. At 1, 3, and 5 days following the last spray, respectively, residues of 0.85, 0.34, and 0.06 mg kg^{-1} were measured. These residues had dissipation percentages of 23.42, 76.58, and 94.59, respectively. Whereas residues were below detectable levels (BDL) and had a 100% dissipation rate 7 days after the third spray, they had not yet been detected. The initial beta cyfluthrin (Solomon 300% OD) deposits on the field bean fell to 0.06 mg kg-1 by the fifth day following the third spray. Following the prior spray, residual concentrations of 0.33, 0.19, and 0.06 mg kg^{-1} were detected at 1, 3, and 5 days later, respectively. But, 7 days after the third spray, all traces were gone and could not be found (BDL).

Keywords: Initial deposit, Dissipation, Waiting Periods.

INTRODUCTION

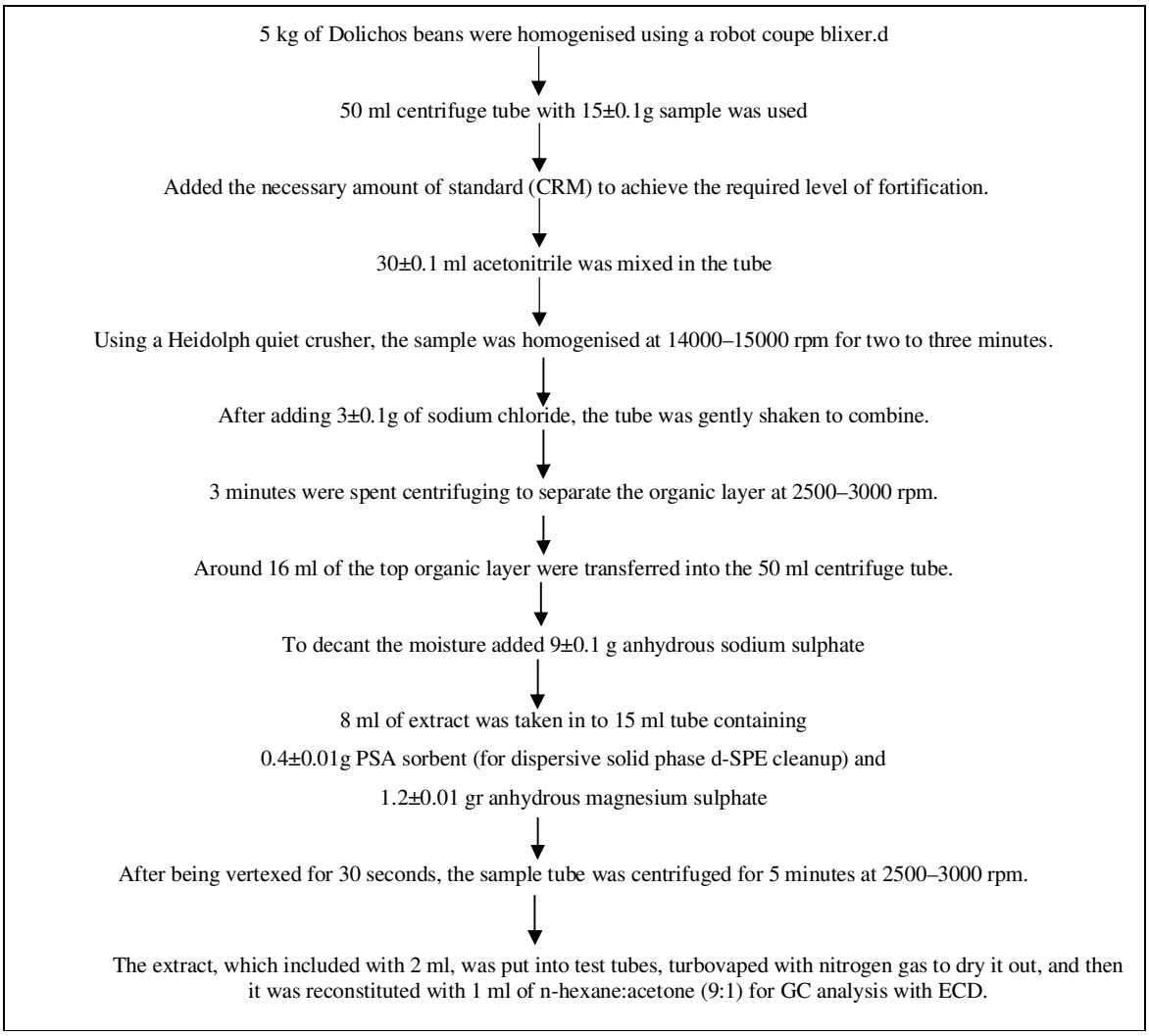
In the family Fabaceae, the Indian bean, *Lablab purpureus var typicus* (L.), also referred to as the garden bean, is one of the significant pulse crops that is grown in both fields and kitchen gardens throughout tropical Asia and Africa. The cultivation of Lablab purpureus var. typicus in India is highly confined to the peninsular region to a large extent in Karnataka and adjoining districts of Tamil Nadu, Andhra Pradesh and Maharashtra (Choudhary *et al*., 2020). The main vegetarian source of proteins in Indians' diets are the fresh and dried seeds. Especially during the winter months in South India, fresh field bean pods are acceptable and well-liked by all. This bean is popularly known as "Sheem" and the scientific name is *Lablab purpureus*, *Dolichos lablab* or *Dolichos niger*. It is said to have its roots in India (Sibiko *et al.,* 2013). However a significant infestation of a variety of pest complex is the main factor blamed for reduced field bean yields. According to Naik *et al*. (2009), pod borers were the main cause of India's low productivity, which resulted in field bean losses of up to approximately 54%. The pod feeders, which comprise both pod borers and pod bugs, caused the majority of the yield loss. Mahalakshmi *et al.* (2016) reported that the principal destructive pest of garden beans, the spotted pod borer *Maruca vitrata* Fabricius (Crambidae - Lepidoptera), is primarily responsible for the crop's low output. The spotted pod borer is an oligophagous pest that affects a variety of legumes, including cowpeas, green and black gram, redgram and yam beans, and field beans. Because to its wide host range, widespread dispersal, and destructiveness, this pest's effects on grain legumes are severe. For the past 20 years, pesticide use has grown significantly, at a rate of 12% annually and since, insecticides are the only option farmers rely on for quick suppression of the pest, the heavy usage of chemicals leads to resistance, residues and environmental pollution. In addition to having negative consequences on the environment, the excessive and illogical application of pesticides left insecticide residues on a variety of edible plant components used for human consumption. The increasing amount of pesticide residues in vegetables has been a big issue to the consumers, as some of these insecticides leave residues on pods which may remain up to harvest. Because of the issue of pesticide residues in the harvested beans, a few consignments have recently been rejected by importing nations. To determine the appropriate waiting periods for safe ingestion, it is crucial to evaluate the pesticide residues in beans and their pattern of dissipation.

MATERIALS AND METHODS

The field experiment involved spraying beta cyfluthrin + imidacloprid 300 OD alongside an untreated control and was replicated three times with individual plot sizes of 20 m^2 (5m × 4 m). The insecticides were applied to

field beans three times at rates of 25 g a.i ha^{-1} and 30 g a.i ha^{-1}, respectively, with the second and third sprays being applied ten days later and the dissipation tests were carried out by gathering samples at certain intervals, specifically 0, 1, 3, 5, 7, 10 and 15 days following the last spray, in polythene bags, and bringing them right away to the lab for additional sample processing as described.

Extraction and Clean –Up



The extract, which included with 2 ml, was put into test tubes, turbovaped with nitrogen gas to dry it out, and then it was reconstituted with 1 ml of n-hexane:acetone (9:1) for GC analysis with ECD (Table 1).

Preparation of working standards. The Certified Reference Materials (CRMs) for beta-cyfluthrin and imidacloprid were obtained from Dr. Erhenstorfer in Germany. Primary, intermediary, and working standards were made from these CRMs using acetone and n-hexane as solvents. Distilled n-hexane was used as the solvent, and a calibrated graduated volumetric flask with a capacity of 10 ml was used to generate working standards for these pesticides. The deep freezer containing all standards was kept at a temperature of -200°C.

Limit of Detection and Linearity of beta cyfluthrin. The working standards of beta cyfluthrin were injected into a gas chromatograph with an electron capture detector (ECD), as given in Table 1, to ascertain the smallest amount of this pesticide that can be detected with an injector split ratio of 1:2 under typical operating conditions.

The limit of detection (LOD) for beta cyfluthrin was found to be 0.01 ng, and the linearity was in the range of 0.01 ng to 0.10 ng as shown in Fig. 1. This was based on the detector's (ECD) reaction to various injections of CRM standards (ng).

Table 1: Details of GC parameters.

Gas Chromatograph	Gas Chromatography- AGILENT- 7890B
Column	VF-5ms Capillary Column 30 m length, 0.25 mm Internal Diameter, 0.25 mm film thickness; 1% methyl siloxane
Column Oven (°C)	Beta cyfluthrin - Initial 180°C - 2 min hold - increase @ 10°C/min upto 260°C - hold time 5 min – increase @2°C/min upto 280°C – hold for 10 min.
Detectors	Electron Capture Detector (ECD)
Detector Temperature (°C)	300
Injector Temperature (°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)	Beta cyfluthrin 19.74

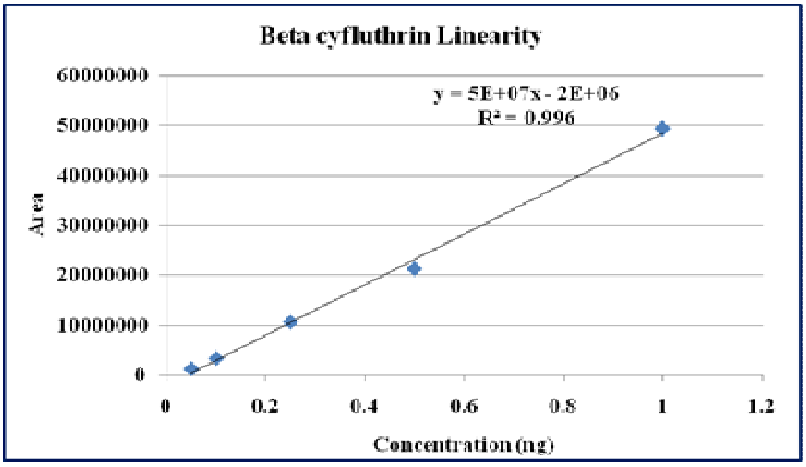


Fig. 1. Calibration curve for beta cyfluthrin

Limit of detection and linearity of Imidacloprid (Solomon 300OD). Working standards of imidacloprid were put into a liquid chromatograph with a mass spectrometer detector to ascertain the smallest amount of imidacloprid that can be detected under typical operating conditions, as indicated in Table 2.

Moreover, samples were injected in HPLC for confirmatory analysis. The LC operating parameters for imidacloprid detection and estimation are given in Table 2. The retention time of imidacloprid is 2.29 min. Each working standards of Imidaclorpid (0.01 ppm, 0.025 ppm, 0.05 ppm, 0.075 ppm, 0.10 ppm, 0.25 ppm and 0.50 ppm) were injected 6 times and the linearity lines were drawn. Based on the response of the Mass Spectrometer to different quantities (ng) of CRM standards injected under the HPLC operational parameters given in table it was found that the LOD (limit of detection) for imidacloprid was 0.05 ng and the linearity was in the range of 0.01 ng to 0.10 ng , as given in Fig. 2.

Table 2: Details of LC operating parameters.

HPLC	SHIMADZU LC-30		
Detector	Mass Spectrometer (MS)		
Column	HPLC Column Kinetex C18 column, 2.6 micron particle size 100 length, 3 mm ID		
Solvents in Pump A Solvents in Pump B	Water Methanol		
Solvents Gradient Program	Water: Methanol (5:95) mixture run for 2 min		
Solvents Gradient rate	0.4 ml min^{-1}		
Quantity of sample injected	1 µl		
Run time	10 min		
Retention time	Imidacloprid- 2.29 min		
LC Program for imidacloprid	Time	Methanol	Water
	0.01	35	65
	4.00	Stop	-

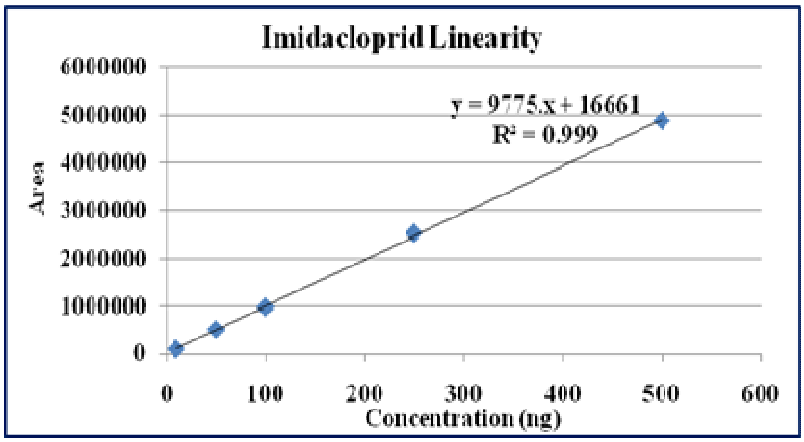


Fig. 2. Calibration curve for imidacloprid.

Method validation. The residue analysis method was confirmed before pesticide application and field sample analysis in accordance with the SANCO document (12495/2011). 5 kg of Dolichos bean pods that were gathered from untreated control plots. The material was homogenised using a Robot Coupe Blixer (High volume homogenizer), and each homogenised sample weighed 15 g before being put into 50 ml centrifuge tubes. Each 15 g sample received the necessary amount of beta cyfluthrin and imidacloprid intermediary standard made from CRMs in order to achieve fortification levels of 0.05 ppm, 0.25 ppm, and 0.5 ppm in each of three replications. These foritifcation values were chosen to determine whether the method was appropriate for locating and measuring pesticides in Dolichos bean.

The AOAC official method 2007.01 (Pesticide Residues of Foods by Acetonitrile Extraction and Partitioning with Magnesium Sulfate) was slightly modified to suit to the facilities available at the laboratory and the same was validated for estimation of LOQ (Limit of Quantification) of above mentioned pesticides in Dolichos bean matrix as given in flow chart above. The final extract of the sample *i.e.* 2 ml equal to 1 g of the sample was evaporated using turbovap and made up to 1 ml (equal to 1 g sample) using suitable solvent for analysis on GC, while for LC analysis, filtered 1 ml final extract (equal to 0.5 g sample) was directly injected in LC and the residues of pesticides recovered from fortified samples were calculated.

Method validation for Beta cyfluthrin (Solomon 300OD). In Dolichos bean samples enriched with beta cyfluthrin at concentrations of 0.05 mg kg^{-1}, 0.25 mg kg^{-1}, and 0.5 mg kg^{-1}, the mean recovery of the residues using the method was 92.43, 89.89, and 97.40%, respectively (Table 3). The approach was demonstrated to allow for the analysis of beta cyfluthrin residues up to 0.05 mg kg^{-1}, and 0.05 mg kg^{-1} was found to be the limit of quantification (LOQ).

Table 3: Recovery of beta cyfluthrin from fortified Dolichos bean samples.

Details	Recoveries of beta cyfluthrin from fortified Dolichos bean samples					
	Fortified level					
	0.05 mg kg^{-1}		**0.25** mg kg^{-1}		**0.50** mg kg^{-1}	
	Residues recovered (mg kg^{-1})	**Recovery %**	**Residues recovered** (mg kg^{-1})	**Recovery %**	**Residues recovered** (mg kg^{-1})	**Recovery %**
R1	0.047	94.29	0.22	86.10	0.49	97.29
R2	0.045	90.84	0.23	93.52	0.47	93.00
R3	0.046	92.16	0.23	90.06	0.51	101.90
Mean		92.43		89.89		97.40
SD		1.74		3.72		4.45
RSD		1.88		4.13		4.57

Method validation for Imidcaloprid (Solomon 300OD). In HPLC analysis, imidacloprid-fortified dolichos bean samples at concentrations of 0.05 mg kg-1, 0.25 mg kg^{-1}, and 0.5 mg kg^{-1} resulted in mean residue recovery rates of 92.50, 97.83, and 96.04 percent, respectively (Table 4). The results shown that the method was suitable for the analysis of imidacloprid residues up to 0.05 mg kg^{-1}, and the limit of quantification (LOQ) was 0.05 mg kg^{-1}.

Table 4: Recovery of imidacloprid from fortified Dolichos bean samples.

Details	Recoveries of imidacloprid from fortified Dolichos bean samples					
	Fortified level					
	0.05 mg kg^{-1}		**0.25** mg kg^{-1}		**0.50** mg kg^{-1}	
	Residues recovered (mg kg^{-1})	**Recovery %**	**Residues recovered** (mg kg^{-1})	**Recovery %**	**Residues recovered** (mg kg^{-1})	**Recovery %**
R1	0.046	91.40	0.235	94.03	0.466	93.21
R2	0.047	93.99	0.244	97.49	0.475	95.04
R3	0.046	92.09	0.255	101.97	0.499	99.86
Mean		92.50		97.83		96.04
SD		1.340		3.979		3.433
RSD		1.449		4.067		3.574

RESULTS AND DISCUSSION

Imidacloprid + Beta cyfluthrin (Solomon 300% OD @ 30 g a.i. ha^{-1}). Three times at 30 g a.i., imidacloprid and beta-cyfluthrin were sprayed. ha^{-1} *viz.*, first spray was given at 50 per cent flowering while second and third spray at 10 days following each spray. After the third spray, field bean pod samples were taken at predetermined intervals of 0, 1, 3, 5, 7, and 10 days. After processing, the samples were analysed for imidacloprid residues using a High Performance Liquid Chromatograph (HPLC) and a Gas Chromatograph (GC).

Imidacloprid (Solomon 300% OD). Initial imidacloprid deposits of 1.11 mg kg^{-1} were reduced to 0.06 mg kg^{-1} by day 5, whereas residues of 0.85 and 0.34 mg kg^{-1} were found at and 3 days, respectively, demonstrating a dissipation percent of 23.42 and 76.58, and were below detectable level (BDL) by day 7. The dissipation pattern of imidacloprid is presented in Table 5 and Fig. 3. The regression equation was Y= 1.0705 + (-0.2132) × with R^2 value of 0.9811 with a waiting period of 4.12 days.

Beta cyfluthrin (Solomon 300% OD). Initial beta cyfluthrin (Solomon 300% OD) deposits of 0.65 mg kg^{-1} dissolved to 0.06 mg kg^{-1} by day five, while residues of 0.33 mg kg^{-1} were detected at days one and three, respectively. By day seven, these residues had decreased to below detectable levels (BDL) (Table 6 and Fig. 4).

Both the Codex Alimentarius Commission (CAC) and the Food Safety and Standards Authority of India (FSSAI) did not have the beta cyfluthrin MRL values for dolichos beans, hence the waiting time began on the day the residues fell below the detectable threshold (7 days).

The current findings are consistent with those of Karabhantanal and Awaknavar (2007) who found that after applying beta cyfluthrin 2.5 EC to tomato variety "PKM-1" at a rate of 7.81 g a.i. ha^{-1}, the initial deposit of 0.925 mg kg^{-1} fell below detectable levels after 7 to 10 days, with a waiting period of 6.75 days. Changes in dosage and matrix could be to blame for the variable in first deposit. The results are in conformity with those of Kousik *et al*. (2010) wherein combination formulation of Solomon 300 0D (beta cyfluthrin 9% + imidacloprid 21%) sprayed @ 60 and 120 g a.i. ha^{-1} at 7 days interval dissipated below the limit of quantification of 0.01 mg kg^{-1} after 5 and 7 days, respectively, whereas, in brinjal it took ten days for imidacloprid in both the dosages to dissipate. The dissipation of beta cyfluthrin 9 percent + imidacloprid 21 percent (Solomon 300 OD) at 60 and 120 g a.i. ha^{-1} on okra, which went below LOQ by 5 and 7 days, respectively, was reported by Sahoo *et al*. (2012). Changes in the crop matrix could be the cause of the variation in the limit of quantification in brinjal and okra. The present findings differ from the results of Dharumarajan *et al*. (2009) who reported the persistence of beta cyfluthrin @ 18.75 g a.i. ha^{-1} and imidacloprid @ 20 g a.i. ha^{-1} as individual and combination mix (Beta cyfluthrin + imidacloprid) @ 40 g a.i. ha^{-1} and 80 g a.i. ha^{-1} on tomato and given that imidacloprid persisted upto 10 days as individual formulation and persisted upto 15 days in combination product. This variation in safe waiting period or maximum persistence period may be due to the change in dose applied and matrix.

Table 5: Dissipation of imidacloprid (Solomon 300 OD) in field bean.

Days after last spray	Residues of imidacloprid (mg kg^{-1})				Dissipation %
	R1	R2	R3	Average	
0	1.11	1.08	1.14	1.11	--
1	0.91	0.78	0.87	0.85	23.42
3	0.38	0.32	0.32	0.34	76.58
5	0.1	0.05	0.05	0.06	94.59
7	BDL	BDL	BDL	BDL	100.00
10	BDL	BDL	BDL	BDL	100.00
15	BDL	BDL	BDL	BDL	100.00
Regression equation	Y= 1.0705+ (-0.2132) X				
R^2	0.9811				
MRL	CODEX Alimentarius Commission (CAC)- 0.2 mg kg^{-1}				
Safe waiting period	4.12 days				
BDL- Below Determination Level					

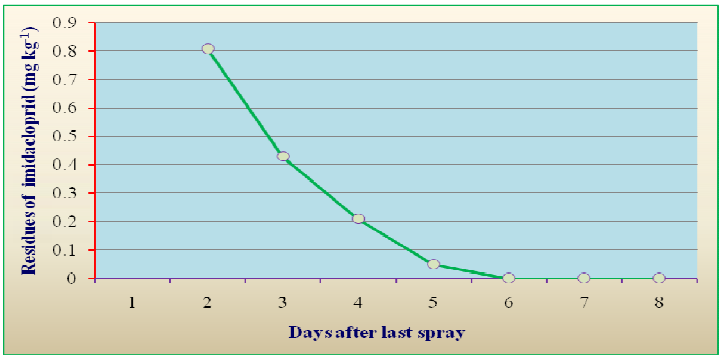


Fig. 3. Dissipation of imidacloprid (Solomon 300 OD) in field bean.

Table 6: Dissipation of betacyfluthrin (Solomon 300 OD) in field bean.

Days after last spray	Residues of betacyfluthrin (mg kg^{-1})				Dissipation %
	R1	R2	R3	Average	
0	0.62	0.67	0.65	0.65	0
1	0.35	0.31	0.33	0.33	49.23
3	0.21	0.17	0.18	0.19	70.77
5	0.05	0.06	0.06	0.06	90.77
7	BDL	BDL	BDL	BDL	100
10	BDL	BDL	BDL	BDL	100
15	BDL	BDL	BDL	BDL	100
Regression equation	Y = 0.5446+ (-0.1063) X				
R^2	0.8637				
MRL	NA				
Safe waiting period	7 days				
BDL- Below Determination Level NA- Not Available					

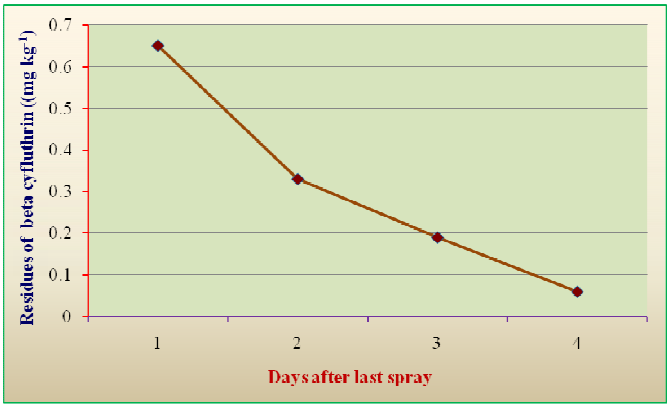


Fig. 4. Dissipation of beta cyfluthrin (Solomon 300 OD) in field bean.

The present findings differ from the results of Dharumarajan *et al.* (2009) who reported the persistence of beta cyfluthrin @ 18.75 g a.i. ha^{-1} and imidacloprid @ 20 g a.i. ha^{-1} as individual and combination mix (Beta cyfluthrin + imidacloprid) @ 40 g a.i. ha^{-1} and 80 g a.i. ha^{-1} on tomato and given that imidacloprid persisted upto 10 days as individual formulation and persisted upto 15 days in combination product. This variation in safe waiting period or maximum persistence period may be due to the change in dose applied and matrix.

The present findings differed from the results of Priyadarshini *et al.* (2014) who reported the dissipation pattern of beta cyfluthrin 25 SC @ 18.80 g a.i. ha^{-1} wherein initial deposit of beta cyfluthrin (18.80 g a.i. ha^{-1}) was 0.12 mg kg^{-1} after second spray on pigeon pea pods. The residues fell below residue level (BDL) after 5th day. The maximum residue limit for beta cyfluthrin (18.80 g a.i. ha^{-1}) is 0.02 mg kg^{-1}. The waiting period for safe harvest of pigeon pea pods after second spray of beta cyfluthrin (18.80 g a.i. ha^{-1}) at pod formation stage was 3.01 days. The variations with respect to initial deposit, below residue level (BDL) and waiting period may be due to number of sprays followed, change in matrix and dosage (Khay *et al.*, 2008).

CONCLUSIONS

The waiting period for safe harvest of field bean green pods when sprayed thrice with imidacloprid (betacyfluthrin + imidacloprid at 30 g a.i. ha^{-1}) and betacyfluthrin (betacyfluthrin + imidacloprid at 30 g a.i. ha^{-1}) were 4.12 and 7 days, respectively. This pre harvest index values are emphasizing the importance of fixing MRL's for safeguarding the human health.

FUTURE SCOPE

MRL's has not been fixed for Dolichos bean separately it is included under podding vegetables so data generation to these chemicals on dissipation in different locations (supervised multi location trials) are essential to set MRL's and waiting periods, so as to suggest the GAP (Good Agricultural Practices) to address the food safety issues, as these insecticides are commonly used by farmers and also field bean is predominantly consumed by people as the fresh vegetable in the form of green pods and further studies on the MRL values for the novel insecticides will give a safeguard measures to human health.

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