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# Persistence of Imidacloprid in/on Cabbage and Cauliflower

V. T. Gajbhiye, S. Gupta, R. K. Gupta

Division of Agricultural Chemicals, Indian Agricultural Research Institute, New Delhi 110 012, India

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Cabbage and cauliflower are two main vegetable crops in northern India. Various insect pests like aphids, white fly, lepidopteran, etc, heavily damage these crops. Insecticides of various groups are being used to control these pests. However, insect pests developed resistance to the conventionally used insecticides. Imidacloprid [1- (6-chloro-3-pyridylmethyl) -N-nitro-imidazolidin-2-ylidene amine], a nicotinoid insecticide, has been found very effective against these resistant pests due to different mode of action (Chalam and Subbaratnam, 1999; Elzen, 1997; Olson et al. 1996; Elbert and Nauen, 1996; Barber et al. 1999; Gorman et al. 1998; Rongai et al. 1998). The literature search revealed that even though some reports are available on bioefficacy of imidacloprid in cabbage and cauliflower (Zhu et al. 1996; Sreekanth et al. 2000; Natwick et al. 1996), not much information is available on its residues. Therefore, experiments were conducted to study the persistence of imidacloprid in cabbage and cauliflower crop.

## MATERIALS AND METHODS

The analytical grade imidacloprid and its formulation (Confidor 200 SL) was supplied by Bayer (India) Limited. All the solvents were glass distilled before use. The standard solutions of imidacloprid were made in HPLC grade acetonitrile.

Field experiments were carried out for two consecutive years using randomized block design with three replications. Cabbage (var.Golden acre) and cauliflower (var. Ketki) were grown in 5 x 5 $m^2$ plots using normal agronomic practices. The row to row and plant to plant distance was maintained at 60 cm. Imidacloprid (Confidor 200 SL) emulsion was sprayed at recommended dose (20 g ai/ha) and double the recommended dose (40 g ai/ha) with hand sprayer (Maruti 1L capacity) at leaf curling stage in cabbage and curd formation stage in cauliflower. The samples of cabbage and cauliflower (3 heads/curds each) were drawn randomly at different time intervals.

The cauliflower samples were separated into curd and leaves. The samples of cabbage heads and cauliflower curd and leaves were cut into small pieces; mixed and representative sub-samples (50 g) from each in duplicate were drawn by

*Correspondence to:* V. T. Gajbhiye

quartering method. The samples were extracted by macerating/blending. The representative samples were transferred to Waring blender, 100 ml distilled acetone added and macerated for 3 minutes at high speed. The contents were filtered through buchner funnel. The solid residues were transferred back to blender jar and extraction procedure repeated two more times using fresh acetone. The acetone from the combined extract was removed under vacuum using rotary evaporator. The aqueous extract was quantitatively transferred to 500 ml separatory funnel and diluted with 100 ml of 10% sodium chloride solution. The aqueous phase was made acidic (pH~ 4.0) by adding dilute HCl and partitioned three times (50 ml each) with distilled hexane. The hexane phases were discarded. The acidic aqueous phase was then made alkaline (pH ~9.0) by adding 10% sodium hydroxide solution and again partitioned three times (50 ml each) with distilled dichloromethane. The dichloromethane phases were combined, passed through anhydrous sodium sulfate to remove traces of moisture and concentrated using rotary vacuum evaporator. Finally residues were dissolved in HPLC grade acetonitrile and analyzed by Water's HPLC equipped with column RP C-8 (250 mm x 4 mm, 10 μm), detector variable UV-Visible (model 484), binary pump (model 501) and injector rheodyne (20 μl loop). The operating parameters were: $\lambda_{max}$ 270 nm, injection volume 20 μl, solvent system acetonitrile-water gradient as given in Table 1.

**Table 1**. Gradient conditions.

| Time | Flow (ml/min) | % Acetonitrile | % Water |
|---|---|---|---|
| 0 | 1.0 | 5 | 95 |
| 4 | 1.0 | 25 | 75 |
| 11 | 1.0 | 40 | 60 |
| 12 | 1.0 | 90 | 10 |
| 17 | 1.0 | 90 | 10 |
| 18 | 1.0 | 5 | 95 |
| 20 | 1.0 | 5 | 95 |

Under these conditions the retention time of imidacloprid was 10.5 minutes.

## RESULTS AND DISCUSSION

The recoveries of imidacloprid from cabbage and cauliflower for the method varied from 82-90%. The persistence of imidacloprid on cabbage and cauliflower was studied for two consecutive years i.e. 2000 and 2001 at recommended and double the recommended dosages. The residue data for imidacloprid on cabbage is presented in Table 2, cauliflower in Table 3-4 and calculated half-life values in Table 5.

The application of imidacloprid on cabbage @ 20 g a.i./ha (recommended dose) resulted in initial deposits of 0.023 and 0.147 μg $g^{-1}$ during 2000 and 2001 year, respectively. At higher dose (40 g a.i./ha) the initial deposits were 0.067 and 0.243 μg $g^{-1}$ during 2000 and 2001 (Table 2).

**Table 2**. Residues of imidacloprid in cabbage.

| Year | Dose (g ai/ha) | Average Residues (μg $g^{-1}$) | | | |
|---|---|---|---|---|---|
| | | 0 day | 2 day | 5 day | 10 day |
| 2000 | 20 | 0.023 | 0.019 (17.4)* | ND (100) | ND |
| | 40 | 0.067 | 0.044 (34.3) | 0.025 (62.7) | ND (100) |
| 2001 | 20 | 0.147 | 0.076 (48.3) | ND (100) | ND |
| | 40 | 0.243 | 0.182 (25.1) | 0.033 (86.4) | ND (100) |

*Figures in parenthesis denotes percent dissipation;
ND – Non detectable (< 0.02 μg $g^{-1}$)

Variation in initial deposits during different years could be attributed to plant size and the weather conditions during the spray. The residues dissipated with time and 62-100% of the applied imidacloprid was lost within 5 days. No residues were detected in cabbage head on 10th day. The dissipation of residues followed first order kinetics (Table 5) and the half-life values varied from 0.6-2.1 days during the two years. Mukherjee and Gopal (2000) have reported half-life of 3.4 days for imidacloprid on cabbage heads.

In cauliflower crop, residues of imidacloprid were determined in curd and leaves. The initial deposits in curd were 2.2 and 0.82 μg $g^{-1}$ at recommended dose and 3.9 and 1.8 μg $g^{-1}$ at double the recommended dose, respectively, during 2000 (Table 3) and 2001 (Table 4). Similar to cabbage, wide variation was observed in initial deposits during the two years. The residues dissipated quite fast and 47-68% dissipation was recorded within two days. The half-life values based on first order kinetics were 0.7-1.3 days (Table 5).

**Table 3.** Residues of imidacloprid in cauliflower (Year 2000).

| Sample | Dose (g ai/ha) | Average Residues (μg $g^{-1}$) | | | | |
|---|---|---|---|---|---|---|
| | | 0 day | 2 day | 5 day | 7 day | 15 day |
| Curd | 20 | 2.20 | 0.70 (68.2) | 0.02 (99.1) | ND (100) | ND |
| | 40 | 3.90 | 2.03 (47.9) | 0.19 (95.1) | 0.07 (98.2) | ND (100) |
| Leaves | 20 | 0.23 | 0.08 (65.2) | ND (100) | ND | ND |
| | 40 | 0.56 | 0.20 (64.3) | 0.08 (85.7) | 0.02 (96.4) | ND (100) |

*Figures in parenthesis denotes percent dissipation;

ND – Non detectable (< 0.02 μg $g^{-1}$)

In leaves, the initial deposits during 2000 (Table 3) and 2001 (Table 4) were 0.23 and 0.27 μg $g^{-1}$ at recommended dose and 0.56 and 0.69 μg $g^{-1}$ at double the recommended dose, respectively. The lower deposits on leaves as compared to curd could be attributed to the fact that the rough surface of curd could retain more spray material as compared to smooth waxy surface of the leaves. The residues dissipated with time and 54-78% dissipation was recorded within two days. The half-life values based on the first order kinetics were 0.6-1.5 days.

The residues in cauliflower, in general, were more than the cabbage during all the years. It seems the cauliflower can retain more spray material due to rough surface and the morphology of the plant.

**Table 4**. Residues of imidacloprid in cauliflower (Year 2001).

| Sample | Dose (g ai/ha) | Average Residues (μg $g^{-1}$) | | | |
|---|---|---|---|---|---|
| | | 0 day | 2 day | 5 day | 10 day |
| Curd | 20 | 0.82 | 0.34 (58.5) | 0.035 (95.7) | ND (100) |
| | 40 | 1.78 | 0.57 (67.9) | 0.12 (93.3) | ND (100) |
| Leaves | 20 | 0.27 | 0.06 (77.8) | 0.02 (92.6) | ND (100) |
| | 40 | 0.69 | 0.32 (53.6) | 0.03 (95.7) | ND (100) |

*Figures in parenthesis denotes percent dissipation;
ND – Non detectable (< 0.02 μg $g^{-1}$)

**Table 5**. Regression equations and half-life of imidacloprid in cabbage and cauliflower.

| Crop | Year | Dose (g ai/ha) | Regression equation | Corr. coeff. | Half-life (days) |
|---|---|---|---|---|---|
| Cabbage | | | | | |
| Head | 2000 | 20 | Y = -1.4793 - 0.4804 X | 0.96 | 0.60 |
| | | 40 | Y = -1.1910 - 0.1399 X | 0.99 | 2.15 |
| | 2001 | 20 | Y = -0.6036 - 0.4487 X | 0.96 | 0.70 |
| | | 40 | Y = -0.5270 - 0.1792 X | 0.97 | 1.70 |
| Cauliflower | | | | | |
| Curd | 2000 | 20 | Y = +0.4684 - 0.4167 X | 0.98 | 0.70 |
| | | 40 | Y = +0.6794 - 0.2639 X | 0.99 | 1.16 |
| Leaves | 2000 | 20 | Y = -0.4464 - 0.4851 X | 0.97 | 0.60 |
| | | 40 | Y = -0.2533 - 0.1952 X | 0.99 | 1.50 |
| Curd | 2001 | 20 | Y = -0.0208 - 0.2783 X | 0.99 | 1.08 |
| | | 40 | Y = -0.2401 - 0.2336 X | 0.99 | 1.30 |
| Leaves | 2001 | 20 | Y = -0.6480 - 0.2208 X | 0.98 | 1.40 |
| | | 40 | Y = -0.0779 - 0.2779 X | 0.98 | 1.10 |

Corr. coeff. – Correlation coefficient

The residues, including initial deposits, in cabbage at both the spray levels were below the prescribed MRL value of 0.5 μg $g^{-1}$ (Anonymous, 2002). In almost all the samples of cauliflower curd the initial deposits were above the prescribed MRL value of 0.5 μg $g^{-1}$ (Anonymous, 2002), however, the residues were lower than the MRL value on 5th day. In cauliflower leaves the initial deposits were below MRL at recommended dose, but at higher dose the residues became lower than the MRL value only on 2nd day. Therefore, a waiting period of 1-2 days can be recommended for cabbage and cauliflower leaves and 5 days for cauliflower curd. The schedules can be considered safe from toxic residues point of view.

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