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RESEARCH ARTICLE

FATE OF BIFENTHRIN AND λ -CYHALOTHRIN IN WATER

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ABSTRACT

The distribution of synthetic pyrethroids in the surface waters was assessed by measuring the concentration in water samples. For this purpose water was collected from the nearby canal of agricultural field CCS HAU, Hisar. Water samples (200 mL) were treated with bifenthrin and λ -cyhalothrin at levels of 0.5mgL⁻¹ and 1.0 mgL⁻¹ separately. Liquid-liquid partitioning was used for the extraction of the pyrethroid from the water. Analysis of the pyrethroids extracts was done with gas chromatography equipped with electron capture detector. In case of bifenthrin residues persisted up to 90 days with half-life 10.38-12.04 while in λ -cyhalothrin the residues reached below detectable level (0.001 mg L⁻¹) within 60 days after treatment with half-life 7.16-7.52 days.

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INTRODUCTION

Human population explosion and technological advancement is a serious and insidious side effect for the contamination of the environment by chemicals. Pesticides are one of the major environmental pollutants and food trace contaminants. Extensive use of pesticides was very less up to 20th century, though chemicals were employed much earlier to control insects, fungi and weeds. In particular, pesticides are increasingly used in agricultural production. With the decline in the use of organochlorine pesticides in the late 1960s, organophosphorus compounds then carbamates and finally synthetic pyrethroids came in to existence. These compounds though short lived than organochlorines and do not accumulate, have resulted in increased mortality of fish and wildlife (Schmitt, 2005). Pesticides are pollutants that commonly occur in surface waters. They have been found in drinking water, underground water, troposphere, or even in water and ice of the Polar Regions (Kalwasin'ska et al., 2011). Pesticide contamination of surface waters and ground waters from agricultural use has been well documented around the world. Pesticide residues in surface waters have been a concern since the 1940s (Richard and Baker, 1993; Larson et al., 1991).

Also, industrial effluents carrying toxic pollutants are reported to be frequently dumped into rivers, if for drinking purpose water is drawn from such reservoirs or river banks can become dangerous and sometimes even up to fatal levels of contamination for human, animal, and plant life. Pyrethroids are the most common pesticides used in agriculture now a day. Bifenthrin, ((2-methyl -1,1-biphenyl-3-yl) -methyl-3 -(2-chloro-3,3,3- trifluoro-1-propenyl) -2,2-dimethylcyclopropanecarboxylate and λ -cyhalothrin, (S)- α -cyano-3- phenoxy benzyl- (Z)-(1R, 3R)-3-(2-chloro-3, 3, 3-trifluoro prop-1-enyl)-2, 2 dimethyl cyclopropane carboxylate, are the member of synthetic pyrethroid family. They have greater photo stability and insecticidal activity than earlier pyrethroids. Being non-polar in nature their solubility is less in water but have strong tendency to bind to soil (Linde, 1994). The continuous monitoring of pollutants in water is increasingly important for food safety to avoid serious health impact. A survey of the literature revealed that no information is available in regard to the fate of bifenthrin and λ -cyhalothrin in canal water. Hence, an attempt has been made to investigate the persistence and degradation kinetics of these two insecticides in canal water under laboratory conditions.

MATERIALS AND METHODS

Products and Reagents: Formulation of bifenthrin (Talstar 10 EC) and λ -cyhalothrin (Karate2.5EC) used for lab study were procured from local market.

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Solvents and reagents like dichloromethane, acetone, sodium chloride and anhydrous sodium sulfate were procured from Merck (Darmstadt, Germany). Before use, all the common solvents were redistilled in glass apparatus. By running reagent blanks, the suitability of all the solvents was ensured before actual analysis.

Water: The experiment was conducted in the Pesticide Residue laboratory, Department of Entomology, CCS Haryana Agriculture University (CCS HAU), Hisar at room temperature to study the persistence and degradation kinetics of bifenthrin and λ -cyhalothrin in canal water. For this purpose water samples were collected from the nearby canal of CCS HAU, Hisar. Canal water samples (200 mL) were treated with bifenthrin and λ -cyhalothrin at levels of 0.5 mg L⁻¹ (single dose) and 1.0 mg L⁻¹ (double dose) separately. Each treatment was replicated thrice. Treated canal water (200 ml) from both the treatments and insecticides was kept in brown color glass bottles including untreated control and bottles were stored under laboratory conditions. The physico-chemical characteristics of canal water were: EC $\times 10^{-6}$ (μsm^{-1}) 240; HCO₃⁻ (me⁻¹) 1.2; Ca⁺² (me⁻¹) 1.1; Ca²⁺ and Mg²⁺ (me⁻¹) 2.0 and pH 7.7.

Extraction

Extraction was performed as per method of Kumari *et al.* 2008. The treated water samples were processed by liquid-liquid partitioning for residues of bifenthrin and λ -cyhalothrin on 0 (1 h after treatment), 3, 7, 15, 30, 60 and 90 days after treatment (DAT). Water samples were taken in 1L separatory funnel, added 10 g sodium chloride and partitioned thrice (70, 50, 30 mL) with mixture of hexane : dichloromethane (85:15 v/v). Combined extract was concentrated to near dryness on a rotary vacuum flash evaporator after addition of one drop of mineral oil followed by gas manifold evaporator. The process was repeated thrice after rinsing with 5 mL hexane to eliminate the traces of dichloromethane. Final volume (2 mL) was made in n-hexane for analysis.

The residues of both the insecticides were quantified on Shimadzu 2010, a gas chromatograph (GC) equipped with fused capillary column, HP-1 of 30 m \times 0.32 mm i.d, 0.25 μm film thickness of polysiloxane (5 % diphenyl and 95 % dimethyl) and electron capture detector (ECD), Ni⁶³. The operating parameters of GC were: carrier gas flow: 60 mL min⁻¹, injector temperature 280°C, oven temperature programme was 150°C (5 min) increasing @ 8°C min up to 190°C (2 min.), further increased @ 15°C min⁻¹ up to 280°C (10 min.) with split ratio 1: 10. The retention times observed for bifenthrin was 18.480 min and for λ -cyhalothrin was 19.427 min. Water samples were spiked with bifenthrin and λ -cyhalothrin at two concentration levels (0.010 and 0.025 mg kg⁻¹) processed and analyzed as per the methodology described above to check the validity of the method. Percent recoveries in case of bifenthrin were observed to be 90.00 and 92.23 whereas in λ -cyhalothrin were 94.70 and 96.56 at these above said fortification levels, respectively. As the percent recovery obtained were more than 90%, therefore, the results have been presented as such without applying any correction factor. Limit of determination/quantification (LODe /LOQ) was 0.001 mg kg⁻¹.

RESULTS AND DISCUSSION

Residue data and percent dissipation of bifenthrin at single and double dose in water are given in Table 1 and Fig. 1. The average initial deposits in water were found to be 0.809 and 1.705 mg L⁻¹ on 0 (1h after application of insecticide) day at single and double dose, respectively. These residues dissipated to an extent of 0.757, 0.612, 0.484, 0.320, 0.144 and 1.610, 1.260, 0.950, 0.700, 0.275 mg L⁻¹ on 1, 3, 7, 15 and 30 days after application at single and double dose, respectively. The per cent dissipation observed on 60th day was 94.80 and 95.42 and thereafter 99.87 and 99.64 mg L⁻¹ on 90th day in single and double dose, respectively. Based on first order kinetics, half-life values for bifenthrin dissipation from canal water were calculated to be 11.31 and 11.75 days in single and double dose, respectively. In case of λ -cyhalothrin, the average initial deposits were found to be 0.765 and 1.562 mg L⁻¹ for single and double dose, respectively (Table 2 and Fig.2).

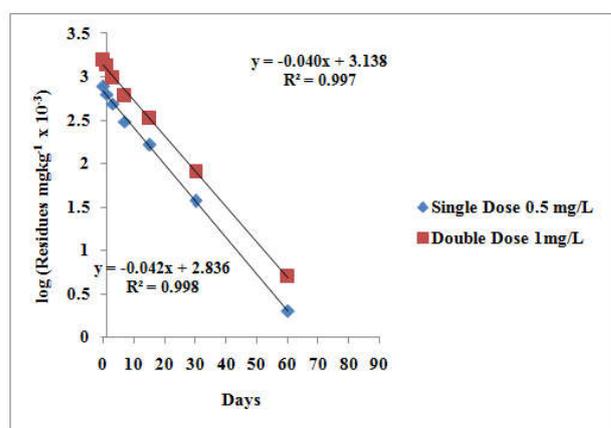


Fig.1. Dissipation kinetics of bifenthrin in water

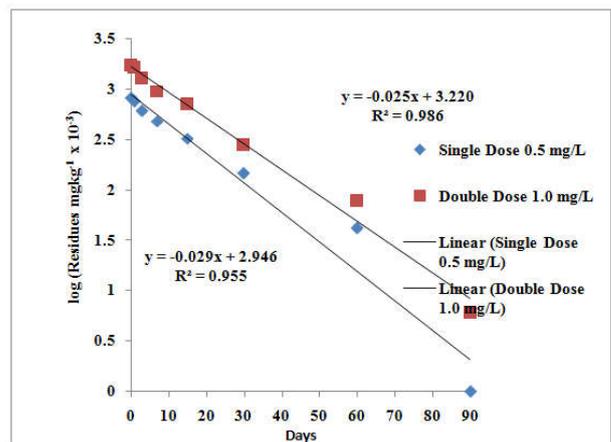


Fig. 2. Dissipation kinetics of λ -cyhalothrin in water

The residues continuously dissipated with the passage of time. The extent of dissipation was 0.630, 0.490, 0.305 and 0.166 in case of single dose on 1, 3, 7, 15 and 30 days after application while in case of double dose, the residues detected on these days were 1.390, 0.970, 0.605, 0.330 and 0.080 mg L⁻¹. Thereafter observed per cent dissipation was 99.74 and 99.68 on 60th day in single and double dose, respectively, and then residues reached below detectable level (BDL) of 0.001 mg L⁻¹ in both the cases.

Table 1. Dissipation of bifenthrin residues (mgL⁻¹)* in canal water at two doses

Days after treatment	Residue (mgL ⁻¹)			
	T ₁ (0.5 mgL ⁻¹)		T ₂ (1.0 mgL ⁻¹)	
	Average	% Dissipation	Average	% Dissipation
0	0.809±0.0081	-	1.705 ±0.0236	-
1	0.757 ±0.0015	6.42	1.610 ±0.0127	5.57
3	0.612 ±0.0134	24.35	1.260 ±0.0255	26.09
7	0.484 ±0.0243	40.17	0.950 ±0.0322	44.28
15	0.320 ±0.0166	60.44	0.700 ±0.016	58.94
30	0.144±0.0029	82.20	0.275 ±0.010	83.87
60	0.042 ±0.0008	94.80	0.078 ±0.0066	95.42
90	0.001 ±0.0008	99.87	0.006 ±0.0014	99.64
$t_{1/2} = 10.31d$			$t_{1/2} = 11.75 d$	

*BDL: 0.001 mgL⁻¹**Table 2. Dissipation of λ-cyhalothrin residues (mg L⁻¹)* in canal water at two doses**

Days after treatment	Residue (mgL ⁻¹)			
	T ₁ (0.5 mgL ⁻¹)		T ₂ (1.0 mgL ⁻¹)	
	Average	% Dissipation	Average	% Dissipation
0	0.765	-	1.562	-
1	0.630	17.65	1.390	11.01
3	0.490	35.94	0.970	37.90
7	0.305	60.13	0.605	61.27
15	0.166	78.31	0.330	78.87
30	0.037	94.29	0.080	94.387
60	0.002	99.74	0.005	99.68
90	BDL	-	BDL	-
$t_{1/2} = 7.16 d$			$t_{1/2} = 7.52 d$	

*BDL: 0.001 mgL⁻¹

Residue data were subjected to statistical analysis for computation of regression equations, half-life ($t_{1/2}$) values and per cent degradation. The residues dissipated with half-life 7.16 days at single dose and 7.52 days at double dose following first order kinetics. Gupta and Gajbhiye, 2008 studied the dissipation behaviour of bifenthrin at different pH and revealed that bifenthrin was most stable under alkaline pH followed by neutral and acidic pH. Half-life values at different pH varied from 14.8-42.4 days. The residues of bifenthrin dissipated fastest at 40°C (T₁, 27.4 days) followed by 25°C (T₁, 32.4 days) and 5°C (T₁, 44.3 days). Dissipation in all cases followed first order kinetics. Biswas *et al.*, 2009 studied the persistence of napropamide in water at three different pH (4.0, 7.0 and 9.2) with two doses i.e. 0.08 µg mL⁻¹ (T₁) and 0.16 µg mL⁻¹ (T₂) under laboratory condition following first order kinetics in all doses.

The persistence of napropamide was higher in acidic solution followed by neutral solution and alkaline solution. Degradation behaviour of propiconazole in canal water was investigated by Chauhan *et al.*, 2008 under laboratory conditions at two doses, 0.0625 and 0.125 µg mL⁻¹. More than 80% dissipation of propiconazole residues in water has been observed in both the treatments in 90 days after application showing half life (DT₅₀) values of 36 to 38 days. Reddy and Reddy, (2010) investigated the levels of pesticides residues contamination in drinking water bodies of Hyderabad and surrounding sub-urban areas. Total of 4 lakes was selected for surface water sample collection. Samples were collected four times from each location i.e. during post-monsoon 2005, pre-monsoon 2006, post-monsoon 2006 and pre-monsoon 2007. None of the pyrethroids was detected in any samples.

The organochlorine pesticides residues were detected in most of the samples. Chlorpyrifos, malathion, ethion residues were detected in the reservoirs where the catchment area is with intensive agriculture. Most of the samples recorded residues above MRL of 0.5 ppb prescribed by Bureau of Indian Standards 2004. Lee *et al.*, 2002 monitored the synthetic pyrethroids (bifenthrin, permethrin, and deltamethrin) in surface water at the sub-parts per billion level and reported that the concentration of all compounds quickly decreased and then remained constant at 58-72% of the initial concentration, likely due to adsorption to the glass surface and nearly 100% extraction recoveries were obtained when C (8) or C (18) membranes were used for extracting runoff samples. Adsorption to glass surfaces and suspended solids can result in substantial underestimation of pyrethroid concentrations or biological activity and therefore should be considered when designing monitoring protocols.

The distribution of pyrethroid insecticides in the Ebro River Delta (NE Spain) was studied by Feo *et al.*, 2010, in surface water and sediments. Cypermethrin was detected in 22 water samples collected from Ebro River Delta, while deltamethrin was present only in three water samples at concentrations ranging from 0.73 ng L⁻¹ to 57.2 ng L⁻¹ and 2 ng L⁻¹ to 58.8 ng L⁻¹ for cypermethrin and deltamethrin, respectively. In case of deltamethrin the concentration levels were found higher than median lethal concentration (LC50). In sediment samples only cypermethrin was detected at concentration levels ranged from 8.27 ng g⁻¹ to 71.9 ng g⁻¹. These levels were higher than its LC50 values. Afful *et al.*, 2013 studied the levels of synthetic pyrethroids in the Weija Lake water and sediments and detected the concentration of pyrethroids ranged from

0.10-3.50 ngL⁻¹ and 0.15-6.60 ngg⁻¹ for the water and sediment samples respectively. The concentrations of detected pyrethroids in water were far below maximum residue limits set by European Union and Japanese Government and therefore suggested safe for health.

Conclusion

This work clearly shows that bifenthrin persists for longer period in the water as compared to λ -cyhalothrin which reached below detectable level (BDL) of 0.001 mg L⁻¹ within 60 days after treatment due to its low solubility in water (0.005 mg/L). Depending upon the very low solubility of both the insecticides in water, their use in agriculture can be considered safe.

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