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Dissipation Kinetics of Chlorfluazuron in Soils of Four Different Agro-Climatic Regions under Laboratory Simulated Condition

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Abstract—*Laboratory simulation studies were performed to investigate dissipation kinetics of chlorfluazuron 5.4% EC in soils of four different agro-climatic regions viz. new alluvial soil from Mohanpur, red & lateritic soil from Jhargram, coastal saline soil from Canning and black soil from Pune at the rates of 1.0 and 2.0 µg ml^{-1}. Soil samples collected on 0 (2h after application), 3, 7, 15, 30, 60 and 90 days after application of chlorfluazuron were processed for residue analysis by HPLC. Chlorfluazuron was dissipated linearly with progress of time irrespective of dose and substrate. The calculated half-life values for black soil (pH 8.14) was found to be in the range between 12.04-14.90 days whereas for saline soil (pH 7.92) it was 15.44-18.47 days, for red & lateritic soil (pH 5.56) it was 23.34-25.51 days and for new alluvial soil (pH 6.85) it was 19.17-21.35 days irrespective of treatment doses.*

1. INTRODUCTION

Chlorfluazuron insecticide, belongs to benzophenyl urea group (see Fig. 1), can work as potent chitin synthesis inhibitor and controls various lepidopteran larvae including diamond back moth (DBM). It has very little or no negative impact on natural enemies and pollinators insects and can be used in integrated pest management programmes. Chlorfluazuron possesses ovicidal activity against adult German cockroach (Blattella germanica), and the common cutworm (Spodoptera litura) [1-2] and has also been evaluated for uses in non-crop applications such as fly control [3] and for use as part of a termite bait matrix [4-5] which may extend the use of this mature compound.

Besides its potent insecticidal property, chlorfluazuron may possess toxicity risk to the non-target organism especially human being. It is very essential to estimate the extent of chlorfluazuron residues that can be found in various substrates like soil, water etc. Thus, the present experiment has been designed to find out the persistence of chlorfluazuron in soils of four different agro-climatic zones viz. new alluvial soil from Mohanpur, red & lateritic soil from Jhargram, coastal saline soil from Canning and black soil from Pune under laboratory simulated condition. The results could be utilized for further research in determining the fate of this insecticide in Indian soils in field conditions.

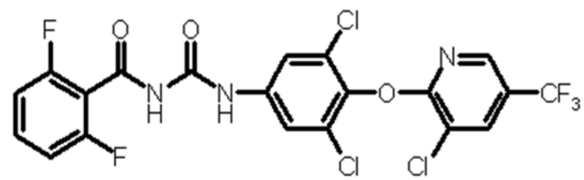

Fig. 1: Structure of chlorfluazuron

2. MATERIALS AND METHOD

Experimental details

The present study has been designed to find out the dissipation kinetics of chlorfluazuron in soils of four different agro-climatic regions *viz.* new alluvial soil from Mohanpur, red & lateritic soil from Jhargram, coastal saline soil from Canning and black soil from Pune under laboratory simulated condition (physico-chemical properties of these soils are given in the table 1). Chlorfluazuron analytical standard (10 mg) as well as chlorfluazuron 5.4% EC formulation (~185.19 mg), supplied by M/S United Phosphorus Ltd., were separately taken in two 100 ml volumetric flasks. The volumes were made up to the mark with HPLC grade methanol to prepare stock standard of 100 µg ml^{-1} of each formulation & technical grade. Necessary dilutions were made from these standards as and when required.

Soil samples (50 g) were kept in 250 ml conical flasks to form a set for each type of soil and 10 ml of water was added to it. Then 1 ml and 2 ml of the 50 µg ml^{-1} stock solution of chlorfluazuron 5.4% EC were added to conical flasks separately containing 50 g of different soil samples. The initial concentrations became 1 µg g^{-1} (T_1) and 2 µg g^{-1} (T_2) respectively. The untreated control soils (50 g) were added with 10 ml of water only. Three replicate flasks for each treatment were taken for analysis on each day of sampling along with untreated control. Samples (three replicates) were processed for analysis of chlorfluazuron residues at intervals of 0 (2h after application), 3, 7, 15, 30, 60 and 90 days after application.

Table 1: Physico-chemical properties of different soils

Location	Texture	pH	Bulk Density (g/cm^3)	Organic Carbon (%)
New alluvial soil	Sandy loam	6.85	1.28	0.76
Red & lateritic soil	Sandy	5.56	1.58	0.52
Coastal saline soil	Silty loam	7.92	1.45	1.21
Black Soil	Clayey	8.14	1.60	0.67

Extraction and clean up

Soil samples in the respective sampling dates were added with 100 ml mixture of acetone and water (8:2), kept for overnight and were shaken for a period of 30 minutes using a mechanical shaker (25˚C). It was then filtered and the extract was collected and re-extracted the sample using 100 ml mixture of acetone and water (8:2). Combined filtrate was transferred to a 500 ml separatory funnel. Then 100 mL of distilled water was added to it. This mixture was partitioned thrice with 200 (100+50+50) ml ethyl acetate. Ethyl acetate fraction was collected through anhydrous Na2SO4. This combined fraction was concentrated to 1-2 ml in rotary vacuum evaporator at 40˚C.

A chromatographic column was packed up with 10 g mixture of silica gel and florisil (1:1). Anhydrous sodium sulphate was placed in bottom & top of the column using hexane. The residue was transferred in to the column. Elution was done with 100 ml hexane followed by 100 ml of methanol. Methanol fraction was evaporated to dryness in a rotary vacuum evaporator at 40˚C and the volume was reconstituted in HPLC grade methanol for HPLC analysis.

HPLC – operating parameters

Peerless Basic C18 column (250 mm X 4.6 mm) was used for chromatographic separation with methanol: water (95:5) as the mobile phase at a flow rate of 1.0 ml min-1. Under these working conditions chlorfluazuron was detected (at λmax = 255 nm) at the retention time of 5.9 ± 0.20 min (see Fig. 2). The LOD and LOQ of the method were determined as 0.05 and 0.10 µg g^{-1}, respectively (see Fig. 3).

Recovery study

Recovery studies were carried out in order to establish the reliability of the analytical methods and to know the efficiency of extraction and clean up step for the present study by fortifying different soils separately with chlorfluazuron 5.4% EC at 0.10, 0.50 and 1.00 µg g^{-1} level.

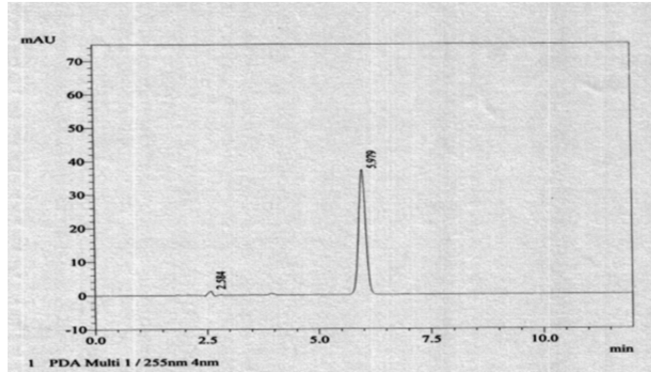

Fig. 2. Chromatogram of chlorfluazuron analytical standard under HPLC operating conditions

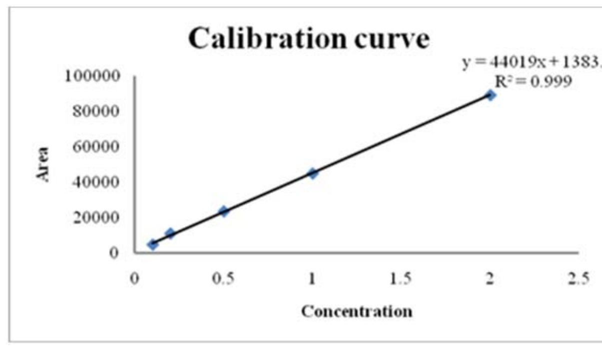

Fig. 3. Calibration curve of areas corresponding to different concentrations of analytical standard of chlorfluazuron

3. RESULTS & DISCUSSION

Results of recovery study

Average recoveries of chlorfluazuron in soils fortified at 0.10, 0.50 and 1.00 µg ml-1 were 88.67% for new alluvial soils, 86.33% for red & lateritic soils, 85.67% for coastal saline soils and 89.67% for black soils (see table 2). Hence the analytical method was quite satisfactory and adopted for the present study.

Table 2: Recovery study of chlorfluazuron 5.4% EC in different soil samples

Fortification level (µg mL^{-1})	Recovery (%)*			
	New alluvial soil	Red & lateritic soil	Coastal saline soil	Black Soil
0.10	85.00	82.00	82.00	87.00
0.50	86.00	90.00	86.00	92.00
1.00	89.00	87.00	89.00	90.00
Average recovery (%)	86.67	86.33	85.67	89.67
Recovery factor	1.15	1.16	1.17	1.12

*Mean of three replications

Persistence in soil

The dissipation pattern of chlorfluazuron was evaluated in four different types of soil viz. new alluvial soil (pH 6.85) from Mohanpur, red & lateritic soil (pH 5.56) from Jhargram, coastal saline soil (pH 7.92) from Canning and black soil (pH 8.14) from Pune under laboratory simulated condition.

The mean residue values, percent dissipation, regression equation and half-life values of chlorfluazuron in four different types of soils are presented in table 3. From the table it was observed that chlorfluazuron dissipates linearly with progress of time i.e. the chlorfluazuron dissipation follows a first order kinetics. Residues were below detectable limit (BDL) of 0.10 µg g^{-1} in all untreated control samples (T_3) throughout the study.

In new alluvial soil (pH 6.85), the initial residues and half-life value of chlorfluazuron were ranged between 0.97-1.98 µg g^{-1} and 19.17-21.35 days irrespective of T_1 (1.0 µg g^{-1} of soil) and T_2 (2.0 µg g^{-1} of soil). More than 60% of initial residues were dissipated within 30 days in both the cases. Chlorfluazuron residues were found below detectable limit (BDL) on 90th day after application irrespective of treatments.

In case of red & lateritic soil (pH 5.56), the initial residues and half-life value of chlorfluazuron were ranged between 0.97-1.96 µg g^{-1} and 23.34-25.51 days irrespective of T_1 (1.0 µg g^{-1} of soil) and T_2 (2.0 µg g^{-1} of soil). Around 50-56% of initial residues were dissipated within 30 days in both the cases for T_1 & T_2. In case of T_1, chlorfluazuron residues were found below detectable limit (BDL) on 90th day after application.

In coastal saline soil (pH 7.92), the initial residues and half-life value of chlorfluazuron were ranged between 0.91-1.94 µg g^{-1} and 15.44-18.47 days irrespective of T_1 (1.0 µg g^{-1} of soil) and T_2 (2.0 µg g^{-1} of soil). The initial residues dissipated to around 72% in case of T_1 and 65% in case of T_2 within 30 days after application. The residues were below detectable limit (BDL) on 60th day and 90th day after application for T_1 & T_2 respectively.

In case of black soil (pH 8.14), the initial residues and half-life value of chlorfluazuron were ranged between 0.93-1.94 µg g^{-1} and 12.04-14.90 days irrespective of T_1 (1.0 µg g^{-1} of soil) and T_2 (2.0 µg g^{-1} of soil). Around 75-80% of initial residues were dissipated within 30 days in both the cases for T_1 & T_2. The residues were below detectable limit (BDL) on 60th day and 90th day after application for T_1 & T_2 respectively.

4. CONCLUSION

It was revealed from the results that the initial concentration of chlorfluazuron was in the range between 0.91-0.97 µg g^{-1} and 1.94-1.98 µg g^{-1} respectively for T_1 (1.0 µg g^{-1} of soil) and T_2 (2.0 µg g^{-1} of soil) and irrespective of soil types (table 3). The dissipation pattern of this particular compound in different soils can be understood in terms of calculated half-life (table 3). Present study shows that the calculated half-life values for black soil (pH 8.14) was found to be in the range between 12.04-14.90 days whereas for saline soil (pH 7.92) it was 15.44-18.47 days, for red & lateritic soil (pH 5.56) it was 23.34-25.51 days and for new alluvial soil (pH 6.85) it was 19.17-21.35 days irrespective of treatment doses.

It was found from the results that chlorfluazuron dissipates somewhat rapidly in black soil than in coastal saline, new alluvial and red & lateritic soil. After 90 days of incubation, chlorfluazuron residues were found below detectable limit for T_1 and below detectable limit to 0.17 µg g^{-1} for T_2 irrespective of soil types. Thus, it may be concluded that chlorfluazuron with particular formulation dissipates rapidly in soils under laboratory simulation study. The half-life values of different soils were in the range between 12-26 days.

5. ACKNOWLEDGEMENT

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Table 3: Dissipation of chlorfluazuron in different soils at different day's interval

Days	Residues (µg ml^{-1})							
	New Alluvial soil (pH 6.85)		Red & Lateritic soil (pH 5.56)		Coastal Saline soil (pH 7.92)		Black soil (pH 8.14)	
	1 µg ml^{-1}	2 µg ml^{-1}	1 µg ml^{-1}	2 µg ml^{-1}	1 µg ml^{-1}	2 µg ml^{-1}	1 µg ml^{-1}	2 µg ml^{-1}
0	0.97±0.04 (-)	1.98±0.04 (-)	0.97±0.02 (-)	1.96±0.05 (-)	0.91±0.02 (-)	1.94±0.04 (-)	0.93±0.03 (-)	1.94±0.02 (-)
3	0.90±0.04 (6.87)	1.84±0.03 (6.90)	0.91±0.03 (6.19)	1.87±0.03 (4.57)	0.85±0.03 (6.23)	1.80±0.04 (7.04)	0.86±0.02 (7.89)	1.79±0.03 (7.73)
7	0.78±0.02 (19.59)	1.64±0.03 (17.17)	0.82±0.02 (15.81)	1.71±0.02 (12.86)	0.80±0.03 (12.45)	1.59±0.02 (17.87)	0.72±0.01 (22.94)	1.45±0.03 (25.26)
15	0.64±0.03 (33.68)	1.24±0.04 (37.21)	0.74±0.02 (24.05)	1.41±0.03 (28.23)	0.50±0.03 (44.69)	1.14±0.04 (41.07)	0.40±0.02 (56.63)	0.98±0.02 (49.66)
30	0.36±0.02 (63.23)	0.76±0.02 (61.78)	0.42±0.01 (56.36)	0.91±0.03 (53.74)	0.25±0.02 (72.53)	0.69±0.02 (64.43)	0.18±0.02 (81.00)	0.49±0.02 (74.91)
60	0.11±0.01 (88.32)	0.29±0.03 (85.52)	0.17±0.02 (82.82)	0.40±0.03 (79.59)	BDL	0.21±0.03 (89.35)	BDL	0.12±0.02 (93.64)
90	BDL	BDL	BDL	0.17±0.02 (91.16)	BDL	BDL	BDL	BDL
Regression Equation	Y=3.0089-0.0157X	Y=3.3058-0.0141X	Y=3.0101-0.0129X	Y=3.3101-0.0118X	Y=2.9927-0.0195X	Y=3.3054-0.0163X	Y=2.9969-0.0250X	Y=3.2981-0.0202X

Half-life ($T_{1/2}$) (days)	19.17	21.35	23.34	25.51	15.44	18.47	12.04	14.9

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