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Persistence and dissipation of chlorantraniliprole residues in cabbage (*Brassica oleracea* L.) and soil

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Abstract

A field experiment was conducted at main agricultural research station, Dharwad during *rabi* 2015-16 to study the persistence of chlorantraniliprole residues in cabbage (*Brassica oleracea* L.) and soil, following four application of chlorantraniliprole at 0.20 ml L⁻¹ (single dose) and 0.40 ml L⁻¹ (double dose) g a. i. ha⁻¹. Analysis of cabbage samples using UPLC revealed that the chlorantraniliprole residue persisted up to two days in both single and double dose after the last spray. The initial deposits of chlorantraniliprole residues in cabbage heads on 0 day (2hrs after last spray) were 0.81 mg kg⁻¹ in single dosage and 2.00 mg kg⁻¹ in double dosage of the chemical and reached below detectable limit by 3rd day, showing 100 per cent dissipation of chlorantraniliprole at both the dosages. Soil samples collected after 15 days of last spray, did not show the presence of chlorantraniliprole residue. Based on complete dissipation of the insecticide the waiting period for harvest of crop was found to be five days after the last spray.

Keywords: cabbage, chlorantraniliprole, dissipation, UPLC

Introduction

Chlorantraniliprole is a new insecticide belonging to the anthranilic diamide chemical class which has a novel mode of action called ryanodine receptor activators. The application of chlorantraniliprole at the recommended dose is quite safe for crop protection and environmental contamination point of view and hence can be a useful tool in the IPM of different insect pests of cauliflower (Kar *et al.* 2013) [2]. Being a “reduced risk” pesticide, it is recommended as an alternative to pyrethroids for vegetables (USEPA 2008) [4]. Farmers rely mostly on chemical insecticides to mitigate the losses due to pests in commercial cultivation. The farmers tend to use higher dosage than recommended and the use of pesticides at higher concentration may leave residues in harvested produce and hence there are risks associated with the continuous consumption of contaminated fruits. The residues, though in minute quantities, can accumulate and magnify in the body causing adverse effects on human health. The information on the residues of the new molecule like chlorantraniliprole in cabbage is scanty. Hence, an investigation was carried out to study chlorantraniliprole residue in cabbage, its dissipation pattern and to find out suitable waiting period for last spray.

Material and Methods

A field experiment was conducted during *rabi*, 2015-16 at Main Agricultural Research Station, University of Agricultural Sciences, Dharwad. The experiment was conducted in a randomized block design using three replicates for each treatment. Four applications of chlorantraniliprole at 0.20 ml L⁻¹ (single dose) and 0.40 ml L⁻¹ (double dose) were given at an interval of 15 days on cabbage heads using knapsack sprayer. The first spray was made at three weeks, after transplanting and water was sprayed in the control plot. Cabbage heads were collected at 0 (2 hrs), 1, 2, 3, 5, 7, 10 and 15 days after the last spray. Soil samples were analysed on 15th day from the last spray.

Sample preparation for insecticide residue analysis by QuEChERS (Quick Easy Cheap Effective Rugged and Safe) method

Reference Standards

Certified pesticide reference standards namely, Chlorantraniliprole (99.1 %), obtained from Riedel-de Haen (Seelze, Germany) were used for the analysis. HPLC grade solvents (acetonitrile and formic acid) were obtained from Merck India Ltd (Mumbai, India).

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Deionised the application of the vortex-assisted MSPD method to the analysis of real samples showed TCS in some fish liver and fish gill samples at trace levels. water for use of a mobile phase was produced using Milli Q ultra-pure water system from Millipore (Milford, MA, USA). Dispersive solid phase extraction (d-SPE) material, viz., Primary Secondary Amine (PSA, 40 μm , Bondesil) was obtained from Agilent Technologies (Bangalore, India).

Estimation of residue content

Residues of insecticides were quantified by comparison of peak area of unknown or spiked samples run under identical conditions with that of standards. The retention time of chlorantraniliprole was found to be 2.303.

Results and Discussion

The UPLC chromatograms for chlorantraniliprole residues are depicted in Fig. 1a to Fig. 1c. The chromatogram of sample analysed on 0 day (2 hrs after last spray) clearly indicated the presence of chlorantraniliprole residue (RT of 2.366 min). However, the peak for the residue was absent on third day, indicating complete dissipation of the chemical. The residue levels of chlorantraniliprole in cabbage heads at different intervals of time after the last spray and at harvest are presented in Table 1. The initial deposits of chlorantraniliprole residues in cabbage heads were found to be above the maximum residue limit (0.3 mg kg^{-1}) on 0 day (2 hrs after last spray) recording 0.81 mg kg^{-1} in single dose and 2.00 mg kg^{-1} in double dose of application.

The residues of chlorantraniliprole in cabbage dissipated to 0.59 and 1.21 mg kg^{-1} after 24 hours (one day) of the last

spray accounting for 26.6 and 39.3 per cent dissipation in single and double dosage, respectively. The levels of residues after one day of spray were above maximum residue limit (MRL). The residue contents were further reduced to 0.26 and 0.43 mg kg^{-1} on 2nd day of spray in single and double dosage, respectively. On 2nd day, chlorantraniliprole residue reached below the MRL in single dosage, whereas in double dosage the residue was still above the MRL. The insecticide was dissipated by 67.2 and 78.4 per cent in single and double dosage, respectively in two days' time. The residue of the insecticide reached below detectable limit by 3rd day and showed 100 per cent dissipation of chlorantraniliprole at both the dosages (Fig. 2a). The chlorantraniliprole residue in cabbage heads and soil at harvest was also below detectable level.

Low residual activity of chlorantraniliprole might be due to photo degradation of the molecule (Kar *et al.*, 2013) [2]. The higher temperature during the last spray of the chemical might have helped in faster degradation. Kar *et al.* (2013) [2] also observed that the residue of chlorantraniliprole reached below detachable limit of 0.1 mg kg^{-1} in 3 and 5 days in cauliflower when applied at single (9.25g a.i.ha⁻¹) and double (18.5 g a.i.ha⁻¹) dosage, respectively. A faster dissipation of chlorantraniliprole applied to tomato crop was observed by Malhat *et al.* (2012) [3] and found its half-life to be 3.30 days. Initial residues of chlorantraniliprole at single and double doses on the fruits of brinjal were 0.72 and 1.48 mg kg^{-1} , while on okra fruits, the residues were 0.48 and 0.91 mg kg^{-1} , respectively. The residues reached below detectable level of 0.01 mg kg^{-1} on the 10th day by Vijayasree *et al.* (2015).

Table 1: Persistence and dissipation of chlorantraniliprole in abbage heads (mg kg^{-1}) at different time intervals after the last spray and at harvest

Days after spraying (DAS)	Single dose @ 0.20 ml L ⁻¹		Double dose @ 0.40 ml L ⁻¹	
	Residue level	Dissipation %	Residue level	Dissipation %
0 (2hrs after harvest)	0.81	-	2.00	-
1	0.59	26.6	1.21	39.3
2	0.26	67.2	0.43	78.4
3	BDL	100	BDL	100
5	BDL	100	BDL	100
7	BDL	100	BDL	100
10	BDL	100	BDL	100
15 (at harvest)	BDL	100	BDL	100
Soil at harvest	BDL	100	BDL	100

MRL: 0.3 mg kg^{-1}

BDL: Below Detectable Limit

MRL: Maximum Residue Limit

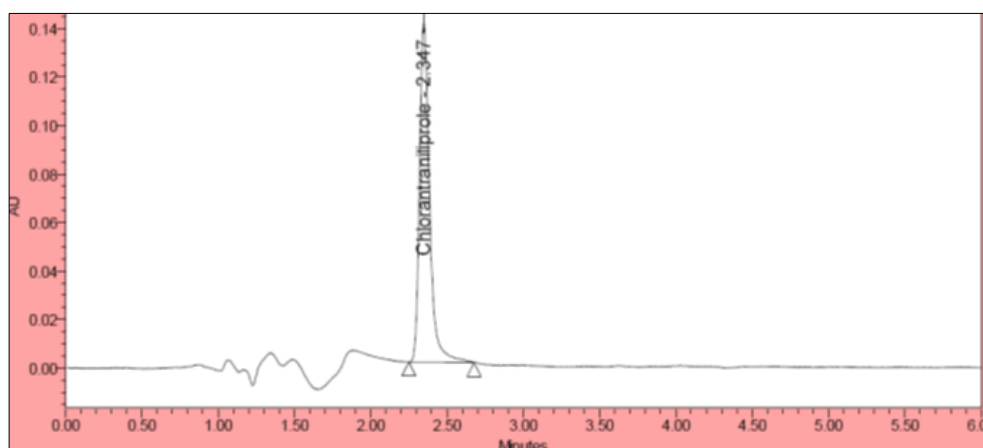


Fig 1a: UPLC chromatogram of standard chlorantraniliprole

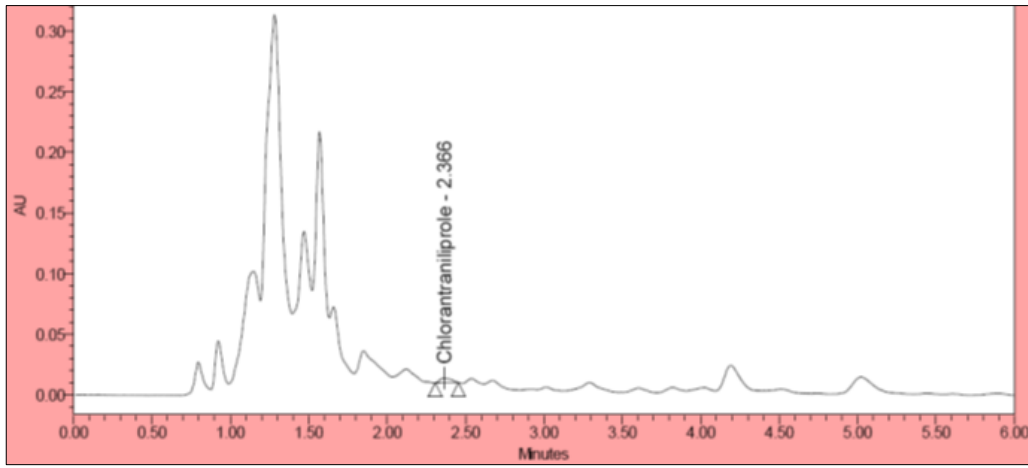


Fig 1b: Chromatogram of chlorantraniliprole (0.40 ml L^{-1}) treated cabbage sample on zero day (2 hrs after last spray)

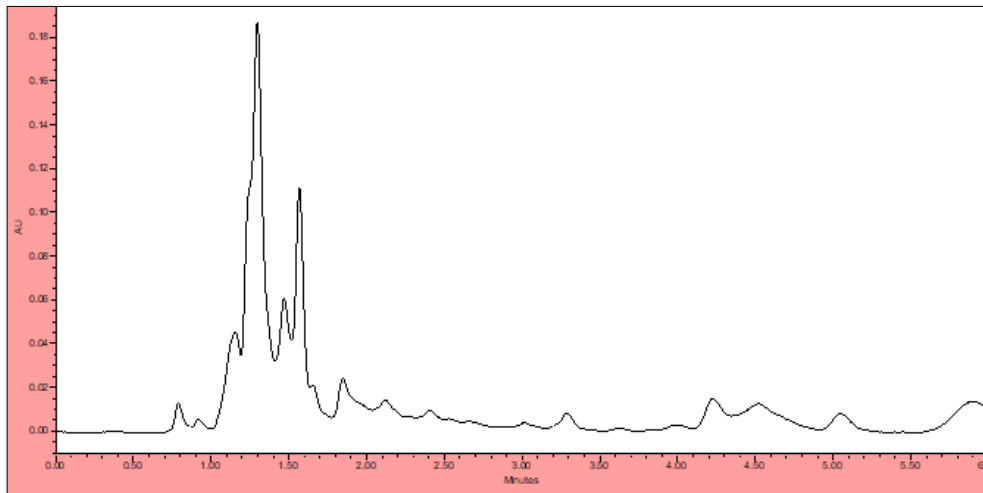


Fig 1c: Chromatogram of chlorantraniliprole (0.40 ml L^{-1}) treated

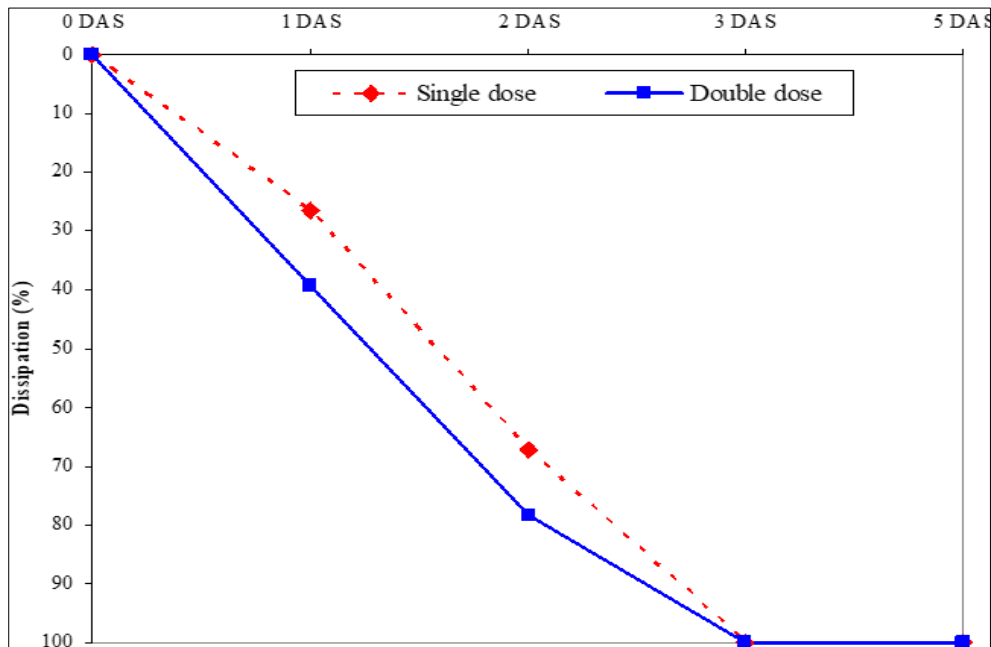


Fig 2a: Dissipation of chlorantraniliprole insecticide in cabbage

Conclusion

The residues of insecticide chlorantraniliprole persisted in cabbage up to two days. The residues reached below maximum residue limit of 0.3 in two days when applied at their normal dosage of 0.2 ml L^{-1} . chlorantraniliprole insecticide dissipated completely on 3rd day of the last spray.

Thus the insecticide chlorantraniliprole may be considered safe chemical and based on complete dissipation of the insecticide the waiting period for harvest of crop was found to be three days after the last spray.

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