

## Behaviour of $\beta$ -Cyfluthrin and Imidacloprid in Mustard Crop: Alternative Insecticide for Aphid Control

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Among various insect pests which attack mustard, aphid, *Lipaphis erysimi* (Kaltenbach) is most harmful and limits in realizing the yield potential of this oilseed crop (Ramkishore and Phadke, 1988). A wide range of insecticides have been used for the control of the insect pests of mustard (Naik and Verma, 1992; Ahmad and Rahman, 1994; Mukherjee and Gopal, 2000). The presence of toxic residues of these insecticides in oil and the green leaves consumed as vegetables by humans is a matter of concern as the residues if present in edible portion are hazardous for human beings. It therefore becomes imperative that each pesticidal schedule on oil seed crop be investigated from the point of view of residue. In the present study, two new insecticides namely,  $\beta$ -cyfluthrin and imidacloprid have been evaluated based on their bioefficacy and persistence of their residues in mustard crop infested with aphids and an alternative for conventional molecule oxydemeton methyl is being suggested.

### MATERIALS AND METHODS

Experiments were conducted at the Indian Agricultural Research Institute, New Delhi with mustard (*Brassica juncea*) crop - variety Pusa Bold. Field trials for studying the bioefficacy and persistence of a systemic insecticide imidacloprid and contact insecticide  $\beta$ -cyfluthrin. The crop was sprayed with its 025 SC formulation (Bulldock) @ 12.5 (T1) and 25 g ai ha<sup>-1</sup> (T2) and 200 SL formulation (Confidor) @ 20 (T3) and 40 g ai ha<sup>-1</sup> (T4), respectively. An untreated control (T5) was maintained in which no pesticide was sprayed.

The experiments were laid out in randomized block design with four replications. The plot sizes were 4 x 4 m<sup>2</sup>. Five plants from each subplot in each treatment were tagged for regular monitoring of the aphids. Aphids were counted on 10 cm inflorescence shoot in each plant. Aphid population per 10 cm central shoot of mustard were recorded in relation to other insecticidal treatments. Aphid counts were transformed by log (X+1) and two way analysis of variance was carried out. The sample of mustard leaves were collected periodically after treatment with the insecticides and the pods were collected at harvest time for estimating residues of

each insecticide in seeds. Mustard leaves and grains were fortified with the two insecticides @ 0.5 and 1 mg kg<sup>-1</sup> level separately. The mustard leaf samples (50 g) from plots treated with  $\beta$ -cyfluthrin were homogenized in a blender with 100 ml acetone for two minutes. The extracts were filtered and the residual pulp was extracted twice with 75 ml distilled acetone. Acetone from the filtrate was evaporated off to around 10 ml using a rotary evaporator and transferred to a separatory funnel. 50 ml saline water (2 %) was added to it and then liquid-liquid extraction was carried out with 3 x 30 mL of dichloromethane and the organic layer was passed through anhydrous sodium sulfate and made up to 100 mL. An aliquot (10 mL) of dichloromethane was completely evaporated and the pesticide residues transferred with 5 mL of hexane-toluene (1:1 V/V) mixture on a pre-washed glass column. The glass column (53 cm x 1.5 cm) packed with 5 g neutral alumina was pre-washed with 25 mL hexane. The column was eluted with 100 mL hexane acetone (9:1 V/V) mixture. The final volume was evaporated and made up to 10 mL with 5% acetone in hexane and analyzed by GLC fitted with an electron capture detector. The glass column (2m, 2 mm id) packed with 3% SE-30 coated on 80-100 mesh Chromosorb W.H.P. was maintained at 250°C, injector at 275°C and detector 280°C. Retention time was 7.9 min when the gas flow was 30 mL min<sup>-1</sup>. The minimum detection limit of  $\beta$ -cyfluthrin was 0.01 mg kg<sup>-1</sup>.

A bulk sample of leaves from the plots treated with imidacloprid were quartered and a representative (50 g) sample of mustard leaves was extracted with acetone thrice (3 x 30 mL) in a Waring blender. After filtration, the extract was concentrated under reduced pressure and transferred to a separatory funnel. After addition of 150 mL saline water (2% W/V), the pesticide was partitioned into dichloromethane (3 x 30 mL). The organic solvent was concentrated under reduced pressure and then subjected to clean-up. The column was packed with sodium sulfate (1 g) + neutral alumina (5 g) + sodium sulfate (1 g) and the concentrate was passed into the column and eluted with 1+1 hexane+acetone (V/V, 100 mL). The eluate was concentrated under reduced pressure to remove all traces of hexane and made up to 10 mL with acetonitrile.

A HPLC method was used for the estimation of imidacloprid in mustard leaf and seed. A RP-8 column, acetonitrile solvent system flow maintained at 1 ml min<sup>-1</sup>, UV detector at 270 nm were employed. The retention time of imidacloprid was 3.5 min. The minimum detection limit of the method was 0.05 mg kg<sup>-1</sup> for leaf and seed samples.

## RESULTS AND DISCUSSION

Table 1 shows that there were 58.0, 57.0, 27.5, 19.4 and 270.0 aphids/10 cm central shoot after 1 day and 26.0, 38.6, 3.4, 3.6 and 256.0 aphids/10 cm central shoot after 4 days of application in plants treated with  $\beta$ -cyfluthrin (T1),  $\beta$ -cyfluthrin (T2), imidacloprid (T3), imidacloprid (T4) and in control (T5) respectively.

**Table 1.** Efficacy of  $\beta$ -Cyfluthrin and Imidacloprid against Mustard Aphids.

Pesticide/ Treatment	*Mean aphid population per 10 cm central shoot of a plant				
	Sampling Days				
	1	4	7	11	15
$\beta$ -cyfluthrin T1 (12.5 g ai ha <sup>-1</sup> )	58.0 (1.7501)**	26.0 (1.3935)	73.2 (1.7165)	80.0	3.6
$\beta$ -cyfluthrin T2 (25 g ai ha <sup>-1</sup> )	57.0 (1.7503)	38.6 (1.5793)	70.0 (1.8191)	85.0	9.6
Imidacloprid T3 (20 g ai ha <sup>-1</sup> )	27.5 (1.4357)	3.4 (0.4863)	15.8 (1.1901)	25.0	10.0
Imidacloprid T4 (40 g ai ha <sup>-1</sup> )	19.4 (1.2910)	3.6 (0.5623)	21.0 (1.2439)	55.0	27.0
Untreated T5	270.0 (2.4185)	256.0 (2.4025)	258.0 (2.4090)	Plants withered and aphids deserted plants	Plants died
S.Em. $\pm$	(0.0890)	(0.1790)	(0.1594)		
C.D. %	(0.1887)	(0.3795)	(0.3379)		

\*Mean of five samples unit, \*\*Figures in parenthesis represent log (X+1) values.

The aphid population was significantly less in plots treated with different insecticides as compared to untreated check up to 7 days after application. The aphid population exceeded economic injury level, EIL (69 aphid / plant; Chander and Phadke, 1994). Based on C.D. 5% value of 0.1887 after 1 day and 0.3795 after 4 day of application, insecticidal treatments contained significantly less population than untreated check after 1 and 4 days after application. The pesticides therefore caused statistically significant reduction in pest incidence on mustard crop. After 4 days of application, there was more than 85-99% reduction in pest population over control with  $\beta$ -cyfluthrin and imidacloprid application, respectively. Imidacloprid was found to be more effective than  $\beta$ -cyfluthrin. The insecticidal treatment seemed to be effective for 5 to 6 days as the population showed an increase by 7th day. The increase in aphid population was more rapid in  $\beta$ -cyfluthrin treated plants in comparison to imidacloprid treated plants. This may be due to the systemic nature of imidacloprid.

Imidacloprid has been found effective against sucking pests of cotton (Mote et al., 1995) and whitefly in pulses (Gopal et al., 1997). Phytotonic effect was noted in imidacloprid treated plots. The plants appeared greener, healthier and taller as compared to control plots. Present findings are in agreement with those of Gupta et al. (1998) who have also reported positive correlation in plant height and yield with increase in the dose of imidacloprid in cotton. There are only a few reports about residues of  $\beta$ -cyfluthrin and imidacloprid on crops on which they are being used. There is a need to develop a sensitive and reliable method of estimation of these two insecticides in the food commodity. Methods reported in literature are

lengthy and tedious. The residues of  $\beta$ -cyfluthrin could be estimated by the method standardized in our laboratory for the determination of other synthetic pyrethroids (Mukherjee and Gopal, 1992). A suitable method was required for the estimation of imidacloprid. Placke and Weber's method (1993) requires three step clean-up with XAD-4, Chemelut and Florisil columns and for GLC analysis the compound has to be oxidized to 6-chloronicotinic acid followed by silylation with N-methyltrimethyl silyl trifluoroacetamide. Thus in GLC total residues (imidacloprid and its degradation products) are estimated as imidacloprid. Therefore, a HPLC method has been developed for estimation of residues of imidacloprid in mustard leaf and seed.

The recoveries of the two pesticides varied from 85-90% from both substrates. The persistence data presented in Table 2 show that the residues of 1.45 and 3.45 mg kg<sup>-1</sup> were observed in 0 day samples (collected 1h after application of imidacloprid at two dosages T1 and T2), respectively. The insecticide dissipated with a half life of 4.2-5 day and the residues were below detectable limit (0.05 mg kg<sup>-1</sup>) in seed samples at harvest time. The residues of  $\beta$ -cyfluthrin were lower than that of imidacloprid in initial samples due to lower rate of application. The half life of  $\beta$ -cyfluthrin was also 4.2-5 day.

**Table 2.** Residues (mg kg<sup>-1</sup>) of  $\beta$ -Cyfluthrin and Imidacloprid on Mustard Leaves and Seed.

Treatment (Rate) Days of sampling	$\beta$ -Cyfluthrin		Imidacloprid	
	T1 (12.5 g ai ha <sup>-1</sup> )	T2 (25 g ai ha <sup>-1</sup> )	T3 (20 g ai ha <sup>-1</sup> )	T4 (40 g ai ha <sup>-1</sup> )
0	1.83	2.21	1.45	3.45
1	0.93	1.97	1.07	2.98
4	0.42	0.89	0.89	1.82
7	0.35	0.51	0.51	1.22
10	0.22	0.46	0.21	0.90
16	0.099	0.26	0.12	0.32
Harvest grains	ND	ND	ND	ND
Regression Equation	y = 0.08 -0.072 x	y = 0.27 -0.06 x	y = 0.15 -0.07 x	y = 0.54 -0.06 x
R <sup>2</sup>	0.93	0.93	0.97	0.99
Half life (days)	4.2	5.0	4.3	5.0

\*Average of three replicates

The MRL of  $\beta$ -cyfluthrin and imidacloprid in mustard have not been set by FAO/WHO (1996). The value in Spain for imidacloprid is however stated as 0.05 mg/kg or at the limit of determination in the seeds of oil plants. The residues of other conventional insecticides namely, lindane and chlorpyrifos, have also been

found below their MRL of 0.05 mg/kg in mustard seeds (Singh et al.,1998). Similar results have been reported in blackgram (*Vigna mungo* L) and soybean (*Glycine max* L) after seed treatment with 70 WS formulation of imidacloprid @ 10 g kg<sup>-1</sup> seed, where no residues were found in harvest time seed of these pulses (Gopal et al., 1997). The residue of both the insecticides were below the detectable limit in harvest time seed.

Use of these insecticides could reduce the pest population over control plots effectively, however systemic insecticide imidacloprid was relatively more effective at both the doses than the contact insecticide  $\beta$ -cyfluthrin. Presently oxydemeton methyl (metasystox®) is most popular insecticide for use in mustard crop. The advantage of  $\beta$ -cyfluthrin and imidacloprid over oxydemeton methyl is low dosage, less toxicity and also the ease of estimation for residue analysis as the organophosphorus insecticide oxydemeton methyl has to be converted to its sulphone (Mukherjee and Gopal, 1993) for its analysis by GLC. Other advantage of their application is absence of foul smell in the field which is invariably reported whenever recommended organophosphorus insecticide oxydemeton methyl was used. This point was appreciated by farmers as imidacloprid is odourless and applied in much lower dose than oxydemeton methyl. The new insecticides are less toxic for mammals (Acute oral LD<sub>50</sub> for rats are 450 mg kg<sup>-1</sup> for imidacloprid and 500 mg kg<sup>-1</sup> for  $\beta$ -cyfluthrin as compared to 50 mg kg<sup>-1</sup> for oxydemeton methyl) thereby resulting in reduced toxicity to worker reentering the field. This study highlights the utility of these new insecticides over conventional insecticide metasystox® recommended to control this pest, similar to our earlier conclusion (Mukherjee and Gopal, 1992) wherein synthetic pyrethroids were found superior to an organophosphorus insecticide from residue angle. This observation gains significance in the light of safer alternative concept wherein older products may be withdrawn if it is judged that a new product imidacloprid provides the same benefits and is judged to be safer.

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