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Persistence and decontamination of bifenthrin residues in okra fruits

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Residue levels of bifenthrin were determined in unprocessed and processed okra fruits to evaluate the effect of different household processes on reduction of bifenthrin residues. Bifenthrin (commercial formulation) was applied at 25 g a.i. ha^{-1} (single dose T_1) and 50 g a.i. ha^{-1} (double dose T_2) in field. Okra fruits samples were collected on 0, 1, 3, 7, 10, 15 and 30 days and at harvest (60 days after treatment). Bifenthrin residues were estimated by gas chromatograph- electron capture detector (GC-ECD) system and reached below detectable level of 0.005 mg kg^{-1} on 15 and 30th day in single and double dose, respectively. Half-life period for bifenthrin were found to be 1.58 and 2.18 days at single and double dose, respectively following first order kinetics. Processing was found quite effective in reducing the levels of bifenthrin residues in okra fruits. Maximum reduction (64.58 to 68.42%) was observed by washing + boiling followed by washing (36.71 to 40.00%).

Key words: Bifenthrin, residues, Okra, half-life period, processing.

INTRODUCTION

Vegetables are inseparable part of our daily diet and the vast reserves of growth promoting factors. Among vegetables, Okra (*Abelmoschus esculentus*), is one of the important vegetable crop grown during spring-summer and rainy season in India. It is also one of the important dietary requirements for Indians containing several nutritional values. This crop is subjected to ravage by over 37 insect-pests throughout its growth from germination till harvest (Nayyar et al., 1976), of which, leafhopper, *Amrascabiguttula biguttula* (Ishida) and shoot and fruit borer, *Earias* spp. is a major insect pest of Okra. Leafhopper alone had caused 32.06 to 40.84% (Singh and Brar, 1994) and shoot and fruit borer caused 50% reduction in fruit yield (Brar et al., 1994). Several insecticides are recommended for the control of these pests (Anonymous, 2008).

Bifenthrin [2-methylbiphenyl-3-ylmethyl (Z)-(1 RS)-cis-3-[2-chloro-3, 3, 3-trifluoroprop-1enyl) -2, 2-dimethyl cyclopropanecarboxylate] is relatively new introduction and have shown promise in the management of insect pests of vegetables. It is a third generation synthetic pyrethroid having applications both in agriculture and in public health control. Bifenthrin is effective for control of insect pets of cotton (Ali and Karim, 1994), vegetables (Gupta et al., 2009) and fruits (Reddy and Rao, 2002). Good knowledge of the pesticide fate in agriculture is necessary to properly assess human exposure and the environmental impact of contaminants and the concentrations of the pesticide residues in food commodities. On the other hand, it was observed that pesticide residues in plant products are reduced by processing or some household preparation stages such

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as washing, peeling, cooking, etc. (Dikshit et al., 2003). The effects of some culinary processing steps such as washing, peeling and cooking on pesticide residues have already been investigated in various fruits and vegetables (Sanyal et al., 2006; Osman et al., 2008). The results suggested that residues decreased due to different treatments. Therefore, the present investigation was carried out with the objectives to examine the persistence and dislodging of bifenthrin residues in Okra fruits.

MATERIALS AND METHODS

Chemicals and reagents

All the solvents used for this study were of analytical grade. Formulation (Talstar 10% EC) was procured from local market. The analysis of the formulation in acetone extract with respect to its active ingredient of bifenthrin was estimated using Gas-liquid chromatography (GLC). The results showed that the concentrations of bifenthrin in the formulation were correct as claimed by the manufacturer. Solvents like acetone, dichloromethane and hexane were procured from Merck, Darmstadt, Germany. Sodium chloride (ASC reagent grade ≥ 99.9%) was also obtained from Merck, Darmstadt, Germany. Sodium sulfate anhydrous (AR grade) was from s. d. fine Chemicals Mumbai. Activated charcoal decolorizing powder was obtained from Qualigens Fine Chemicals, Mumbai. All the common solvents were redistilled before use in glass apparatus and their suitability was ensured by running reagent blanks before actual analysis. The stock solution of bifenthrin formulation was prepared at concentration of 100 µg ml^{-1}.

Preparation of standard solution

A standard stock solution of bifenthrin having concentration of 1 mg ml^{-1} was prepared in acetone. The standard solutions required for plotting a calibration curve (2.00, 1.50, 1.00, 0.50, 0.25 and 0.10 µg ml^{-1}) were prepared from stock solution by serial dilution using n-hexane. All standard solutions were stored at 4°C.

Field study

A field study was conducted to determine the persistence of bifenthrin at the Vegetable Research Farm CCS HAU, Hisar in *Pre-kharif* season 2011 on Okra [variety- Varsha uphar] crop. Field trial was laid out in Randomized Block Design (RBD) and replicated three times. Plot size was 5m × 5m with net size 25 m^2. The experimental area was located at 29°10' North latitude and 75°46' East longitude at an elevation of 215.2 m above mean sea level. The region has a semi-arid climate with sever cold winter and hot dry summer with annual rainfall of about 400 mm. Commercial available formulation of bifenthrin as Talstar 10% EC was applied on Okra crop at 25 and 50 g a.i./ha at fruiting stage on 17th May, 2011 with the help of Knapsack sprayer.

Sampling

For residue studies, about 1 kg of marketable size fruits were collected from each treated and control plots on sampling days [0 (1 h), 1, 3, 7, 10, 15 and 30 days and at harvest (60 days)] and brought to the laboratory in polythene bags and processed immediately for residue analysis. The samples were divided into three portions, one portion was processed as such, second after washing and third one after washing followed by boiling/cooking. Washing was performed by placing Okra fruits, in a container and rinsed under normal water for 30 s, with gentle rotation by hand as described by Walter et al. (2000) and blotted dry with a paper towel and divided into two parts. For cooking, in 20 g representative samples of Okra 10 ml water was added and boiled till softness of Okra pieces following the method of Kumari (2008).

Extraction

A representative 20 g sample of chopped and macerated Okra fruit was extracted with 100 ml acetone by shaking on mechanical shaker for 1.5 h (Nath et al., 2005) and filtered in a separatory funnel by passing through anhydrous sodium sulphate. The filtrates in separatory funnel were diluted with 50 ml of saturated solution of sodium chloride and partitioned thrice with dichloromethane (75, 50 and 30 ml). The dichloromethane fractions were combined dried over anhydrous sodium sulphate. The organic layer was concentrated on rotary vacuum evaporator to reduce the volume approximately to 10 ml.

Clean-up

Glass column (60 cm × 22 mm i.d.) was packed compactly with activated charcoal and silica gel (0.3:3 w/w). Pre-wetted the column with 40 ml of hexane, loaded the concentrated extract in the column and eluted with 125 ml of hexane: acetone (9:1 v/v) mixture at flow rate of 2 to 3 ml/min., concentrated the eluate on vacuum evaporator followed by gas manifold evaporator. Final volume was made to 2 ml in n-hexane for GC analysis.

Estimation

The final extracts were analyzed on Shimadzu 2010 Gas chromatograph (GC) equipped with electron capture detector (ECD), capillary column, HP-I (30 m × 0.32 mm × 0.25 µm film thickness of 5% diphenyl and 95% dimethyl polysiloxane) and ECD. The operating parameters of the instrument were: Oven temperatures (°C) 150 (5 min) → 8°C min^{-1} → 190 (2 min) → 15°C min^{-1} → 280° (10 min), injection port 280°C and detector 300°C. Flow rate of nitrogen (carrier gas) was 60 ml/min; through column was 2 ml/min and split ratio 1:10. Under these operating conditions the retention time of bifenthrin was found to be 18.161 min. Chromatograms of bifenthrin, standard curve, control and Okra samples are shown in Figure 1a to d.

RESULTS AND DISCUSSION

Efficiency of the method

In the present investigations, recovery experiments were carried out at different levels to establish the reliability and validity of analytical method and to know the efficiency of extraction and cleanup procedures for soil and water. The control samples of Okra were spiked at 0.01, 0.10 and 0.25 mg kg^{-1}, respectively, and processed by following the methodology as previously described. Mean recoveries of bifenthrin in Okra were found to range from 85.42 to 93.05% (Table 1).

The average recovery values from the fortified samples were found to be more than 85%. Therefore, the results

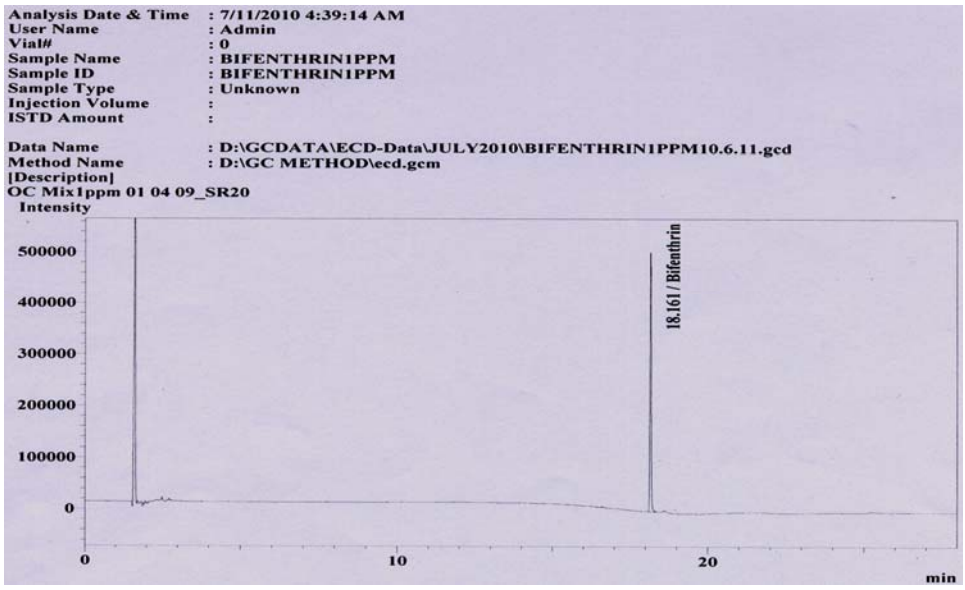

Figure 1a. Chromatogram of standard bifenthrin**.**

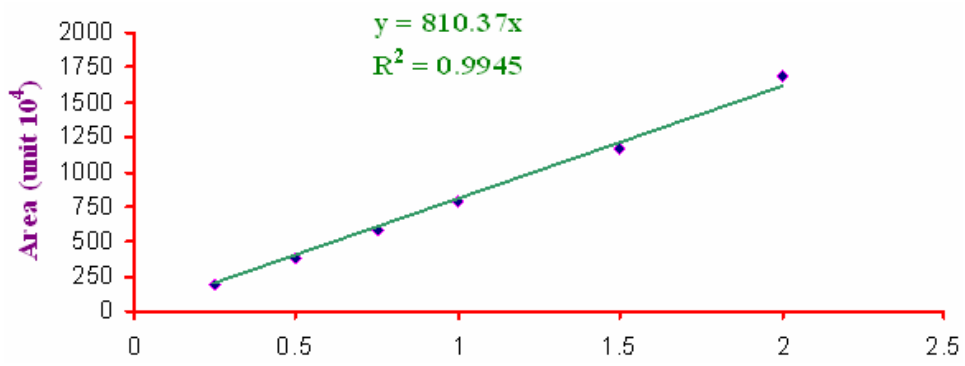

Figure 1b. Standard curve of bifenthrin.

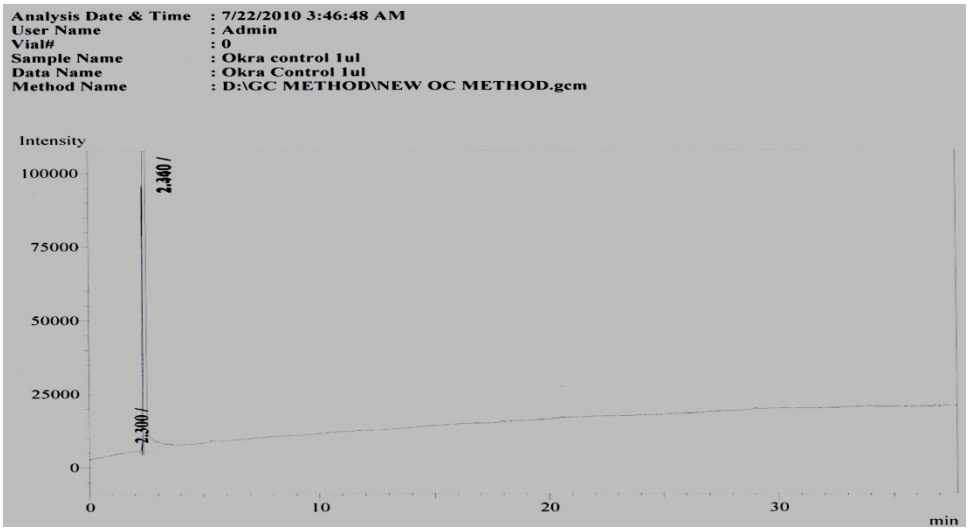

Figure 1c. Chromatogram of control sample of Okra.

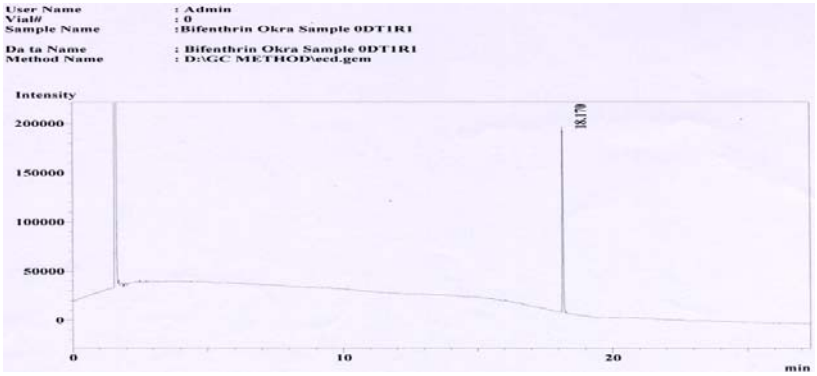

Figure 1d. Chromatogram of Okra sample.

Table 1. Recovery studies of bifenthrin in Okra.

Fortification levels (mg kg^{-1})	Average* recovery (%) ±SD	Mean
0.01	85.42± 325	
0.10	91.00 ± 2.60	89.82
0.25	93.05 ± 4.40	

Table 2. Dissipation of bifenthrin residues (mg kg^{-1})* of on okra fruits.

Days after treatment	Residue (mg kg^{-1})			
	T_1 (25 g a.i.ha^{-1})		T_2 (50 g a.i.ha^{-1})	
	Average ±SD	Dissipation (%)	Average ±SD	Dissipation (%)
0	0.365 ± 0.015	-	0.580 ± 0.002	-
1	0.188 ± 0.007	48.49	0.303 ± 0.008	47.75
3	0.048 ± 0.021	86.84	0.156 ± 0.020	73.10
7	0.008 ± 0.002	97.80	0.038 ± 0.007	93.44
10	0.005 ± 0.012	98.63	0.009 ± 0.002	98.44
15	BDL	-	0.006 ± 0.005	98.96
30	BDL		BDL	
At harvest	BDL	-	BDL	-
Correlation coefficient r	-0.9778		-0.9758	
Regression equation y	2.4231 - 0.1903x		2.6180- 0.1379x	
$t_{1/2}$ (Days)	1.58		2.18	

CD (p = 0.05) for days = 0.002 for dose = 0.001 for days x dose = 0.003. For regression equation, [Residues (mg kg^{-1}) × 10^3] is taken. *Average of three replicates; BDL, Below Detectable Level (0.005 mg kg^{-1}).

have been presented as such without applying any correction factor.The parameters like limit of detection (LOD), limit of quantification (LOQ), precision and accuracy were derived keeping in view the guidelines as mentioned by Thompson et al. (2002). Accordingly, the LOQ was found to be 0.005 mg kg^{-1} and LOD being 0.002 mg kg^{-1}.

The average initial deposits of bifenthrin at single and double dose were observed to be 0.365 and 0.580 mg kg^{-1}, respectively as shown in Table 2. The data shows that the residues were dissipated with time and reached below determination level (BDL) of 0.005 mg kg^{-1} within 15 days in single dose and 0.006 mg kg^{-1} within 30 days in double dose after application. After 15 days, no residues were detected in the marketable fruits in any dose. The half-life period was found to be 1.58 and 2.18 days at single and double dose, respectively, following first order kinetics. The value maximum residue limit (MRL) for bifenthrin is not available, as the insecticide is new introduction. Therefore, the waiting period has been calculated based on acceptable daily intake values (ADI) taken from literature (Anonymous, 2007).

Table 3. Maximum permissible intake (MPI) and theoretical maximum residue contribution (TMRC) values for insecticides on Okra.

Insecticide	ADI	MPI	Dose a.i./ha	TMRC						Waiting period (days)
				0 day	1 day	3 days	7 days	10 days	15 days	
Bifenthrin	0.02	1.00	25	0.091	0.047	0.012	0.002	0.001	-	1
			50	0.145	0.075	0.039	0.009	0.002	0.001	1

MPI (mg per person per day) = ADI × Average body weight (50 kg); TMRC (mg per person per day) = Residue × Average daily consumption (250 g); residues safe when TMRC<MPI.

Table 4. Effect of processing on reduction of bifenthrin residues in okra fruits at single and double dose.

Day	T_1 (25 g a.i.ha^{-1})					T_2 (50 g a.i.ha^{-1})				
	Initial residues ±SD	Washing		Washing+ Boiling		Initial residues ±SD	Washing		Washing + Boiling	
		Residue ±SD	% Reduction	Residue ±SD	% Reduction		Residue ±SD	% Reduction	Residues ±SD	% Reduction
0	0.365 ± 0.010	0.231 ± 0.005	36.71	0.223 ± 0.007	38.90	0.580 ± 0.005	0.348 ± 0.004	40.00	0.334 ± 0.005	42.41
1	0.188 ± 0.005	0.139 ± 0.002	26.06	0.111 ± 0.005	40.95	0.303 ± 0.003	0.210 ± 0.003	30.69	0.161 ± 0.005	46.86
3	0.048 ± 0.005	0.037 ± 0.003	22.91	0.017 ± 0.003	64.58	0.156 ± 0.003	0.118 ± 0.003	24.35	0.068 ± 0.004	56.41
7	0.008 ± 0.002	BDL	-	BDL	-	0.038 ± 0.003	0.030 ± 0.003	21.05	0.012 ± 0.002	68.42
10	0.005 ± 0.002	-	-	-	-	0.009 ± 0.003	BDL	-	BDL	-
15	BDL	-	-	-	-	0.006 ± 0.003	-	-	-	-
30	-	-	-	-	-	BDL	-	-	-	-
At harvest	-	-	-	-	-	-	-	-	-	-

*Average ±SD of three replicates; BDL: 0.005 mg kg^{-1}.

According to calculations (Table 3), a waiting period of 1 day is suggested for bifenthrin. Okra fruits were subjected to processing like washing and washing followed by boiling in order to investigate the reduction of residues.

Effect of processing

An experimental data (Table 4) shows that washing followed by boiling was found to be more effective than washing in reducing the residues. The residues were reduced up to 38.90% in single dose and 42.41% in double dose on 0 day by washing followed boiling/cooking. Thereafter, reduction of residues was 64.58 to 56.41% on 3rd day in case of single dose and double dose, respectively. Whereas no residues were found on 7 and 10th days in case of washing followed by boiling/cooking at single and double doses, respectively. However, by washing, residues were reduced in the range of 36.71 to 22.91% at lower and 40.00 to 21.05% at higher dose. This shows that with the passage of time residues penetrate into fruit and less reduction was observed on successive days.

The results was according to Chauhan et al. (2012) observations in which dissipation of bifenthrin residues in tomato was 0.107 and 0.234 mg kg^{-1} on 0 day after application and residues were reached below detectable level on 10 and 15th days after application. Kaur et al. (2011) reported that initial deposit of cypermethrin was 0.6 and 1.095 mg kg^{-1} in brinjal which declined to 0.1 and 0.189 mg kg^{-1}, 1 day after treatment in case of single and double dose, showing percent dissipation of 95.0 and 94.06 on 10th day at single and double dose. Similar types of observation have been reported by Singh and Kalra (1992). Present results are in agreement with earlier reports (Samriti, 2010) in which 39.49 to 41.17% reduction of cypermethrin residues on Okra was

observed, while in washing plus cooking reduction was from 71.64 to 78.87%. Dislodging of cypermethrin (0.001%) residues following cooking of brinjal in boiled water reduced by 41.40 to 36.4% from 0 to 3 days samples (Walia et al., 2010).

Conclusion

Thus, a comparison of the overall effects of different household processes indicated that the levels of bifenthrin residues can be reduced significantly by washing or by washing + boiling/cooking. The reduction in residue level makes these procedures worthwhile for adoption by the consumers. Residues of bifenthrin dissipated BDL of 0.005 mg kg^{-1} on 15 and 30^{th} day at single and double dose, respectively, hence applied doses were safe. Washing and washing followed by boiling/cooking was found effective in reducing the residues of bifenthrin and therefore these practices should be followed before consumption.

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