The Pharma Innovation Journal 2022; 11(12): 6360-6363

www.ThePharmaJournal.com

The Pharma Innovation

ISSN (E): 2277-7695
ISSN (P): 2349-8242
NAAS Rating: 5.23
TPI 2022; 11(12): 6360-6363

www.thepharmajournal.com
Received: 06-09-2022
Accepted: 15-10-2022

Dr. S Srinivasa Reddy
Assistant Professor, Department of Entomology, Agricultural College, Palem, Professor Jayashankar Telangana State Agricultural University, Hyderabad, Telangana, India

Dr. C Narendra Reddy
Associate Dean, College of Agriculture, Rajendranagar, PJTSAU, Hyderabad, Telangana, India

Corresponding Author:
Dr. S Srinivasa Reddy
Assistant Professor, Department of Entomology, Agricultural College, Palem, Professor Jayashankar Telangana State Agricultural University, Hyderabad, Telangana, India

Dissipation pattern of Imidacloprid on field bean (*Dolichos lablab*)

Dr. S Srinivasa Reddy and Dr. C Narendra Reddy

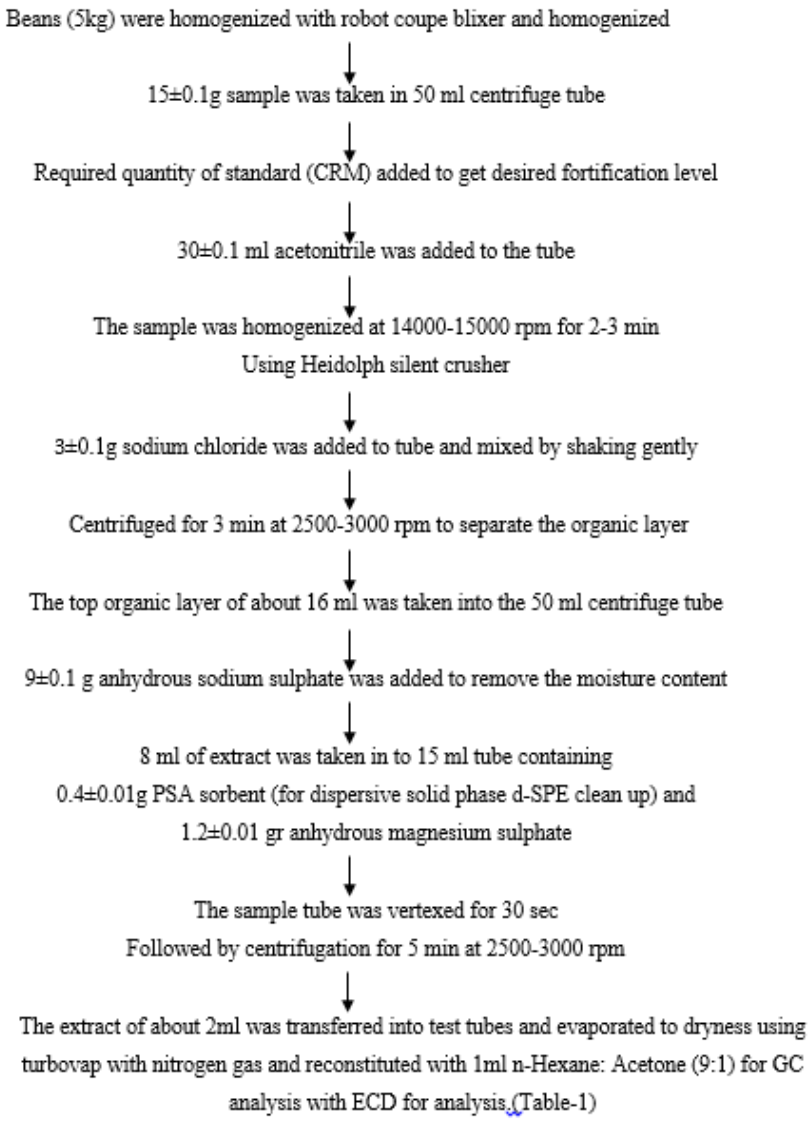
Abstract
Imidacloprid 17.8% SL @ 25 g a.i ha^{-1} was sprayed thrice on fieldbean *viz*., first spray was given at 50 per cent flowering stage while 2nd and 3rd sprays were given at 10 days interval between each spray. Green pod samples were collected at regular intervals at zero (2 hours after spray), 1, 3, 5, 7, 10 and 15 days after third spray for dissipation pattern analysis. The results of imidacloprid residues showed that, the initial deposits of 1.24 mg kg^{-1} were detected on field bean pods. The residues recorded at 1, 3, 5 and 7th day after third spraying were found to be 0.72, 0.31, 0.08 and 0.06 mg kg^{-1}, respectively and showing a dissipation per cent of 41.94, 75.00, 93.55 and 95.16, respectively. The residues reached below detectable level (BDL) at 10 days after last spray showing 100 per cent dissipation.

Keywords: insecticides, efficacy, initial deposit, dissipation, waiting periods

Introduction
Field bean belongs to the family Leguminosae, is an important pulse cum vegetable crop in India and is cultivated extensively for its fresh tender pods, leaves and seeds and as cattle feed. In India this is grown mostly in Andhra Pradesh, Karnataka, Tamil Nadu, Kerala and Assam. 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 are rich in nutritive value and are 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) [7]. However the primary cause attributed for lower yields of field bean is due to the heavy infestation of an array of pest complex. Govindan (1974) [2] recorded as many as
55 species of insects and a species of mite feeding on the crop from seedling stage to the harvest of the crop in Karnataka, of which, the pod borers were considered to be most important as they regularly caused crop loss to the tune of 80-100
per cent (Katagihallimath and Siddappaji, 1962) [4]. Pod borers were the key impediments for the low productivity in India to a loss up to nearly 54 per cent in field beans (Naik *et al*., 2009) [6]. The major yield loss was inflicted by the pod feeders which include both the pod borers and pod bugs. 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 different edible plant parts used for human consumption resulting in various public health problems beside environmental ill effects. The increasing amount of pesticide residues in vegetables has been a major concern to the consumers, as some of these insecticides leave residues on pods which 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. Hence, great significance has to be given to estimate pesticide residues in beans and their dissipation pattern to fix waiting periods for safe consumption.

Materials and Methods
The Field experiment was conducted by spraying imidacloprid 17.8% SL along with untreated control and replicated thrice with individual plot size of 20 m^2 (5mx4 m) and the insecticides were sprayed thrice on field bean @ 25 g a.i ha^{-1} first at 50% flowering, second and third spray ten days later and the dissipation studies were conducted 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.

Extraction and Clean –Up



Preparation of working standards

Certified Reference Materials (CRMs) of imidacloprid was procured from Dr. Erhenstorfer, Germany. Primary, intermediary and working standards were prepared from these CRMs using acetone and *n*-hexane as solvents. Working standards of these pesticides were prepared in the range of 0.01 ppm to 0.5 ppm in 10 ml calibrated graduated volumetric flask using distilled *n*-hexane as solvent. All the standards were stored in deep freezer maintained at -20 °C

Limit of detection and linearity of imidacloprid

The working standards of imidacloprid were injected in Liquid Chromatograph with Mass Spectrometer Detector for estimating the lowest quantity of imidacloprid which can be detected under standard operating parameters as given below in table 1. For confirmatory analysis samples were also injected in HPLC The LC operating parameters for imidacloprid detection and estimation are presented in table 1. The retention time of imidacloprid is 2.29 min.

Table 1: 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	-

Each working standards of above mentioned pesticides (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 1.

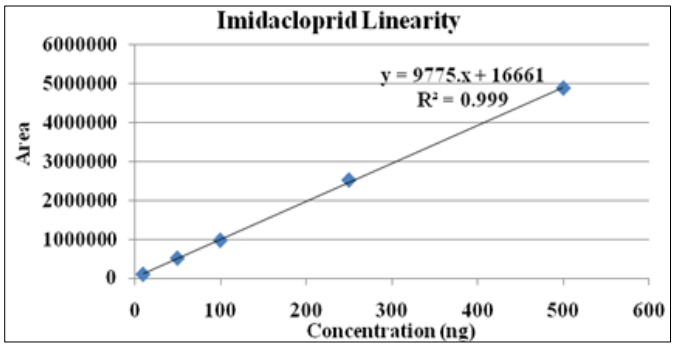


Fig 1: Calibration curve for imidacloprid

Method validation

Prior to pesticide application and field sample analysis, the residue analysis method was validated following the SANCO document (12495/2011). The field bean pods (5 kg) collected from untreated control plots were brought to the laboratory. The sample was homogenized using Robot Coupe Blixer (High volume homogenizer) and homogenized sample of each 15 g was taken in to 50 ml centrifuge tubes. The required quantity imidacloprid intermediary standard prepared from CRMs were added to each 15 g sample to get fortification levels of 0.05 ppm, 0.25 ppm and 0.5 ppm, in three replications each. These foritifcation levels were selected to know the suitability of the method to detect and quantify pesticides in field 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 field 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 Limit of quantification (LOQ) / Limit of determination for Imidacloprid

Filed bean samples fortified with imidacloprid at 0.05 mg kg^{-1}, 0.25 mg kg^{-1} and 0.5 mg kg^{-1} were analysed under HPLC and the mean recovery of the residues using the method was 92.50, 97.83 and 96.04 per cent, respectively (Table 2). 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 2: Recovery of imidacloprid from fortified filed bean samples

Details	Recoveries of imidacloprid from fortified filed 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 17.8% SL @ 25 g a.i ha^{-1}

Imidacloprid 17.8% SL @ 25 g a.i ha^{-1} was sprayed thrice *viz*., first spray at 50 per cent flowering stage while 2nd and 3rd sprays were given at 10 days interval. Green pod samples were collected at regular intervals at zero (2 hours after spray), 1, 3, 5, 7, 10 and 15 days after third spray on field bean. The collected pod samples were processed and estimated for imidacloprid residues on High Performance Liquid Chromatography (HPLC). Dissipation pattern of imidacloprid presented in the table 3 and figure 2. showed that initial deposits of 1.24 mg kg^{-1} were detected on field bean pods and by 1, 3, 5 and 7th day dissipated to 0.72, 0.31, 0.08 and 0.06 mg kg^{-1}, respectively with a dissipation percentage of 41.94, 75.00, 93.55 and 95.16, respectively and fell below detectable level (BDL) at 10 days..The regression equation was Y= 0.9934+ (-0.1598) X with R^2 value of 0.8374. Maximum Residue Limit for imidacloprid in field bean as per Codex Alimentarius Commission (CAC) was 0.2 mg kg^{-1} with a waiting period of 4.35 days.

The present findings are in conformity with the results of Anjumoni and Baruah (2010) [1] who reported the dissipation pattern of imidacloprid on *Brassica campestris*, rapeseed wherein the initial deposits at 20, 40 and 60 g a.i. ha^{-1} were 0.830, 1.126 and 1.280 ppm which fell below detectable level after 5th and 10th day of its application at lower and higher rates, respectively. Based on the observations, a waiting period of at least 4 days after imidacloprid application at recommended dose (20 g a.i. ha^{-1}) was suggested.

Hassanzadeh *et al*. (2012) [3] studied the residues of imidacloprid applied @ 30.0 g a.i. ha^{-1} and its double @ 60.0 g a.i. ha^{-1} in greenhouse cucumbers. The average initial deposits of imidacloprid were 1.93 and 3.65 mg kg^{-1} at the single and double dosages, respectively. A waiting period of 3 days was suggested for safe consumption of cucumber. The variation in initial deposits in the present investigation when compared to Hassanzadeh *et al*. (2012) [3] may be due to the variation in dosages of application in field bean and green house cucumber.

The present findings differ from the results of Swarupa *et al*.

(2016) [8] in curry leaf where initial deposits of imidacloprid were 12.00 mg kg^{-1} and rapidly dissipated to below determination level by 20th day. The variation in dissipation pattern of imidacloprid with the present investigation may be due to change in the matrix and also the dosages applied.

Khay *et al*. (2008) [5] reported that the initial deposits and dissipation pattern vary from crop to crop depending up on the crop canopy, season, age of the crop, sample matrix, surface area of sample etc., and the same was observed by Hassanzadeh *et al*. (2012) [3] and Swarupa *et al*. (2016) [8] on various crops at different doses.

Table 3: Dissipation of imidacloprid 17.8% SL (25 g a.i. ha^{-1}) in field bean

Days after last spray	Residues of imidacloprid				Dissipation %
	R1	R2	R3	Average	
0	1.23	1.24	1.26	1.24	--
1	0.72	0.73	0.72	0.72	41.94
3	0.30	0.32	0.32	0.31	75.00
5	0.07	0.08	0.08	0.08	93.55
7	0.06	0.07	0.07	0.06	95.16
10	BDL	BDL	BDL	BDL	100
15	BDL	BDL	BDL	BDL	100
Regression equation	Y= 0.9934+ (-0.1598) X				
R^2	0.8374				
MRL	CODEX Alimentarius Commission (CAC)- 0.2 mg kg^{-1}				
Safe waiting period	4.35 days				
BDL- Below Determination Level					

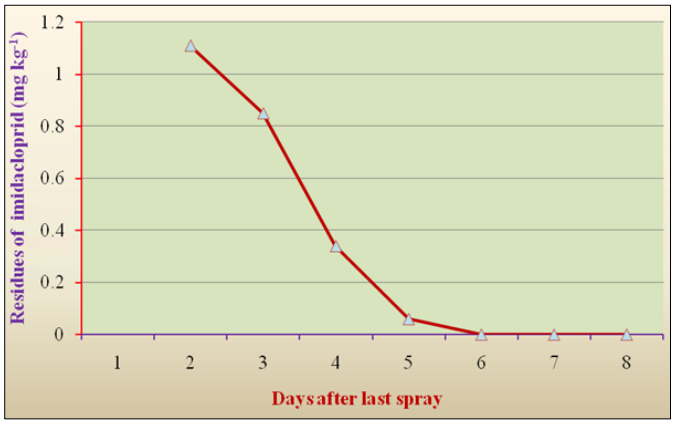


Fig 2: Dissipation of imidacloprid 17.8% SL in field bean

References

1. Anjumoni D, Baruah AALH. Persistence and dissipation of imidacloprid and bifenthrin on rapeseed leaves. Pesticide Research Journal. 2010;22(1):59-62.
2. Govindan R. Insects of the field bean, Lablab purpureus var. lignosus medikus with special reference to the biology and ecology of the pod borer, Adisura atkinsoni Moore (Leipdoptera: Noctuidae). M. Sc. Thesis. University of Agricultural Sciences, Bangalore; c1974. p. 1-3.
3. Hassanzadeh N, Sari AE, Bahramifar N. Dissipation of imidacloprid in greenhouse cucumbers at single and double dosages spraying. Journal of Agricultural Science and Technology. 2012;14(3):557-564.
4. Katagihallimath SS, Siddappaji C. Observations on the incidence of lepidopteran pod borers of Dolichos lablab and the results of preliminary insecticidal trials to control them. 2nd All India Congress of Zoology; c1962. p. 59.
5. Khay S, Choi JH, Abd El-Aty MA. Dissipation behavior of lufenuron, benzoyl phenyl urea insecticides, in/on Chinese cabbage applied by foliar sprayingunder greenhouse conditions. Bulletin of Environmental Contamination and Toxicology. 2008;81:369-372.
6. Naik MI, Tejaswi L, Sridhara S, Manjunath M, Pradeep, S. Yield loss and economic injury level for pod borers of field bean (*Lablab purpureus* L.). Environment and Ecology. 2009;27(3):1044-1047.
7. Schaaffhausen RV. Dolichos lablab or Hyacinth bean, its use for feed, food and soil improvements. Economic Botany. 1963;17:146-153.
8. Swarupa S, Shashi Vemuri, Venkateswar Reddy V, Kavitha K. Dissipation of certain pesticide residues in curry leaf. IOSR Journal of Applied Chemistry. 2016;9(11):19-23.