Propargite residues in okra and brinjal fruits

H. BANERJEE, A. GHOSH CHOWDHURY, D. BANERJEE, M. PARAMASIVAM, T. BANERJEE AND S. ROY.

All India Network Project on Pesticide Residues, Department of Agricultural Chemicals, Bidhan Chandra Krishi Viswavidyalaya, Mohanpur-741252, Nadia, West Bengal

ABSTRACT

A supervised field trial was carried out to examine the persistence and dissipation behaviour of propargite (Omite 57 % EC) in okra and brinjal following two spray application @ 570 (T_1) and 1140 (T_2) g. ai/ha. Propargite residue was estimated by HPLC. The LOD and LOQ were found to be 0.03 mg/kg and 0.1 mg/kg respectively for both the substrates. Initial deposits of propargite ranged from 2.95-5.68 mg/kg in okra and 2.20-4.30 mg/kg in brinjal at T_1 and T_2 dosages respectively. The dissipation followed first order reaction kinetics with half-life of 2.38-3.04 days for okra and 5.63-7.06 days for brinjal. The calculated PHI of propargite ranged from 1.08-3.40 for okra and 1.0-7.63 days for brinjal. The residues reached below MRL of 2 mg/kg at 1 days for both the crop.

Key Words: Brinjal, HPLC, okra, propargite and residue

India, blessed with the diverse agro-climatic conditions and soil, is the second largest producer of vegetables in the world and account for 14% share in the total world's vegetable production. government of West Bengal has identified the districts of Nadia, Murshidabad and 24 paraganas as Agri Export Zone (AEZ) of vegetables. Brinjal and okra are considered as the most common food item of the human diet. These crops are also susceptible to pest attack throughout the season and extensive pesticide applications are done by the farmers. These crops ravaged by a wide range of insect pests and mites during its growth period resulting to yield loss (Patil and Nandihalli, 2008). Propargite, 2-[4-(1,1-Dimethylethyl) phenoxy] cyclohexyl 2-propynyl sulfite, is a non-systemic acaricide (Xu, S., 2001), effective in controlling variety of phytophagous mites on okra, brinjal and other solanaceous & malvaceous crops (Aplada Sarlis et al., 1994 and Wilson et al., 2007), but at the same time it may do leave toxic residues (Yu et al., 1997, Jian-hui et al., 2006; Kumar et al., 2008 and Kang et al., 2009). Thus, there is a need to examine the persistence behaviour of propargite residues in brinjal and okra following supervised field trial using GAP under West Bengal agro-climatic condition. The residue data will be useful for fixing the MRL to safeguard the interest of the consumers. With this objective, the present study was undertaken.

MATERIALS AND METHODS

Propargite (98.6 %, purity) and formulation (Omite 57 % EC) was supplied by M/S Crompton Uniroyal Chemicals, New Delhi. All the chemicals and solvents used in the study were analytical grade and solvents were glass distilled prior to use.

Supervised field trial was conducted with propargite in brinjal (cv. Muktakeshi) in the year (2006-07) at District Seed Farm, 'D' Block, BCKV Kalyani, West Bengal and okra (cv. Sravan hybrid) at Horticultural Research Station, B.C.K.V., Mondouri, West Bengal during 2007-08. Propargite (Omite 57 % EC) was sprayed twice at 10 days interval @ 570 (T₁) and 1140 (T₂) g ai/ha along with untreated control (T₃) on brinjal and okra in 90 m² per replication.

Samples of brinjal and okra fruits (1 kg) were collected randomly from each treatment at 0 (2 hr after 2nd spray), 1, 3, 5, 7, 10 and 14 days after the (2nd) spray. These fruits sample were chopped separately and mixed thoroughly. Representative 25 g samples in triplicate were drawn and processed for estimation of propargite residues using HPLC.

A representative chopped and homogenized sample (25 g) was extracted with 100 ml of Isopropanol + hexane (1:1) and filtered through buchner funnel using Whatman no.41 filter paper. The process was repeated twice with the solid residues by using fresh solvent mixture (50 ml). The organic layer were combined and concentrated to 50 ml using rotary vacuum evaporator (RVE) at 40°C. The concentrated extract was partitioned thrice with hexane with addition of 150 ml of 3% aqueous sodium chloride solution. The combined hexane extract was passed over anhydrous sodium sulphate, concentrated (~1-2 ml) and subjected to column chromatography [anhydrous sodium sulphate (5 g) + florisil (10g) deactivated with 5 % water + anhydrous sodium sulphate (5 g)]. After prewash with hexane (50 ml), the extract was loaded and propargite residue was eluted with 5 % acetone in hexane (100 ml). The eluate was evaporated to near dryness by gentle flow of nitrogen stream and residue redissolved in 5 ml of methanol: water (9/1, v/v).

E-mail: hembckv@rediffmail.com

Propargite residues were estimated by HPLC (Agilent - 1200) equipped with UV-Vis detector at 220 nm. The mobile phase used was methanol: water (9/1, v/v) at a flow rate of 1 ml per minute using a RP C-18 (250 x 4.6mm) column. The cleaned samples were injected, retention time and peak area were recorded and verified with the external standard solutions. Under these ideal conditions, the retention time of propargite was found to be 5.25 min, limit of detection (LOD) and limit of quantification (LOQ) were found to be 0.03 mg/kg and 0.1 mg/kg respectively. The residue data was subjected to regression analysis (linearised form of first order kinetics equation) to calculate the half life and Pre Harvest Interval (PHI) of propargite in brinjal and okra.

RESULTS AND DISCUSSION

Recovery experiment of propargite in Brinjal and Okra were conducted at 0.1, 0.5 and 1.0 mg/kg level (Table1). The recovery of propargite ranged from 85.6-88.7 % in brinjal and 85.2-87.5 % for okra. Residue data of propargite (Omite 57 % EC) in brinjal and okra fruit at recommended (T₁) and the double the recommended dose (T2) were presented in Table-2 with their corresponding regression equation, half-life and PHI. In brinjal, the mean initial residue deposit (2) hr after application) of propargite was found to be 2.20 mg/kg (T_1) and 4.30 mg/kg (T_2) , which dissipated to 0.92 and 2.16 mg/kg in seven days, recording a loss of 58.18 and 49.77 % respectively. The residues gradually dissipated to 81.81 and 77.67% on 14th day. Similarly, the initial deposits of propargite in okra was found to be $2.95 (T_1)$ and 5.68(T₂) mg/kg. In this case irrespective of doses, around 50% dissipation took place at three days after last application, which dissipated to 0.19 and 0.39 mg/kg on 10th day amounting to the loss of 93.56% and 93.13% for T₁ and T₂ respectively. No residues of propargite were detected in okra on 14th day onwards at recommended rate of application. The dissipation of propargite was faster in okra than brinjal fruit which may be accounted for their morphology and physiology. For both the crops, propargite was proved to be a pesticide with long persistence.

The dissipation rates of propargite followed first order kinetics. The half life values of propargite residues worked out by using Hoskin (1961) formula were 5.63 and 7.06 days for (brinjal) and 2.35 - 3.04 days (okra) at the application rate of 570 and 1140 g ai/ha, respectively which is comparable with the results reported by Kang *et al.* (2009). The present results indicated that at the recommended dose, the residues of propargite persists upto 10th days in okra fruits and beyond 14th days in brinjal fruits. Government of India has not yet fixed MRL for propargite in brinjal and okra, however, considering the MRL value (2 mg/kg) of other countries the

calculated PHI found to be 1.08 -3.40 days for okra and 1.0 -7.63 days for brinjal. As the initial deposit of propargite on brinjal and okra reduced below MRL (2 mg/kg) at 1 days after application at the recommended dose, it is considered to be safe for human consumption keeping in view of propargite as an alternative acaricide for the management of mites in brinjal and okra.

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Table 1: Results of recovery experiment of propargite in brinjal and okra fruits

Substrates	Fortification levels (mg/kg)	Mean recovery %	RSD
	0.1	85.6	1.52
Brinjal	0.5	87.7	1.37
	1.0	88.7	1.30
Okra	0.1	85.2	0.18
	0.5	86.2	1.06
	1.0	87.5	1.43

Table 2: Residues and dissipation of propargite (Omite 57 % EC) in okra and brinjal fruit @ 570 (T₁) and 1140 (T₂) g a.i./ ha

	Mean residues (mg/kg)[% dissipation]				
Days interval	Okra		Brinjal		
	T_1	T_2	T_1	T_2	
0	2.95	5.68	2.20	4.30	
1	1.95 [33.90]	3.52 [38.03]	1.95[11.36]	3.90[9.30]	
3	1.45 [50.85]	2.26[60.21]	1.66[24.54]	3.03[29.53]	
5	0.51 [82.71]	1.06[81.34]	1.25[43.18]	2.52[41.40]	
7	0.27 [90.85]	0.70[87.68]	0.92[58.18]	2.16[49.77]	
10	0.19 [93.56]	0.39[93.13]	0.66[70.00]	1.63[62.09]	
14	BDL	0.24[95.77]	0.40[81.81]	0.96[77.67]	
Regression equation	Y= 3.4378-0.1264X	Y= 3.6357- 0.0987X	Y= 3.354- 0.0535X	Y= 3.626- 0.0426X	
Half life (-) (days)	2.38	3.04	5.63	7.06	
PHI (days)	1.08	3.40	1.0	7.63	

Figures in the parentheses are the percent dissipation values