Bull. Environ. Contam. Toxicol. (2006) 76:356–360

DOI: 10.1007/s00128-006-0929-7

Bulletin of Environmental Contamination and Toxicology

Persistence of Acetamiprid in/on Mustard (*Brassica juncea* L.)

S. K. Pramanik,[1] J. Bhattacharyya,[1] S. Dutta,[1] P. K. Dey,[2] A. Bhattacharyya[1]

[1] Pesticide Residue Laboratory, Department of Agricultural Chemicals, Bidham Chandra Krishi Viswavidyalaya, Mohanpore-741252, Nadia, West Bengal, India
[2] Department of Agricultural Entomology, Bidhan Chandra Krishi Viswavidyalaya, Mohanpore-741252, Nadia, West Bengal, India

Received: 22 August 2005/Accepted: 28 November 2005

Mustard (B*rassica juncea* L.) is one of the essential important oil seed crop in India which is attacked by various insect pests at the different stages of plant growth, which of them mustard aphid (*Lipaphis erysimi* Kalt), mustard saw fly (*Alhalia lugens proxima* klug.), painted bug (*Bagrada cruciferarum* kirkaldy), bihar hairy caterpillar (*Spilosoma obliqua* walker), cabbage butterfly (*Pieris brassicee* Linn.) are some of the important ones and account for most of the total field losses (Kumar R *et al.* 2001, Bakhetia DRC and Sekhon BS 1989, Narang DD *et al.* 1993, Kumari S and Sing IP1998). It has been found that most of the insects are resist to many insecticides recommended for its control or most of the insecticides which are effective against the above said insects are banned due to presence of their toxic residue in the crop. From safety point of view, eco-friendly insecticides are more convenient to control the aphid, which causes damage of the mustard. Acetamiprid {(E)-N^1- [(6-chloro-3-pryidyl) methyl]-N^2-cyano-N^1-methylaceta-midine} a new generation highly active neonicotinoid group of insecticide has been used to control Hemiptera, especially aphids, Thysanoptera and Lepidoptera on a wide range of crops, especially vegetables, fruits and tea (Roberts T and Hutson D 1999, Mateu-Sanchez et al. 2003). In recent year, a no. of field trials of acetamiprid was done on various crops (Tokieda M *et al.* 1999, Zhen L *et al.* 2000). In order to find out the harvest residues, dissipation pattern of acetamiprid (pride 20 SP) in mustard plant, a three season residue study was carried out at University Research Farm, B.C.K.V., Kalyani.

MATERIALS AND METHODS

The experiment was carried out at the Research Farm of BCKV, West Bengal, Plot size; 50 sq. m, Design; Randomised Block Design (RBD). Acetamiprid (Pride 20 SP) was applied on mustard crop (Variety 'Varunu') at the rate of recommended dose (T_1) and double the recommended dose (T_2), 20 g. a.i./ha and 40 g. a.i./ha respectively along with untreated control (T_3) with three replication for consecutive three season. As PRIDE 20 SP is a soluble powder formulation it was applied by foliar sprayer thrice in 15 days interval. For dissipation study about 1 kg mustard sample were taken randomly from each of treatment replication wise at different time intervals [0, 1, 3 & 7 days] after last

Correspondence to: A. Bhattacharyya

application of acetamiprid. Sample for the control was taken in the same way. Solvents such as methanol, dichloromethane, hexane, acetonitrile and acetone were distilled prior to use. Other chemicals used were of analytical grade.

Plant sample (50 g) was homogenized in a blender with methanol (150 ml). The homogenate was filtered through a celite layer (1-2 con thickness) under reduced pressure. The filter cake and the vessel were washed twice with each 30 ml of methanol. The combined filtrate was placed in a separatory funnel and 5% aqueous sodium chloride solution (150 ml) was added to it. Then the combined aqueous methanolic solution was shaken with 150 ml hexane for few minutes in a separating funnel. The hexane layer was discarded. The aqueous methanolic solution was shaken twice with 125 ml dichloromethane. The dichloromethane layer was passed through anhydrous sodium sulphate and 1 g florisil was added to the organic layer. The dichloromethane layer was concentrated in a rotary vacuum evaporator under reduced pressure at 40 ^{0}C. The residue with 1 g florisil was transferred to the top of the column packed with florisil (10 g) with the aid of hexane. The column was first eluted with 150 ml of mixed solvent of acetone and hexane (20: 80) and it was discarded. Then it was eluted with 150 ml of mixture of acetone and hexane (50: 50). The eluate was collected and concentrated to dryness by rotary vacuum evaporator at 40^0 C. and volume was made upto 10 ml with HPLC acetone for GC-analysis.

The mustard seed and soil samples were collected at harvest for determining the concentration of acetamiprid. The seed sample (20 g) was powdered with the help of electrical grinder and extracted with 100 ml of acetone by homogenization for 5 min in a mixture grinder and filtered through buchner funnel. After collecting the filtrate, the same methodology as described for mustard plant was followed.

The soil sample at harvest (50 g) was dissolved in 150 ml methanol and shaken for 2 hr in a mechanical shaker. Then it was filtered through buchner funnel with 100 ml methanol. The filtrate was concentrated to dryness under reduced pressure at 40^{0}C and thereafter similar steps, which had already been mentioned in mustard plant, were followed. About 50 g mustard seed sample was extracted with n-hexane in a soxhlet apparatus for 4 hr. After removal of the solvent, 1 g oil was taken out and dissolved in 10 ml of hexane and then partitioned thrice with (20 + 20 + 20 ml) acetonitrile. The combined acetonitrile fraction was evaporated in a rotary vacuum evaporator at 40^{0}C and the residue was reconstituted upto 10 ml with HPLC acetone for GC-analysis. The deoiled cake sample (20 g) was blended with acetone for 2-3 min and then akin steps were taken as stated in mustard plant.

Final analysis of acetamiprid residue in mustard plant, mustard oil, deoiled cake and soil were done by GC (Model 5890A, Hewlett packard USA, attached with ECD) coupled with Chemito 5000 data processor. Operating parameters were; Column: DB-5 (Megabore); 30 m x 0.53 mm i.d., 1.5 µm film thickness (J & W Scientific, USA). The temperatures were: Oven temp. 260^{0}C, Injection temp. 300^{0}C, Detector temp. 300^{0}C. Flow rate of carrier gas (Nitrogen) was 40 ml/min. The retention time was 1.88 min.

The average recovery of acetamiprid from mustard plant, mustard seed, mustard oil, deoiled cake and soil samples spiked at 5.0, 1.0 and 0.5 were in the range of 80-90.8%, 86.5-88%, 85-89.6%, 87.2-92% and 84-93% respectively.

RESULTS AND DISCUSSION

The residue data of Acetamiprid in Mustard plant at different days interval was represented in the following table (1). The corresponding dissipation rate, half-life and regression equation has also been calculated on the basis of residue data. Acetamiprid residue in mustard seed, soil, mustard oil and deoiled cake at harvest time were given in the table (2-5). It has been found from the result that the Acetamiprid residue in Mustard plant declined progressively with time irrespective of any dose taking residues at 0 days as initial residues. The initial deposits (4 hr after spraying) of Acetamiprid in Mustard plant resulting from the recommended dose (T_1) and double the recommended dose (T_2) were 0.38 and 0.91 ppm respectively (Table 1). The dissipation of Acetamiprid in Mustard plant followed first order kinetics. The half-life values were calculated from the regression equation, which were found to be 1.02 and 1.59 d for T_1 and T_2 respectively. No residue was detected in mustard seed, soil, mustard oil and deoiled cake at harvest irrespective of any treatment and season. It was observed that there was no residue detected in the untreated control. The Codex Alimentarus Commission has not established a maximum residue level (MRL) for acetamiprid in any crop. As no residue was detected in the harvest samples it might be stated that acetamiprid may not pose any residual toxicity problem in different mustard samples.

Table 1. Dissipation of acetamiprid 20 SP in mustard plant.

Season	Days after application	Treatment	Residue in ppm (µg/g) M* ± S.D.	Dissipation (%)
Season-I (2001)	0	T_1 (20 g a.i./ha)	0.38 ± 0.14	-
	1		0.20 ± 0.08	47.37
	3		0.05 ± 0.02	86.84
	7		ND	-
	0	T_2 (40 g a.i./ha)	0.91 ± 0.17	-
	1		0.42 ± 0.13	53.85
	3		0.09 ± 0.03	90.11
	7		0.01 ± 0.01	98.90
Regression equation : T_1, $Y = 2.586 - 0.295X$; T_2, $Y = 2.804 - 0.189X$				
Half life ($T_{½}$) : T_1, 1.02d; T_2, 1.59 d.				

Table 2. Harvest time residues of acetamiprid 20 SP in mustard seed.

Treatments	Dosage (g a.i./ha)	Replications	Residues in ppm (µg/g)		
			Season –I (2001)	Season-II (2002)	Season-III (2003)
T_1	20	R_1	ND	ND	ND
		R_2	ND	ND	ND
		R_3	ND	ND	ND
T_2	40	R_1	ND	ND	ND
		R_2	ND	ND	ND
		R_3	ND	ND	ND

Table 3. Residues of acetamiprid 20 SP in mustard oil.

Treatments	Dosage (g a.i./ha)	Replications	Residues in ppm (µg/g)		
			Season –I (2001)	Season-II (2002)	Season-III (2003)
T_1	20	R_1	ND	ND	ND
		R_2	ND	ND	ND
		R_3	ND	ND	ND
T_2	40	R_1	ND	ND	ND
		R_2	ND	ND	ND
		R_3	ND	ND	ND

Table 4. Residues of acetamiprid 20 SP in deoiled cake of mustard.

Treatments	Dosage (g a.i./ha)	Replications	Residues in ppm (µg/g)		
			Season –I (2001)	Season-II (2002)	Season-III (2003)
T_1	20	R_1	ND	ND	ND
		R_2	ND	ND	ND
		R_3	ND	ND	ND
T_2	40	R_1	ND	ND	ND
		R_2	ND	ND	ND
		R_3	ND	ND	ND

Table 5. Harvest time residues of acetamiprid 20 SP in soil cropped with mustard.

Treatments	Dosage (g a.i./ha)	Replications	Residues in ppm (μg/g)		
			Season –I (2001)	Season-II (2002)	Season-III (2003)
T_1	20	R_1	ND	ND	ND
		R_2	ND	ND	ND
		R_3	ND	ND	ND
T_2	40	R_1	ND	ND	ND
		R_2	ND	ND	ND
		R_3	ND	ND	ND

ND = Not detectable (<0.01 ppm)
M* = Mean of three replication

Acknowledgement. We thank DENOCIL Crop Protection Ltd., Mumbai for financial assistance and to BCKV for infrastructural facilities.

REFERENCES

Bakhetia DRC, Sekhon BS (1989). Insect pests and their management in repeseed and mustard. Indian oil seeds Res J 6: 269-299.

Kumar R, Dikshit AK and Prasad SK (2001). Bioefficacy and Residues of Imidachoprid in Mustard. Pestic Res J 13: 213 – 217.

Kumari S and Sing IP (1998). Dissipation of Endosulfan Residue in/on Mustard (*Brassica juncea* L.) and its efficacy against the Mustard Aphid, *Lipaphis erysimi* Kalt. Pestic Res J 10: 59 – 63.

Li Z, Ye XX, Bao HY, Li K (2000). Study on safety assessment of some suitable insecticides applied in citrus of Zhejiang province. Acta Agricultural Zhejiangensis 12 : 390-392.

Mateu-Sanchez M, Moreno M, Arrebola FJ and Martinez Vidal JL (2003). Analysis of acetamiprid in vegetables using gas chromatography-tandem mass spectrometry. Analytical Sci 19: 701-704.

Narang DD, Josan VS and Dilowari VR (1993). Control of mustard aphid, *Lipaphis erysimi* Kalt on *Brassica juncea* coss and *B. campestris* L. grown in the field under different soil water regimes. J Insect Sci 6: 195-196.

Roberts T and Hutson D (1999). Metabolic pathways of agrochemicals. Part two: Insecticides and fungicide. The Royal Society of Chemistry, Cambridge, p 111-120.

Tokieda M, Ozawa M, Kobayashi S, Gomyo T, Takeda M (1999): Research on the actual residues for acetamiprid in crops an soils. J Pest Sci 24: 115-122.