# Studies on Dissipation Kinetics and risk Assessment of Imidacloprid Residues in Tarai Region of Uttarkhand

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Received February 10, 2020 & Revise May 15, 2020 & Accepted June 20, 2020

## Abstract

Dissipation behavior of neonicotinoid insecticide, imidacloprid (Imidacel 17.8 SL), in okra fruit was studied following application at dose 20 g a.i. ha-1 at VRC G.B.P.U.A.T. Pantnagar Uttrakhand, India, showed a initial deposit of 1.027 mg kg-1 imidacloprid in okra fruit samples. The residue level in okra fruit dissipated to 33.008 % of initial deposition after one day and it further declined to 80.817% after 5 days of last spraying. The residues dissipated below detection limit within 7 days in okra fruits. The half life ($t_{½}$) of imidacloprid in okra fruits was estimated 2.094 days. The residue level went below MRL (0.7mg kg-1 for okra) within 3 days at the recommended dose.

**Keywords:** Imidacloprid, Okra, Abelmoschus Esculentus, Residues, Uttarakhand, Dissipation.

## Introduction

Okra [Abelmoschus esculentus (L.) Monech] is a member of family Malvaceae and close relative of cotton and hibiscus, having good nutritive value and is cultivated during summer and rainy seasons in all over India. Use of various insecticides is normal and traditional practice for the control of different insect pest in okra (Singh SP, Kumar KS and Tanwar RS,2014; Sinha SR and Sharma RK., 2007) Imidacloprid, is a systemic insecticide of neonicotinoid group and is widely used against variety of pest in okra(Dhanalakshmi DN and Mallapur CP,2018 ; Venkataravanappa VM, Krishnareddy M, Lakshinimarayanreddy CN and Salil J, 2011) Mode of action of imidacloprid is similar to naturally occurring nicotinoids. Imidacloprid disturbs transmission of impulse in nervous system of insect, by acting as an antagonist to the nicotinic acetylcholine receptor, it causes continuous excitation of nerve cells, finally resulting in death of treated insect( Yamamoto I,1993)

Although there have been various studies on the dissipation of imidacloprid on food (Utture SC, Banerjee K, Kolekar SS, Dasgupta s, Oulkar DP, Patil SH et al.,2012 ; Phartiyal T, Srivastava RM. ; Gupta M, Sharma A and Shanker A, 2008) few articles have been published on degradation of its residues on okra and as significant concern is being given over magnitude of pesticide left in vegetable following their use it is therefore, present study is conducted in okra to evaluate the level of imidacloprid residues following its application under tarai agro-climatic condition of

Uttarakhand.

## Objective of Research

1.Dissipation behavior of neonicotinoid insecticide, imidacloprid (Imidacel 17.8 SL), in okra fruit

## Research methodology

### 1. Material and Methods:

Chemical and Reagents

Analytical standard of imidacloprid (purity>99%) used in present study was purchased from SIGMA-ALDRICH Inc, USA. Imidacloprid 17.8 % Sl ( Imidacel® ) was used in the field study. The Chemicals and reagents used were acetone (HPLC grade) and acetonitrile (HPLC grade) were purchased from HiMedia Laboratories Pvt.Ltd. Water used was double distilled. To 10 mg of imidacloprid standard, 100 ml of acetonitrile was added to prepare a stock standard solution of imidacloprid, which was further diluted to prepare working solution of different concentration ranging from 1 to 100 µg/ml.

Field Study

Field experiment was conducted during Kharif (summer) 2017, using randomized block design (RBD) with three replication at Vegetable Research Centre G.B.P.U.A.T. Pantnagar. This area is under tarai agro climatic zone of Uttarakhand with hot and humid conditions during rainy season. The okra variety Parbani kranti, best suited in this area for good yield, was cultivated adopting all recommended agronomic practices. An aqueous solution of the insecticide Imidacel® (imidacloprid 17.8 Sl) was sprayed at the time of fruiting at recommended dose ($T_1$:20 g a.i./ha) along with untreated control($T_2$: 0). In order to evaluate dissipation of imidacloprid in okra, fruit samples of okra were collected from each of treated plots (including control) by using standard sampling procedure on 0 (2 hr. after spray), 1, 3, 5, 7, 10, 15 days after last spraying was transferred to laboratory in dry Ice box for further analysis. All the samples were taken for residue analysis after using quartering method.

Laboratory Processing of Samples

Extraction and Cleanup

The extraction and cleanup was done by the method[9] described earlier with some modification. To 50 g okra fruit samples 100 ml extracting solvent (acetone) was added and blended in a pestle mortar and were kept in orbital shaker for 2 hours. The extract was then filtered and decanted in a round bottom flask. The process was repeated two times to achieve quantitative extraction and the extract was kept for ten minutes each time and then pooled. It was then filtered through Whatman No. 1 filter paper and concentrated in rotary vacuum evaporator to near dryness, dissolved in as low volume of HPLC grade acetonitrile as possible. Extract was

then filtered through SPE cartrideges before analyzing in HPLC.

## Research Methodology

A recovery experiment was conducted just before analyzing the samples, to evaluate the reliability and efficiency of extraction and cleanup process of analytical method chosen. For this okra fruit samples (untreated) were spiked at two different concentration level (25 and 50 µg/gm).The spiked samples were allowed to stand for 15 minutes before extraction. The amount of residues was evaluated by comparing the response of sample with the standard response under same operating conditions.

HPLC Analysis

The estimation of imidacloprid residues in okra fruit samples was done by using high performance liquid chromatography. The HPLC system ( Shimadzu Corporation, Kyoto, Japan, Model SPD 10A LC 20 AD) comprised of double plunger pump, Rheodyne injector with a 20 µl loop coupled with PDA detector. The chromatographic separation was achieved on C18 reverse phase column (4×150mm), particle size of 5 µm. The mobile phase consisted of acetonitrile : water (25: 75 v/v) with a constant flow rate of 0.6 ml/min. The chromatography was performed at 25±1°C. The UV detection will be at 270 nm. The chromatogram was analyzed by software and under this operating condition retention time of imidacloprid was 6.22 min.

Statistical Analysis

The quantification of imidacloprid residues was done by comparing the peaks of sample with that peak of standard. The imidacloprid dissipation in fruits of okra follows the first- order dissipation kinetics. The degradation rate constant and half life period were calculated using first – order rate equation, $C=C_0\ e^{-kt}$ , where $C_o$ represents pesticide concentration in µg/g at the 0 days after spray (initial concentration), C represents concentration of pesticide (here imidacloprid) residues at time t; and k is degradation rate constant. And the half life ($t_{½}$) was calculated from k value (rate contant) for each and every experiment (t1/2=ln 2/k).

## Research Results

Method Efficiency

The mean percent recoveries of imidacloprid from okra fruit samples at fortification level of 50, 100 mg/kg were 97.63 and 98 per cent, respectively. Mean percent recovery was found to be more than 85 per cent so the results have been discussed as such without employing any correction factor. The calibration curve for imidacloprid standard displayed good linearity with correlation coefficient 0.996 within the test range. The retention time of tested sample analyte in the spiked samples matched with those of the standards. Thus the used extraction and cleanup procedure for methodology was observed to be highly précised and efficient.

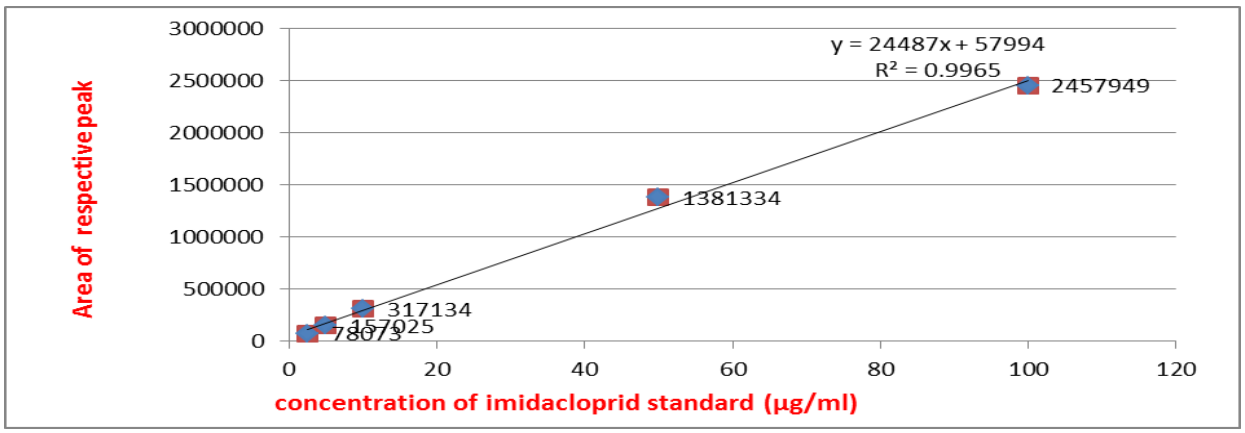


**Figure 1**: Calibration Curve of Standards of Imidacloprid

Dissipation of Imidacloprid

The results of dissipation dynamics of imidacloprid residues in okra fruits at different time intervals at 20 g a.i ./ha and percent dissipation on residues have been presented in Table 1. The initial deposit of 1.027 mg/kg of imidacloprid in okra fruits against recommended was found to decline to 0.688 mg/kg on 1 day after the last application contributing a dissipation of 33.008 percent. Within 5 days of the last application, the dissipation reached 80.817% of the initial amount and within 7 days the residue content reached below detectable level (BDL). Hence more than 80 per cent of imidacloprid residues got dissipated in five days following its spray @ 20 g a.i. /ha. The fast and rapid dissipation of imidacloprid in okra fruit samples might be due to the dilution of chemical because of plant growth. Several agro climatic factors such as temperature, radiation and relative humidity also would have play considerable role in dissipation of imidacloprid residue.

**Table 1:** Residues of Imidacloprid (mg/kg) on Okra Fruits at Different Time Interval After The Application of Imidacloprid 17.8 SL at 20 g.a.i./ha:

| Interval (day) | Imidacloprid residue (mg/kg) | Dissipation rate (%) |
|---|---|---|
| 0(2 hr after spray) | 1.027 | - |
| 1 | 0.688 | 33.008 |
| 3 | 0.367 | 64.264 |
| 5 | 0.197 | 80.817 |
| 7 | BDL | 100 |
| 10 | BDL | - |
| Dissipation rate regression equation between days after treatment and residue level:<br>y = -0.159x + 0.928 | | |
| Correlation coefficient = 0.931 | | |

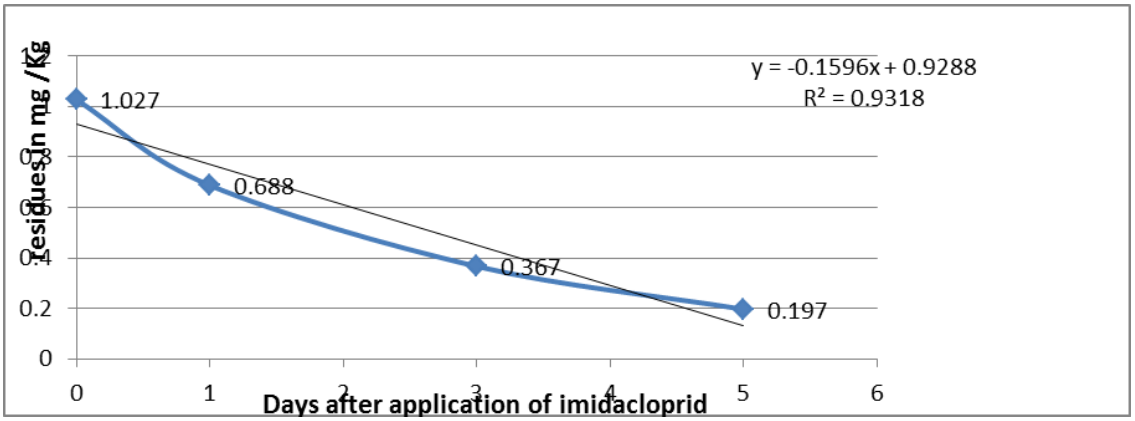


**Figure 2:** First Order Dissipation of Imidacloprid 17.8 SL in Okra Fruit

## Discussion of Research Results

The results are in agreement with those of Pandit et al(2011) who reported an initial deposit of 1.96 mg/kg following application of imidacloprid at 24.5 g a.i./ha in okra fruit at tarai region of west Bengal. It has been reported that imidacloprid residue persisted upto 5 days after treatment with imidacloprid at doses 24.5 g a.i./ha and 49 g a.i./ha in okra fruits(2015) The results are not in agreement with those of Preterhuman et al.(2016) who studied the persistence of imidacloprid in fresh cardamom capsules following application at 20 and 40 g a.i./ha and reported that residues were persisted to 21 days after last application for both doses. And also with Sahoo et al.(2012) and Utture et al.6 who reported an initial deposition of 0.18 mg/kg and 0.12 mg/kg following application of imidacloprid at 0.42g. a.i./ha and 0.25 ml/l in okra and pomegranate fruits, respectively. These differences in persistence of imidacloprid may be due to varied weather conditions, variation in doses and variation in substrates in which insecticide applied.

**Table 2** Statistical Data on Regression Analysis and Half Life for Dissipation of Imidacloprid on Okra Fruit:

| Insecticide | Dose (g a.i./ha) | Regression equation | Half life (days) | Correlation coefficient(R2) |
|---|---|---|---|---|
| Imidacloprid | 20 | Y= 57944+24487X | 2.094 | 0.996 |

The dissipation of imidacloprid residues followed first order kinetics (fig 2) with half life of 2.094 days for recommended dose, generally the persistence of imidacloprid is expressed in terms of half-life ($t_{½}$), i.e time for degradation of pesticide to 50% of its initial concentration. The half-life value of imidacloprid was observed 2.66 days in okra (2016) and 2.31 days in brinjal fruits.(2010) While earlier studies reported 1.04 and 1.13 days half-life ($t_{½}$) of imidacloprid at applied doses of 24.5 and 49g a.i. ha, respectively in okra(2016). This variation might be due to difference in agro climatic conditions of areas under these studies.

Hence, based on the MRL prescribed in okra (0.7µg/g) by European (EU) and approved by Agricultural and Processes Food Products Export Development Authority (APEDA), a waiting period of 3 days is suggested before consumption of okra fruits to reduce health hazards. Therefore, application of imidacloprid 17.8 Sl at recommended dose on okra is quite safe from consumer's health risks and environment contamination point of view.

## Suggestions

This variation might be due to difference in agro climatic conditions of areas under these studies. Therefore, application of imidacloprid 17.8 Sl at recommended dose on okra is quite safe from consumer's health risks and environment contamination point of view. If it is studied in different contexts in the next study, new signs of interest may be discovered for further academic benefits.

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