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Dissipation of imidacloprid residues in okra fruits in Tarai region of Uttarakhand

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Abstract
Dissipation behavior of neonicotinoide insecticide, imidacloprid (Imidacel 17.8 SL), in okra fruits was studied following application at dose 20g *a.i.* ha^{-1}. Observations showed an initial deposition of 1.027 $mgkg^{-1}$ imidacloprid residues in okra fruit samples. Imidacloprid residue levels on 1, 3, 5 days after spray was observed 0.688 $mgkg^{-1}$, 0.367 $mgkg^{-1}$, 0.197 $mgkg^{-1}$, respectively. The residues of imidacloprid went below detection limit within 7 days of application. The estimated half life ($t_{1/2}$) of imidacloprid in okra fruits was observed to be 2.094 days. The residue level went below maximum residue limit (0.7mg kg^{-1} for okra) within 3 days at the recommended dose.

Keywords: Imidacloprid, okra, *Abelmoschus esculentus*, residue, Uttarakhand, Dissipation

1. Introduction
Okra [*Abelmoschu sesculentus* (L.) Monech] is a member of family Malvaceae and close relative of cotton and hibiscus, having good nutritive value and is cultivated during the 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 [1, 2]. Imidacloprid, is a systemic insecticide of neonicotinoid group and is widely used against a variety of pest in okra [3, 4]. Mode of action of imidacloprid is similar to naturally occurring nicotinoids. Imidacloprid disturbs transmission of impulse in the 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 [5].
Although there have been various studies on the dissipation of imidacloprid on food [6, 7, 8], few articles have been published on degradation of its residues on okra. At the present time significant concern is being given over the magnitude of pesticide left in vegetable following their use. It is therefore, present study is conducted at tarai region of Uttarakhand to evaluate the level of imidacloprid residues left in the okra fruits following its application.

2. Material and methods
2.1. Chemical and reagents
Analytical standard of imidacloprid (purity>99%) used in the 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, 100ml of acetonitrile was added to prepare a stock standard solution of imidacloprid, which was further diluted to prepare the working solution of different concentration ranging from 1 to 100 µg/ml.

2.2. Field study
Field experiment was conducted during *Kharif* 2017, using randomized block design (RBD) with three replications at Vegetable Research Centre G.B.P.U.A.T. Pantnagar. This area is under the tarai agro climatic zone of Uttarakhand with hot and humid conditions during the rainy season. The okra variety *Parbanikranti*, 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 the laboratory in dry Ice box for further analysis. All the samples were taken for residue analysis after using quartering method.

2.3. Laboratory processing of samples (Extraction and cleanup)

The extraction and cleanup were done by the method[9] described earlier with some modification. To 50 g okra fruit samples 100ml extracting solvent (acetone) was added and blended in a pestle mortar and were kept in a 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 and then dissolved in a little amount of HPLC grade acetonitrile. Before analyzing in the extract using HPLC it was filtered through SPE cartrideges.

2.4. Method validation

A recovery experiment was conducted just before analyzing the samples, to evaluate the reliability and efficiency of extraction and cleanup process. For this okra fruit samples (untreated) were spiked at two different concentration level (25 µg/gm 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.

2.5. 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 C_{18} 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 was at 270 nm. The chromatogram was analyzed by software and under this operating condition retention time of imidacloprid was observed at 6.22 min.

2.6. Statistical analysis

The quantification of imidacloprid residues was done by comparing the peaks of the sample with that peak of standard. The imidacloprid dissipation in the fruits of okra follows the first- order dissipation kinetics. The degradation rate constant and half life period were calculated by using the first–order rate equation, $C=C_0\ e^{-kt}$, where C_0 represents a 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 constant) for each and every experiment (t1/2=ln 2/*k*).

3. Results and Discussion

3.1. Method Efficiency

The mean percent recoveries of imidacloprid from okra fruit samples at fortification levels 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 (Figure 1). The retention time of 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.

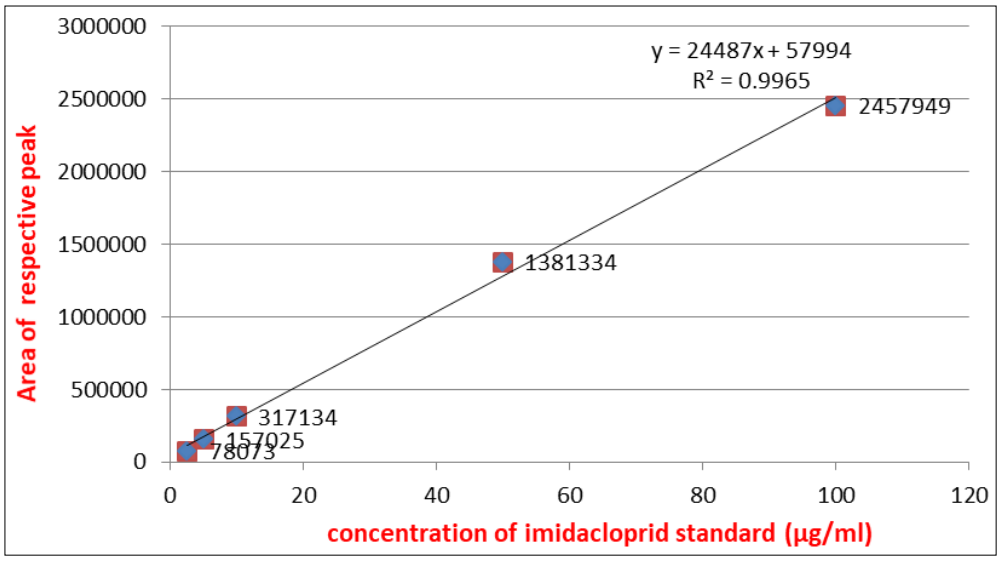

Fig 1: Calibration curve of standards of imidacloprid

3.2. Dissipation of imidacloprid

The results of dissipation dynamics of imidacloprid residues in okra fruits have been presented in Table 1. An initial deposition of 1.027 $mgkg^{-1}$ imidacloprid residues was observed in the okra fruits samples. The amount of imidacloprid residue on 1, 3, 5 days after spray was found 0.688 $mgkg^{-1}$, 0.367 $mgkg^{-1}$, 0.197 $mgkg^{-1}$, respectively. Residue levels of imacloprid went below detectable limit after 7 days of spray. 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 a 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 20g*a.i.* ha^{-1}

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: y = -0.159x + 0.928		
Correlation coefficient = 0.931		

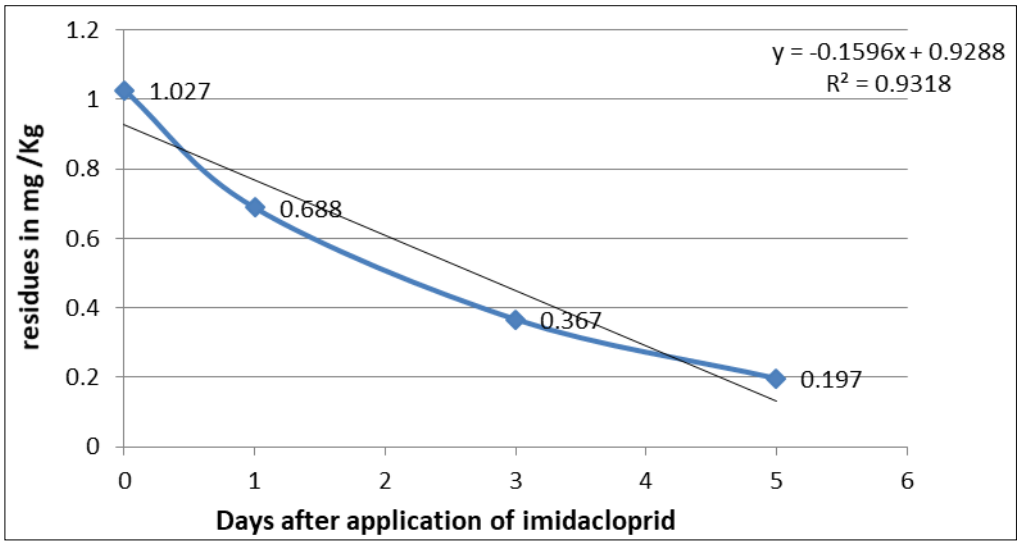

Fig 2: First order dissipation of imidacloprid 17.8 SL in okra fruit.

The results are in agreement with those of Pandit *et al*. (2016) [10] 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 also 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 [11]. The results are not in agreement with those of Pratheeshkumar *et al*. (2016) [12] 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 upto 21 days after last application for both doses but in present study residues went below detectable limit within 7 days of spray. While the results are also not in agreement with those of Sahoo *et al. (*2012) [13] and Utture *et al.* (2012) [6] who reported an initial deposition of 0.18 mg/kg and 0.12 mg/kg following the application of imidacloprid at 0.42g. *a.i.*/ha and 0.25 ml/l in okra and pomegranate fruits, respectively but here in present study initial deposition of imidacloprid was found much more. These differences in persistence of imidacloprid may be due to varied weather conditions, variation in doses and variation in substrates in which insecticide applied.

Here as shown in Figure 2 dissipation of imidacloprid residues in okra fruit samples followed first order kinetics. After putting the observed values in first order equation the half life of imidacloprid in okra fruit samples was calculated as 2.094 days for recommended dose, generally the persistence of imidacloprid is expressed in terms of half-life ($t_{1/2}$), *i.e.* time for degradation of pesticide to 50% of its initial concentration. The findings of present study are in agreement with those where the half-life value of imidacloprid was observed 2.66 days in okra [10] and 2.31 days in brinjal fruits [14]. While earlier studies reported 1.04 and 1.13 days half-life ($t_{1/2}$) of imidacloprid at applied doses of 24.5g and 49g *a.i.* ha, respectively in okra [11]. 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 the 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 environmental contamination point of view.

4. Acknowledgement

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