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# Persistence of a Neonicotinoid Insecticide, Acetamiprid on Chili (*Capsicum annum* L.)

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**Abstract** An experiment was conducted to find out the persistence of two different formulations of Acetamiprid, namely 20% SP (soluble powder) and 20% SL (soluble liquid) in chili. Following three applications @ 20 g a.i./ha and 40 g a.i./ha, the initial deposits were found to be 0.0207, 0.0405, 0.0244 and 0.1039 μg/g. The half-life values of acetamiprid in chili were in the range of 2.24–4.84 days. A waiting period of 1 day is suggested.

**Keywords** Acetamiprid · Chili · Soil · Persistence

Chili (*Capsicum annum* Linn) is an important spice crop grown in India and used in every vegetarian and non-vegetarian preparation, curries and pickles. It is also consumed raw as green chili in salads. India is the only country rich in many varieties of chili having different quality factors. While consumption of chili is highest in India, maximum export is also from here. Chili continued to be the major item of export in the world in terms of quantity. Chili production in 2004–2005 was estimated around 12 lakh tonnes and about 13% of the production was exported (Kochi 2005). The chili crop is ravaged by a wide array of insect pests among which thrips alone causes 60–74% yield losses (Reddy et al. 2007). In addition to thrips; aphids and whiteflys also fall among the major pests of chili. Farmers apply different pesticides for controlling them. Acetamiprid ((E)-$N^1$-[(6-chloro-3-pyridyl) methyl]-$N^2$-cyano-$N^1$-methylacetamidine) is a systemic neonicotinoid insecticide developed by Nippon Soda Co. Ltd., for soil and foliar applications (Anonymous 1998). Like other neonicotinoid compounds, acetamiprid shows excellent activity against Hemipteran, Thysanopteran and Lepidopteran insects. It is used for the control of sucking insects in leafy & fruiting vegetables, cole crops, citrus fruits, pome fruits, grapes, cotton, ornamental plants and flowers (USEPA 2002). In particular, acetamiprid shows excellent activity against aphids, whiteflys and thrips (Anonymous 1998). It acts on the central nervous system causing irreversible blocking of the postsynaptic nicotinergic acetylcholine receptors (Anonymous 1998). Acetamiprid degrades rapidly and poses low risks to the environment (USEPA 2002). In recent years, works on persistence of acetamiprid on various crops like gram, mustard etc. has been conducted (Gupta et al. 2005; Pramanik et al. 2006). In the present study, a field trial was undertaken to find out the dissipation pattern of acetamiprid on chili.

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## Materials and Methods

The field experiment was carried out at the experimental farm of Institute of Pesticide Formulation Technology (IPFT), located at Gurgaon, Haryana, India. The plots ($5 \times 3$ $m^2$) were laid out in randomized block design (RBD) and planted with the Pusa Jwala variety of chili. Two formulations of Acetamiprid namely 20% SP (soluble powder) and 20% SL (soluble liquid) were examined for their persistence behavior on the chili fruit and their residues in soil after 7 days of application. An aqueous solution of the formulations were applied at the rates of 20 g a.i./ha (5 g a.i./100 L water) and 40 g a.i./ha (10 g a.i. /100 L water), which corresponded to recommended ($T_1$ & $T_3$) and double the recommended doses ($T_2$ & $T_4$), respectively. The application was done by a knapsack sprayer. Each plot

($5 \times 3$ m$^2$) was sprayed with 0.60 L of the formulation solution. The first application was made when the plants were at the flowering bud initiation stage. The next two applications were followed at 14 days interval. Control plots were maintained for blank matrix. Samples (1 kg), from both treated and control plots were collected randomly from different heights of the plants at different time interval (0, 1, 3, 5 and 7 days) after the last application. Soil samples (2 kg) were collected after 7 days of the last application.

Chili samples were homogenized after chopping into small pieces and the representative sample (20 g) was extracted with acetone (100 mL) in a rotary shaker for 1 h. The extract was filtered through buchner funnel. Extraction was repeated two more times with 50 mL acetone each time. The collected filtrate was concentrated to 5 mL using rotary vacuum evaporator (40°C). The extract was taken in a 500 mL separatory funnel containing 100 mL of 5% sodium chloride solution. The extract was then partitioned with hexane (50 + 50 mL) twice. The hexane layer was discarded. The extract was finally partitioned with dichloromethane (100 + 50 + 50 mL) three times and collected by passing through anhydrous sodium sulfate. The extract was concentrated to near dryness using rotavapor (35°C). The extract was cleaned up by column chromatography using florisil PR (6 g) in a 2 cm i.d. glass column. The extract was eluted with hexane + acetone (1: 1, 150 mL). The residue was concentrated to dryness and diluted with methanol (HPLC Grade) before HPLC analysis.

Soil samples were air-dried, ground and passed through 2 mm sieve before sampling and 50 g of representative sample was processed following the same method as described for chili samples.

The residues of acetamiprid were estimated by high-pressure liquid chromatography (HPLC) using DIONEX® HPLC equipped with quaternary pump, column oven, photo diode array detector and ODS Hypersil column ($250 \times 4.6$ mm). The detector was set at 246 nm wavelength. The solvent system used was Methanol: Water (60: 40) at 1.2 mL/min flow rate. The retention time was 3.83 min.

**Table 1** Average recovery percentage and relative standard deviation at different fortification levels

| Substrate | Fortification level | Average recovery percentage[a] | Relative standard deviation |
|---|---|---|---|
| Chili | 0.1 | 78.0 | 12.4 |
| | 0.5 | 88.0 | 11.6 |
| | 1.0 | 96.0 | 11.4 |
| Soil | 0.1 | 98.6 | 10.8 |
| | 0.5 | 107.6 | 10.5 |
| | 1.0 | 101.6 | 10.7 |

[a] Average of three replications

## Results and Discussion

The limit of detection (LOD) and limit of quantification (LOQ) of the method were calculated according to the

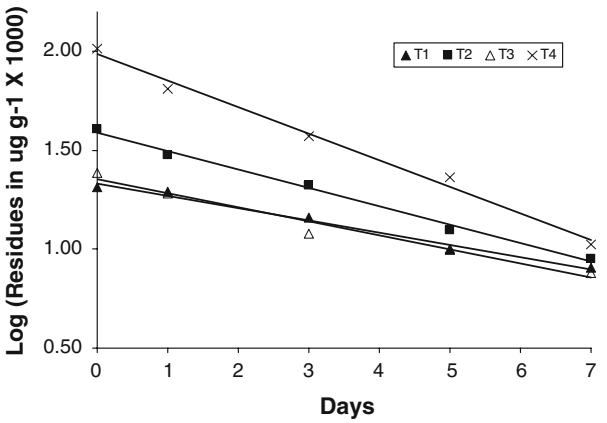


**Fig. 1** Linear plot for first order kinetics of Acetamiprid residues on chili ($T_1$, $T_2$, $T_3$ and $T_4$)

**Table 2** Residues of acetamiprid 20% SP vis-à-vis 20% SL in chili

| Days | Residues in µg/g ± SD (% dissipation)* | | | |
|---|---|---|---|---|
| | $T_1^a$ | $T_2^b$ | $T_3^c$ | $T_4^d$ |
| 0 | 0.0207 ± 0.010 | 0.0405 ± 0.022 | 0.0244 ± 0.015 | 0.1039 ± 0.120 |
| 1 | 0.0196 ± 0.012 (5.31) | 0.0301 ± 0.018 (25.68) | 0.0192 ± 0.010 (21.31) | 0.0648 ± 0.085 (37.63) |
| 3 | 0.0144 ± 0.010 (30.43) | 0.0210 ± 0.012 (48.15) | 0.0120 ± 0.014 (50.82) | 0.0371 ± 0.025 (64.29) |
| 5 | 0.0099 ± 0.014 (52.17) | 0.0125 ± 0.016 (69.14) | 0.0100 ± 0.010 (59.02) | 0.0232 ± 0.012 (77.67) |
| 7 | 0.0081 ± 0.010 (60.87) | 0.0090 ± 0.010 (77.78) | 0.0076 ± 0.012 (68.85) | 0.0106 ± 0.014 (89.80) |

[a] Acetamiprid 20% SP (recommended dose); [b] acetamiprid 20% SP (double the recommended dose); [c] acetamiprid 20% SL (recommended dose); [d] acetamiprid 20% SL (double the recommended dose); * Average of three replications; Values in parenthesis are % dissipation in comparison to 0 day

**Table 3** Regression equation and half-life of acetamiprid on chili

| Treatment | Half-life, $T_{1/2}$(days) | Regression equation | Correlation coefficient ($r$) | Regression coefficient ($R^2$) |
|---|---|---|---|---|
| $T_1$ | 4.84 | $y = 1.33 - 0.0622x$ | −0.99 | 0.98 |
| $T_2$ | 3.23 | $y = 1.59 - 0.0933x$ | −1.00 | 0.99 |
| $T_3$ | 4.23 | $y = 1.35 - 0.0711x$ | −0.98 | 0.97 |
| $T_4$ | 2.24 | $y = 1.98 - 0.1341x$ | −1.00 | 0.99 |

**Table 4** Theoretical maximum residue contribution (TMRC) and waiting period for different treatments

| Insecticide | MPI[a] (mg/person/day) | Treatments | TMRC[b] (theoretical maximum residue contribution) (mg/person/day) | | | | | Waiting period (days) |
|---|---|---|---|---|---|---|---|---|
| | | | 0 Day | 1 Day | 3 Day | 5 Day | 7 Day | |
| Acetamiprid | 3.5 | $T_1$ | 0.00104 | 0.00098 | 0.00072 | 0.00050 | 0.00041 | 1 |
| | | $T_2$ | 0.00203 | 0.00151 | 0.00105 | 0.00063 | 0.00045 | 1 |
| | | $T_3$ | 0.00122 | 0.00096 | 0.00060 | 0.00050 | 0.00038 | 1 |
| | | $T_4$ | 0.00520 | 0.00324 | 0.00186 | 0.00116 | 0.00053 | 1 |

[a] MPI = ADI (0.07 mg/kg) × Average body weight (50 kg); [b] TMRC = Residues × Average daily consumption (50 g); residues safe when TMRC < MPI

peak-to-peak noise method as proposed by US EPA (Corley 2003). The LOD and LOQ were found to be 0.033 and 0.1 µg/g, respectively. To validate the method, recovery experiments were carried out at three fortification levels (1, 5 and 10 LOQ). The average recovery percentages for chili were ranged between 78% and 96% with relative standard deviations (RSD) from 11.4 to 12.4; while for soil the average recovery percentages varied between 98.6% and 107.6% and the relative standard deviations (RSD) ranged from 10.5 to 10.8 (Table 1).

The average concentration of acetamiprid residues on chili as obtained from different treatments are shown in Table 2, which shows the average initial deposit of 0.0207 µg/g for $T_1$, 0.0405 µg/g for $T_2$, 0.0244 µg/g for $T_3$ and 0.1039 µg/g for $T_4$. After 1 day, the residues were found to dissipate by about 5–21% in cases of recommended dose ($T_1$ & $T_3$) of acetamiprid 20% SP and 20% SL formulations. In case of double the recommended dose ($T_2$ & $T_4$) of the said two formulations of acetamiprid, the dissipation was 25–38% after 1 day. Dissipation percentage increased with the progress in time and reached 60–69% for the recommended doses and 78–89% for double the recommended doses after 7 days of last application. The log values of the mean residues ($\times 10^3$) for the four different treatments were plotted against different days of observation to interpret statistically (Fig. 1). The declining nature of the residues followed first order kinetics for all the treatments.

The regression equation and half-life values for the different treatments were computed statistically and is presented in Table 3. The half-life values ($T_{1/2}$) of acetamiprid in chili were found to be 4.84 days for recommended dose ($T_1$) and 3.23 days for double the recommended dose ($T_2$) when sprayed with 20% SP formulation and when sprayed with 20% SL formulation; the half-life values ($T_{1/2}$) of acetamiprid in chili were found to be 4.23 days for recommended dose ($T_3$) and 2.24 days for double the recommended dose ($T_4$). No residues were detected in the samples of chili of control plots during the entire study.

Soil samples, collected from the four different treatments in chili plot after 7 days of the last application, showed presence of residues below the quantifiable limit (BQL).

The MRL value of acetamiprid for chili is neither available in India nor has it been set by codex alimentarius commission. Therefore the waiting period has been calculated based on the acceptable daily intake (ADI) values (SANCO 2004). Based on the calculations (Table 4) a waiting period of 1 day is suggested as the initial deposits itself were safe. Therefore, the new generation insecticide, acetamiprid can safely be used on chili.

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