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Persistence of Propargite on Okra Under Subtropical Conditions at Ludhiana, Punjab, India

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Abstract Residues of propargite were estimated in okra fruits by high performance liquid chromatography following single application of Omite 57 EC @570 and 1,140 g a.i./ha. Residues of propargite were confirmed by high performance thin layer chromatography. The average initial deposits of propargite were observed to be 1.36 and 3.32 mg/kg, respectively, which were below its maximum residue limit of 2 mg/kg. The residue levels of propargite dissipated below its limit of quantification of 0.02 mg/kg after 7 days at single dose and after 10 days at double dose. The half-life values ($T_{1/2}$) of propargite were worked out to be 0.79 and 0.73 days, respectively, at recommended and double the recommended dosages.

Keywords Okra fruits · HPLC · MRL · Propargite · Residues · HPTLC

Okra, *Abelmoschus esculentus* (L.) also known as lady's finger or bhindi is an export-oriented crop in India, occupied an area of 1.94 thousands hectares in Punjab with an annual production of 14.61 thousands tones per annum. The crop is attacked by leaf hopper, *Amrasca biguttula* (Ishida) and shoot and fruit borer, *Earias vittella* Fab. resulting in deterioration of quality. Several insecticides are recommended for the control of these pests (Anonymous 2008).

Propargite, 2-(4-tert-butyl phenoxy) cyclohexyl prop-2-ynyl sulphite, is a non-systemic acaricide (Fig. 1). It is used for controlling a variety of phytophagous mites on many crops including cotton, vines, fruit trees, vegetables, hops and nuts, etc. (Royal Society of Chemistry 1987). The compound is recently introduced in India as an effective acaricide on a large number of crops most of which are consumed by human beings directly or after processing. Therefore, it has become important to check the dissipation behaviour of propargite in edible raw commodities, processed products and in the environment. Taking all these points into consideration, an attempt was made to study the residue levels and dissipation behaviour of propargite on okra.

Materials and Methods

Okra (var. Punjab-8) was raised and transplanted during February, 2007 according to recommended agronomic practices at Punjab Agricultural University Seed Research Farm, Ladhowal, Ludhiana. Application of propargite (Omite 57 EC) was made at 50% fruit formation @ 570 and 1,140 g a.i./ha using water @ 400 L/ha. Each treatment was replicated thrice and size of each plot was 100 sq m. In control plots, only water was sprayed.

For residue studies, about 1 kg of marketable size fruits were collected from each treated and control plots and brought to the laboratory in polythene bags and processed immediately for residue analysis. Okra fruit samples were collected before and 0 (2 h), 1, 3, 5, 7, 10 and 14 days after application of the insecticide. From the composite samples, a sub sample of 250 g per replicate for all the treatments was drawn after cutting into small pieces. Then a 20 g representative sample of okra fruit was selected from each replicate of all the treatments, for extraction of residues. The residues were determined by using High Performance

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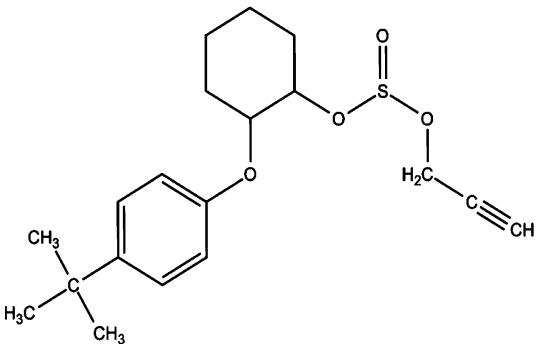

Fig. 1 Structure of propargite

Liquid Chromatography (HPLC) Shimadzu model SPD-M 20A.

A representative 20 g sample of chopped and macerated okra fruit was dipped for overnight into 100 ml acetone in an erlenmayer flask. The extract was filtered in 1L separatory funnel along with rinsings of acetone. The filtrates in separatory funnel were diluted with 600 ml brine solution and partitioned thrice into 75 ml dichloromethane. The dichloromethane fractions were combined, dried over anhydrous sodium sulphate and treated with 500 mg activated charcoal powder for about 2–3 h at room temperature. The clear extracts so obtained were filtered through Whatman filter paper No. 1, concentrated to near dryness and added about 5 ml acetonitrile (HPLC grade) and again concentrated using rotary vacuum evaporator at 30°C. The process was repeated to completely evaporate dichloromethane and final volume was reconstituted to about 5 ml using HPLC grade acetonitrile.

The residues of propargite were estimated on HPLC by employing C_{18} column at 225 nm wavelength and using acetonitrile water (90:10 v/v mobile phase @ 1 ml/min. Under these operating conditions the retention time of propargite was found to be 6.5 min. Residues of propargite were quantified by comparison of peak height/peak area of standards with that of unknown or spiked samples run under identical conditions. Half-scale deflection was obtained for 20 ng propargite. When sample was concentrated up to 2 ml and 20 µl of it was injected (equivalent to 200 mg) did not produce any background interference in the region of the compound being estimated, therefore, limit of quantification (LOQ) was found to be 0.02 mg/kg. The percent recoveries of propargite in okra fruits were found to be consistent and more than 80% (Table 1). Therefore, the results are presented as such without applying any correction factor.

The residues of propargite were confirmed on HPTLC-CAMAG, Linomat 5 applicator, Camag Linomat syringe(100 µl), UV lamp with cabinet; TLC scanner with

Table 1 Recovery of propargite from okra fruits

Level of fortification (mg/kg)	µg added	µg recovered	Recovery (%) (mean ± SD)[a]
1.0	20	18.5	92.5 ± 2.00
0.5	10	9.0	90.0 ± 6.00
0.1	02	1.75	87.5 ± 5.77

[a] Each value is mean ± SD of three replicate determinations

WINCATS (Camag,muttenz,Switzerland); percolated silica gel (60F254)TLC aluminium sheet (Merck,Germany).

Bands of 8 mm length and 6 tracks were applied on each pre-coated TLC plate (10 cm × 10 cm). Linomat-5 applicator was programmed so that bands were applied at a distance of 10 mm from the bottom and at least 12 mm distance between tracks. Distance between tip of the syringe and TLC plate was fixed at 1 mm for sharp application of bands. Sample bands were applied on the TLC plate at the delivery rate of 200 nl/s and nitrogen gas used for drying the spots. Loaded plate was viewed inside the cabinet under UV light (225 nm) to ensure proper application before development. Loaded TLC plates were developed in Camag-Automatic development chamber up to 8 cm in a paper lined chamber pre saturated with 20 mL of chloroform. The propargite bands of developed plate were quantified by absorbance at single wavelength in the Camag TLC scanner 3 using WIN CATS software. Scanning parameters were deuterium lamp wavelength 225 nm, Slit length 6.00 mm, slit width 0.3 mm, band width 30 nm, micromode. Each band was quantified in a single beam, single wavelength reflectance mode, and peak area was taken into consideration for quantitative estimation (Fig. 2). No other information regarding confirmation of propargite by HPTLC is available in literature.

Results and Discussion

The results of dissipation of propargite in okra fruits are presented in Table 2. The average initial deposits estimated in okra fruits were found to be 1.36 and 3.32 mg/kg, respectively, following single application of omite 57EC at @ 570 and 1,140 g.a.i./ha. One day after application, these residue levels dissipated to the extent of about 56% and 57%, respectively, at single and double the application rate (Fig. 3). Half-life ($T_{1/2}$) of propargite calculated as per Hoskins 1961 was observed to be 0.79 and 0.73 days, respectively, when applied @ 570 and 1,140 g.a.i/ha. Residues of propargite in okra fruits dissipated below limit of quantification (LOQ) of 0.02 mg/kg after 7 days at single dose and 10 days at double the recommended dosage (Fig. 4).

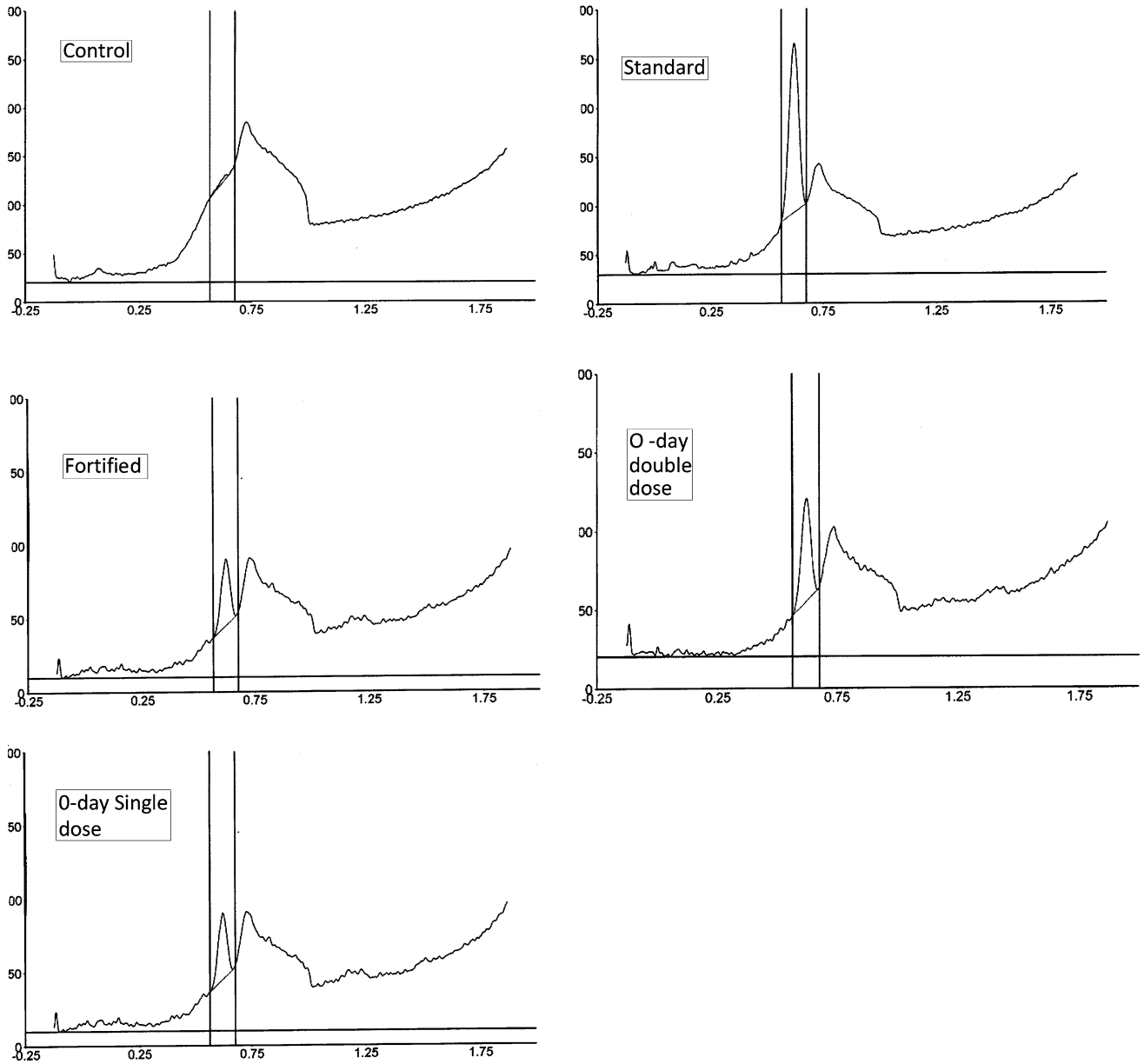

Fig. 2 Confirmation of propargite residues on HPTLC

Aplada-Sarlis et al. (1994) also reported initial deposits of 0.95 and 2.13 mg/kg on green house tomatoes when Omite was applied at 60 and 120 g.a.i/ac, respectively. Samples were analysed at 0, 1, 2, 3, 6, 13, 20, 27 and 34 days after application. The residues of Omite ranged from high 0.95 mg/kg at day 0 to a low of 0.42 mg/kg after 34 days at 60 g dose where as at double the recommended dose, the residue concentrations varied from 2.13 to 1.05 mg/kg at day 0 and 34 days, respectively. Uniroyal (1976) studied the residues of Omite on cotton. They had applied Omite® to cotton foliage at the rates of one or two pounds in 25 gallons water per acre. Samples were analyzed at 0, 14, 28 and 42 days after application and the average residues of propargite were found to be 55, 9.25, 0.40 and 0.10 mg/kg, respectively. The estimated half-life was found to be 3.69 days on cotton. Kumar et al. (2005) studied the dissipation behaviour of propargite in soil, apple fruit and tea. In soil, the half-life was found to range from 43 to 45 days while in plant matrices (tea and apple) it ranged from 1.66 to 2.61 days. According to them the dissipation rate of Omite was found to vary with nature of crop or sample. Kang et al. (2009) reported overage initial deposits of 0.51 and 0.92 mg/kg on brinjal fruits when Omite was applied at 570 and 1,140 g.a.i./ha, respectively.

Table 2 Residues of propargite (mg/kg) in okra fruits

Days after application	570 g a.i./ha			1,140 g a.i./ha		
	Replicates	Mean ± SD	Dissipation (%)	Replicates	Mean ± SD	Dissipation (%)
Before application	BDL	BDL		BDL	BDL	–
	BDL			BDL		
	BDL			BDL		
0	1.48	1.36 ± 0.20	–	3.42	3.32 ± 0.08	–
	1.12			3.26		
	1.48			3.29		
1	0.69	0.59 ± 0.08	56.00	1.55	1.40 ± 0.22	57.00
	0.54			1.51		
	0.55			1.14		
3	0.36	0.38 ± 0.04	72.00	0.79	0.90 ± 0.13	72.00
	0.35			0.88		
	0.44			1.05		
5	0.27	0.23 ± 0.05	83.00	0.50	0.50 ± 0.01	84.00
	0.27			0.52		
	0.17			0.50		
7	BDL	BDL	–	0.19	0.16 ± 0.02	95.00
	BDL			0.14		
	BDL			0.17		
10	BDL	BDL	–	BDL	BDL	–
	BDL			BDL		
	BDL			BDL		
14	BDL	BDL	–	BDL	BDL	–
	BDL			BDL		
	BDL			BDL		
$T_{1/2}$		0.79			0.73	

BDL below determination limit of 0.02 mg/kg

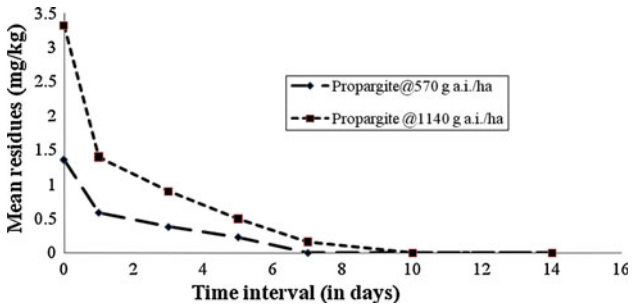

Fig. 3 Dissipation of propargite residues on okra fruits

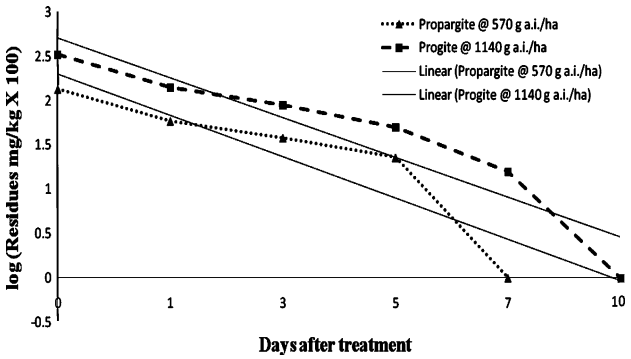

Fig. 4 Semi-logarithm graph showing dissipation kinetics of propargite on okra. Regression equation $y = -0.381x + 2.760$ (for single dose) and $y = 0.412x + 3.151$ (for double dose)

Samples were analysed at 0, 1, 3, 5, 7, 10 and 14 days after application. The residues of Omite ranged from 0.51 mg/kg at day 0 to a low of 0.05 mg/kg after 10 days at 570 g dose where as at double the recommended dose, the residue concentrations varied from 0.92 to 0.12 mg/kg at days 0 and 10 days, respectively. So far, no maximum residue limit (MRL) of propargite on Okra has been assigned, the data may be useful for establishing it. When compared with (MRL) of 2 mg/kg of propargite in vegetables (Food (AMENDMENT) Regulations 2004). It was found that residues of propargite in okra fruits dissipated to safe level after 1 day at higher dose whereas at recommended dose initial deposits of propargite was found to be below the MRL. These studies, therefore, suggests that the use of propargite at the recommended and double the recommended dosages can be considered quite safe from the point of view of health hazards to consumers and a

waiting period of 1 day is suggested to reduce the risk before consumption of okra fruits.

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