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Dissipation of Bifenthrin in Medium Black Calcareous Soil under Laboratory conditions

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ABSTRACT: An experiment was conducted during 2017-18 at AINP on Pesticide Residues, ICAR Unit- 9, AAU, Anand, Gujarat to study the dissipation behavior of bifenthrin in medium black calcareous soil under laboratory conditions. The bifenthrin was spiked in soil @ 0.06 and 0.12 µg g^{-1}. The experiment was conducted with three replications along with a control. The samples were collected on 0 (one hour after treatment), 1, 3, 5, 7, 10, 20, 30, 40, 50 and 60 days after treatment. Residues of bifenthrin were estimated by validated QuEChERS multi residue method using GLC equipped with electron capture detector. The mean recovery was in the range of 82.00- 96.67 percent. The residues of bifenthrin in medium black calcareous could be detected up to 40 and 50 days after application at single and double dose, respectively. Higher dose revealed longer persistence in soil than lower one. Bifenthrin showed longer persistent in medium black calcareous soil due to strong adsorption and the presence of soil clay and organic matter, the persistence of bifenthrin in soil increases. Degradation of bifenthrin in soil samples followed first order kinetics.

Key words: Bifenthrin, Biphasic dissipation, QuEChERS, medium black calcareous soil.

INTRODUCTION

Pyrethroid pesticides are widely employed in both farm and urban areas. Photo stable pyrethroids once accounted for over 25% of the global foliar pesticide market and had a high toxicity to benthic aquatic species. They replaced some of the more toxic and ecologically persistent organochlorine compounds because of their great insecticidal action paired with low mammalian toxicity. Pyrethroids, on the other hand, are among the most effective insecticides now available. Natural pyrethrins have great insecticidal effects and minimal mammalian toxicity, but their low photo stability and high biodegradability limit their application. The widespread usage of pyrethroids began in the 1970s, following their development. Over the last several decades, several synthetic pyrethroids have been structurally generated from pyrethrins. Pyrethrins' basic component, a chrysanthemic acid linked to an aromatic alcohol via an ester linkage, has been preserved. Although low mammalian toxicity makes pyrethroid pesticides harmless, they cause nervous system damage in vertebrates through a variety of mechanisms (Miller and Salgado, 1985). Interference with sodium channel gating in nerve cell terminals is one of the most important (Lund and Narahashi, 1981). Pyrethroid insecticides effectively paralyse organisms by severely restricting neuro-transmission by acting on sodium channels to depolarize presynaptic terminals (Salgado *et al*., 1983). Due to the hyperactivity of nerve terminals, this paralysis is frequently preceded by spastic activity of the organism. The spastic activity is induced by sodium channels polarising and depolarizing frequently, simulating neurotransmission where none exists. They are the most widely used pesticide class today. In India, two novel synthetic pyrethroids, bifenthrin and - cyhalothrin, have been approved for use in plant protection methods; nevertheless, public health concerns must be considered for residual levels, which must not exceed the maximum allowable intake to maintain food safety. Bifenthrin is a third-generation pyrethroid (2-methyl-3-phenylbenzyl (1RS) -cis - 3 - (2- chloro - 3, 3, 3 trifluoroprop-1-enyl) -2,2-dimthylcyclo-propanecarboxylate). It's an insecticide and acaricide that affects insects' nerve systems and causes them to become paralysed. Bifenthrin is used in agriculture to manage sucking and biting insects such as aphids, white fly, and Colorado beetle in a variety of crops, as well as in public health applications (mostly as emulsifiable concentrates or wettable powder) to control mosquitoes, houseflies, and cockroaches (Lee *et al.*, 2004).

Although soil texture has an impact on termiticide performance, the effects vary depending on the termiticide. According to Wiltz (2012), termite mortality is highly related to soil clay concentration, regardless of termiticides used, treatment rates, or exposure times. C. formosanus mortality was highest when clay content was low in assays using bifenthrin, fipronil, and chlorfenapyr (Wiltz, 2012). The mortality of C. formosanus workers was higher in the fipronil-treated sand than in the treated soil and clay mixture, according to Osbrink and Lax (2002). The amount of clay and organic matter in the soil has a big impact on sorption. Fipronil's adsorption coefficient drops significantly as soil clay concentration decreases, resulting in increased bioavailability (Bobé *et al.*, 1997; Cox *et al*., 1998; 2001).

Because bifenthrin is heavily adsorbed on soil particles, its bioavailability is expected to be low. It degrades with time, with half-lives ranging from 122 to 345 days. In the presence of sediment, S. acidaminiphila's breakdown of bifenthrin was greatly hindered, and this result was likely produced by high adsorption to the solid phase (Lee *et al.*, 2004). Bifenthrin (10 percent EC) is approved for cotton, sugarcane, tea, and apple in India, while 2.5 percent EC is approved for termite and wood borer control. Bifenthrin products are classified as "limited use pesticides" due to their significant toxicity to aquatic organisms. In addition to polluting the environment, indiscriminate use of such pesticides could pose a major health danger to consumers. Pesticide residues in fruits and vegetables have also been documented in monitoring studies conducted around the world. Residue analysis determines the kind and extent of any chemical pollution in the environment, as well as its persistence.

MATERIAL AND METHODS

A. Soil

A laboratory experiment was conducted at All India Network Project on Pesticide Residues, Indian Council of Agricultural Research (ICAR) Unit- 9, Anand Agricultural University (AAU), Anand to study the dissipation behavior of bifenthrin in medium black calcareous soil collected from Junagadh Agricultural University (JAU) Junagadh, Gujarat, India, were used in this experiment. Further soil was spiked and used for residue analysis of bifenthrin.

B. Chemicals and reagents

All the analytical grade reagent and chemicals were provided by Merck Life Science Private Ltd. Certified reference material (CRM) were procured from Sigma Aldrich India Limited. The purified bifenthrin is melting at 68-70°C, with a fatty odour under normal temperature and pressure conditions. It decomposes before boiling at 285°C. Its relative density is 1.316 g cm^{-3}. It is almost insoluble (< 1 µg/L) in water at 20°C at all tested pH's (Meena *et al.*, 2021). Bifenthrin is not flammable, and has a flash point higher than 110°C. It does not present explosive or oxidising properties. It is stable in storage conditions for up to 2 years.

C. Extraction procedure: (QuEChERS method)

A representative 10 g soil was transferred to a 50 mL polypropylene centrifuge tube and 20 mL acetonitrile followed by mixture of 1 g NaCl + 4 g $MgSO_4$, shaken vigorously by hand (1 min.) before centrifugation. The tubes were centrifuged at 3500 rpm for 3 minutes. A 10 mL aliquot from the supernatant was transferred by auto pipette into a 15 mL polypropylene centrifuge tube containing 1.5 g $MgSO_4$ and 0.25 g PSA (Primary Secondary Amine), followed by centrifugation at 2500 rpm for 2 minutes. A 4 mL aliquot was transferred into glass test tube and acetonitrile was completely evaporated on Turbo Vap® LV. Final volume (2 mL) was made by petroleum spirit: acetone (1:1 v/v) and residues were quantified on GC-ECD.

D. Recovery and limit of quantification

Before starting the experiment, method performance studies were carried out to check the efficiency for determining bifenthrin in soil sample as per SANTE guideline[16]. The calibration curve was determined by considering matrix-matched standards of bifenthrin at five different concentrations (0.01 – 1.0 µg mL^{-1}). The matrix standards were prepared by collecting matrix of control sample. The limit of detection (LOD) was defined as the analyte concentration that produced a signal-to-noise (S/N) ratio of 3 and the limit of quantification (LOQ) was related to an S/N ratio of 10. Soil was spiked at 0.05, 0.25 and 0.5 µg g^{-1} levels in five replicates to evaluate the percentage recovery. The spiked samples were extracted and analyzed.

RESULTS AND DISCUSSION

A. Linearity and recovery

The calibration curve of matrix-matched standards of bifenthrin demonstrated linear response in the range of 0.01- 1.0 ppm with a correlation coefficient, R2 >0.99 as per the SANTE guideline. The LOQ was 0.01 µg g^{-1}. The mean recovery of bifenthrin in soil was in the range of 82.00- 96.67 percent at three spiking levels, *i.e.* 0.05, 0.25 and 0.5 µg g^{-1}. The per cent relative standard deviation within the replicates (RSD) obtained for respective soils at different spiking levels was in the range of 6.93-15.91 percent. As per SANTE guidelines, recoveries (Table 1) were above 85 per cent and RSDWR below 20% is accurate and precise.

Table 1: Recovery of bifenthrin in medium black calcareous soil.

Parameters	Fortification level (µg/g)		
	0.05	0.25	0.50
Mean Recovery (%) *n** = 5	82.00	90.72	96.67
Standard Deviation (±SD)	13.04	6.29	9.41
% RSD	15.91	6.93	9.74

*=No. of replication

B. Dissipation and residue status

The bifenthrin was spiked in medium black calcareous soil @ 0.06 and 0.012 ppm. The experiment was conducted with three replications along with a control. The samples were collected on 0 (one hour after treatment), 1, 3, 5, 7, 10, 20, 30, 40, 50 and 60 days after treatment. Residue of bifenthrin in soil was estimated by validated multi residue method QuEChERS using Gas Liquid Chromatography equipped with electron capture detector. The data obtained from the study were subjected to regression analysis to determine the dissipation pattern and half-life by adopting first order degradation kinetics. The dissipation of bifenthrin in medium black calcareous soil was biphasic in nature at either dose (Fig. 1).

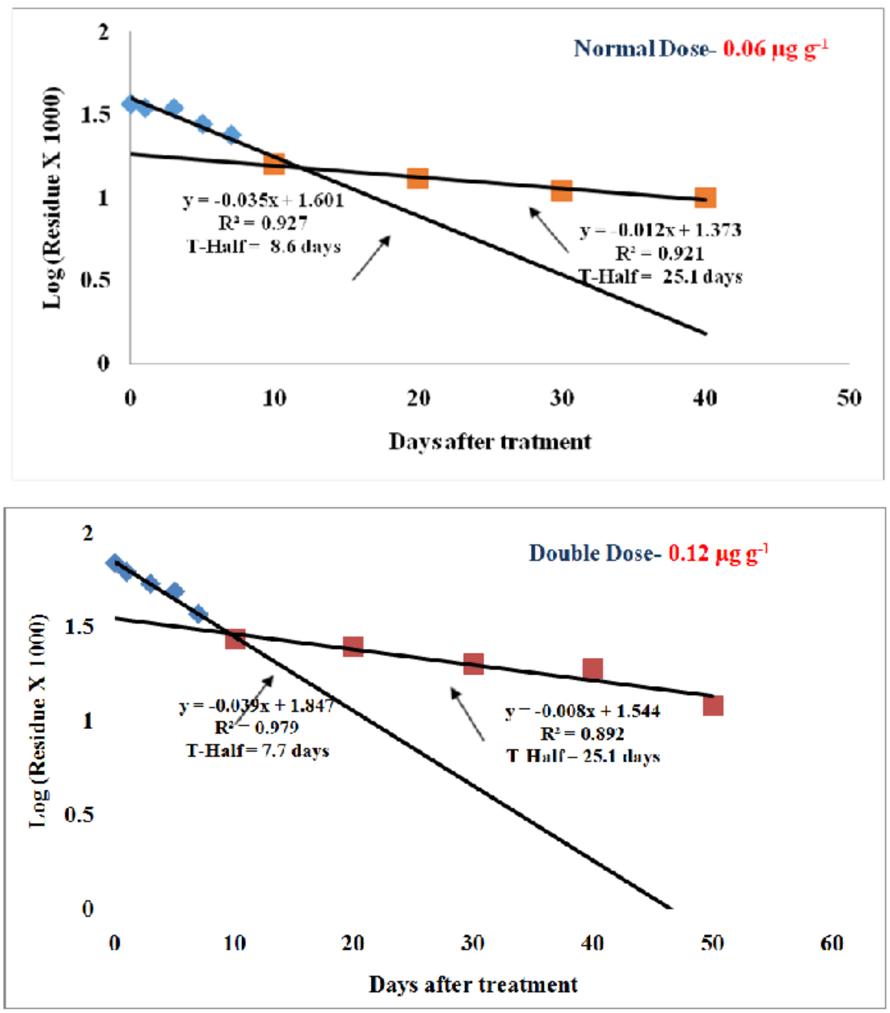

Fig. 1. Dissipation pattern of bifenthrin at normal and double dose in medium black calcareous soil.

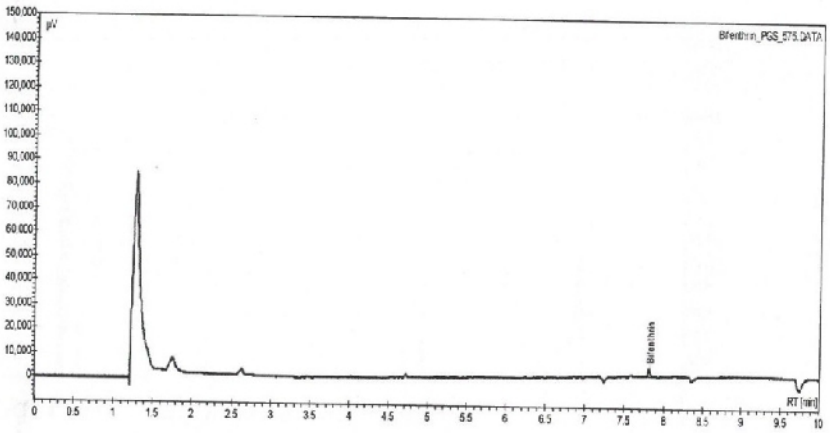

Fig. 2. The chromatogram of bifenthrin in medium black calcareous soil at 0.06 µg g^{-1}.

On the 0 day (i.e. one hour after treatment) the levels were 0.037 and 0.069 µg g^{-1} which declined to 0.035 and 0.062 µg g^{-1} within 24 hours for the corresponding dose rendering 5.40 and 10.14 per cent loss. At normal dose, half-life values were 8.6 and 25.1 days during phase-I (0-10 days) and phase-II (10-40 days), respectively. At double dose, during phase- I (0-10 days) and phase- II (10-50 days) half- life values were 7.7 and 25.1 days, respectively are presented in Table 2. The gas chromatogram of bifenthrin in medium black calcareous soil is depicted in Fig. 2.

Table 2: Residue and dissipation pattern of bifenthrin in medium black calcareous soil.

Days after treatment	Residue (µg g^{-1})				
	Dose				
	T_1 (Control)	T_2 (0.06 µg g^{-1})		T_3 (0.12 µg g^{-1})	
		Mean (±SD) (*n* = 3)	% loss over initial	Mean (±SD) (*n* = 3)	% loss over initial
0	ND	0.037 (±0.001)	-	0.069 (±0.003)	-
1	ND	0.035 (±0.002)	5.40	0.062 (±0.001)	10.14
3	ND	0.035 (±0.002)	5.40	0.054 (±0.007)	21.73
5	ND	0.028 (±0.001)	24.32	0.049 (±0.002)	28.99
7	ND	0.024 (±0.003)	35.13	0.037 (±0.003)	46.38
10	ND	0.016 (±0.001)	56.76	0.027 (±0.003)	60.87
20	ND	0.013 (±0.001)	64.87	0.025 (±0.000)	63.77
30	ND	0.011 (±0.000)	70.27	0.020 (±0.012)	71.01
40	ND	0.010 (±0.001)	72.97	0.019 (±0.001)	72.46
50	ND	<LOQ	-	0.012 (±0.001)	82.60
60	ND	<LOQ	-	<LOQ	-
Regression equation R^2	**Phase- I**	Y = -0.035X+ 1.601 R^2 = 0.927 (0- 10 days) T-Half = 8.6 days		Y = -0.039X + 1.847 R^2 = 0.979 (0-10 days) T-Half = 7.7 days	
	Phase- II	Y = -0.012X + 1.373 R^2 = 0.921 (10- 40 days) T-Half = 25.1 days		Y= -0.008X + 1.544 R^2 = 0.892 (10- 50 days) T-Half = 25.1 days	

LOQ- Limit of Quantification = 0.01 µg g^{-1}

From the results of the study, it was found that bifenthrin residues persisted for more than 40 and 50 days at normal dose and double dose in medium black calcareous soil, respectively. Higher persistence of bifenthrin in heavy black soil due to strong adsorption. It's because there's a lot of organic debris and clay in there, which could bind the bifenthrin. Higher soil clay concentration suggests fewer big holes, a larger specific surface area per unit volume of soil, and a higher adsorption affinity of soils for pesticides, resulting in less pesticide mobility (Petrovic and Larsson-Kovach, 1996).

Similar findings were seen with pyraclonil, which had a stronger adsorption affinity for clay minerals and poor soil mobility (Zhang *et al.*, 2020; Ou *et al*., 2020).

In comparison to fipronil and imidacloprid, bifenthrin had a higher concentration in the top section of the soil column, indicating that it is the most persistent of the termiticides studied (Rashid *et al.,* 2018). Horwood (2007) observed that bifenthrin was greater in concentration than chlorfenapyr, chlorpyrifos, fipronil, and imidacloprid.

Because bifenthrin has a higher Koc value, it has a greater capacity to persist in soil particles, resulting in low leaching potential, according to Kamble and Saran (2005).

Meyer *et al.* (2013) discovered that bifenthrin connected to soils and sediment due to its excellent soil particle binding capabilities. Organic matter is a crucial component in the absorption and leaching process (Bajeer 2012). Therefore, the higher amount of cation exchange capacity and organic matter in soil could increase the adsorption of termiticide, and simultaneously reduce the leaching probability. Similar results were reported by Bhuva *et al.,* (2015) that bifenthrin residues in clay, sandy loam, and sandy soils were found up to 30 days and half-life was 21.2 days. The sterile and non-sterile soils had half-life values of 330 and 147 days, respectively. The large variation in half-life values between the two soils indicated that microbial degradation had a significant role in bifenthrin dissipation (Sharma and Singh, 2012). The study revealed pesticide applied at higher dose showed erratic pattern and longer persistence in soils as compared to the lower dose.

CONCLUSIONS

The results of the present investigation demonstrated that bifenthrin persists for more than 40 and 50 days at normal and double dose in medium black calcareous soil, respectively. Higher dose revealed longer persistence in soils than lower one. Because of strong adsorption and the presence of soil clay and organic matter, the persistence of bifenthrin in soil increases. Further studies could be conducted under different field and climatic conditions.

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Conflict of Interest. None.

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