"BEST POSTER AWARD IN 1st NATIONAL AGROCHEMICALS CONGRESS AT IARI, PUSA, NEW DELHI"

Best poster awarded to Mr. Milan N. Joshi, Dr. Suchi Chawla, Dr. Kaushik D. Parmar, Dr. Nitesh S. Litoriya, Dr. Ravi L. Kalasariya, Prof. Nirmal R. Chauhan, Mr. Mitesh R. Patel and Dr. Paresh G. Shah working in AINP on Pesticide Residues, ICAR, Unit-9, Anand Agricultural University, Anand has been awarded for "Best Poster Award" on "Dissipation and Dietary Risk Assessment of Imidacloprid and Spiromesifen in *Brassica* and Fruiting Vegetables Samples following Good Agricultural Practices" under Poster Session of the event in 1st National Agrochemicals Congress : Country's Status On Various Fronts of Agrochemicals Jointly Organized by Indian Council of Agricultural Research (ICAR), Indian Agricultural Research Institute (IARI), Society of Pesticide Science India (SPS), Association of Analytical Communities (AOAC India Section) and Crop Care Federation of India (CCFI) during November 13-16, 2019 at IARI, Pusa, New Delhi.

Dissipation and Dietary Risk Assessment of Imidacloprid and Spiromesifen in *Brassica* and Fruiting Vegetables Samples following Good Agricultural Practices

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1st NATIONAL AGROCHEMICALS CONGRESS

Country's Status on Various Fronts of Agrochemicals

November 13-16, 2019

Best Poster Award

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Certificate awarded to.....for the best poster presentation in the 1st National Agrochemicals Congress: Country's Status on Various Fronts of Agrochemicals organized during 13-16th November 2019 at ICAR-Indian Agricultural Research Institute, New Delhi.

1st National Agrochemicals Congress

(Anupama Singh)
President
Society of Pesticide Science India

(Supradip Saha)
General Secretary

Dissipation and dietary risk assessment of imidacloprid and spiromesifen in brassica and fruiting vegetables samples following good agricultural practices.

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Introduction

Food safety has become very important to study its impact on consumer health. Food safety is a part of food security. There is an increase in use of pesticides to feed the ever increasing human population. The injudicious use of pesticides to control pests may lead to accumulation of the residues in the crops which in turn may pose serious health hazards to human beings and risk to environmental ecosystems. Various measures are taken by national and international regulatory agencies to monitor the residues of pesticides in the crops and in food so as to control human exposure. Maximum residue limits (MRL) and acceptable daily intakes have been established for various pesticide-commodity combinations by codex, European Food Safety Authority (EFSA), Food Safety and Standards Authority of India (FSSAI). However, MRLs have not been established for many pesticide commodity combinations. The consumption of many vegetables is area specific. For e.g. bitter gourd is very commonly consumed in India but not in many other countries. As new and new molecules are coming up in the market individually or in combination with their dissipation and risk assessment need to be conducted regularly.

Objectives

- To study the dissipation pattern of both insecticides in different vegetable crops followed by Good Agricultural Practices (GAP)
- To establish the maximum residue limits on different fruiting vegetables
- To establish the safe waiting period and pre-harvest interval
- To find out acceptable daily intake for health
- To study the dietary risk assessment.

Materials and Methods

All field experiment was conducted at Main Vegetable Research Station, Anand Agricultural University, Anand during different interval from August, 2015 to February, 2016 and June, 2016 to February, 2017. The experiments were conducted in a randomized block design using three replicates for each treatment. At the time of fruiting, head and curd formation two foliar sprays were applied for both imidacloprid (Confidor 17.8 SL) and spiromesifen (Oberon 22.9 SL) at standard and double dose of 20 and 40, 96 and 192 g a. i. ha^{-1} respectively at an interval of 10 days on different vegetable crops using Knapsack sprayer, respectively. Samples in the form of fruits, heads and curds were drawn at 0 (1 h), 1, 3, 5, 7, 10 and 15 days after the last applications. The samples were brought to the laboratory and analysed immediately using QuEChERS method

GLC Parameters:

GLC Model:	Varian 450 GC equipped with ECD
Column:	TG-5 SILMS (30 m x 0.25 mm i.d. x 0.25 µm film thickness)
Oven Programming:	250 °C initially for 1.0 min programmed at 9 °C/min up to 295 °C for 1 min
Column Flow (N_2):	1 mL min^{-1}
Injector Temperature:	235 °C
Detector Temperature:	300 °C
Makeup gas (N_2) flow:	15 mL min^{-1}

LC Parameters:

Analysis of imidacloprid and spiromesifen (in some samples) were performed on ACQUITY UPLC, Waters, Milford, USA, coupled to a triple quadrupole mass spectrometer (API 3200, AB Sciex, USA), in electrospray ionization mode. C^{18}Reprosil Gold XBD with a pore size of 1.8 µm and dimensions of 100 mm×2.1 mm was used for separation of analytes. The mobile phase constituted 5 mM ammonium formate in water as solvent A and acetonitrile/methanol (50:50 v/v) as solvent B. The gradient was run to get separation. The gradientstartedwith65%Aand35%B.to 90%AandI0%B in 1.0 min, 90% A and 10% B in 4 min, returning to 65% A and 35% B in 5 min. The flow rate was 0.2 mL min^{-1} with a column temperature of 35°C, and the sample temperature was kept at 5 °C.

Figure 1: Chromatogram of Imidacloprid and Spiromesifen of LC-MS/MS.

Method of Analysis

Extraction:

- Take 15 ± 0.1 g of representative sample from the homogenized samples having water content > 80% in a clean 50 ml. capacity polypropylene tube
- Add 15 mL of 1% Acetic Acid in Acetonitrile (v/v) & shake well. **Note**. Place the tube in deep freezer (-10 °C) for 20 minutes, if required
- Add 6.0 g $MgSO_4$ (anhydrous) and 1.5 g Sodium Acetate (anhydrous)
- Shake vigorously for 1.0 min followed by vortex mixing & Centrifuge for 2.0 min at 3500 rpm

Cleanup:

- Transfer 6.0 mL (equivalent to 6 g sample) supernatant to 15 mL polypropylene centrifuge tube containing 900 mg PSA + 900 mg $MgSO_4$
- Vortex the sample for 30 sec & Centrifuge for 2.0 min at 2500 rpm to get clean supernatant
- Take 2 mL supernatant (equivalent to 2 g sample) and evaporate to dryness on TurboVap at 45 °C, make up to 2.0 mL with petroleum spirit (40:60°C)/ acetone (3:1 v/v) for estimation on GC using FPD/PFPD/ECD and confirmation on GC-MS/MS or LC-MS/MS

NB: *For LC-MS/MS analysis, the samples were diluted Tii times with mobile phase (80 parts organic: 20 parts aqueous containing 5 mM ammonium formate) (HPLC grade solvents) and filtered through 0.22 µm PFDF filters,* 250 °C initially for 1.0 min programmed at 9 °C/min upto 295 °C for 4 min

Results

- ✓ Recovery of both imidacloprid and spiromesifen from different samples was within the acceptable range of 70-120% with relative standard deviation (RSD) ≤20%.
- ✓ Limit of quantitation (LOQ) were 0.05 mg kg^{-1} for both pesticides.
- ✓ In all fruiting vegetables residue of imidacloprid were below determination level (BDL) of 0.05 mg kg^{-1} on 1 day (24 hr) after application in okra and brinjal, while in other vegetables the residues were BDL at 3 to 7 days.
- ✓ On the other hand, residues of spiromesifen were below determination level (BDL) of 0.05 mg kg^{-1} on 10 to 20 days after second application.
- ✓ Half-life ranged from 1.37 to 3.85 for imidacloprid and 2.28 to 6.73 days for spiromesifen in different vegetables.
- ✓ Hazard Quotient (HQ) were ≤ 1 for both imidacloprid and spiromesifen.
- ✓ As no MRL is available for bitter gourd an MRL of 0.02 mg kg^{-1} can be proposed.

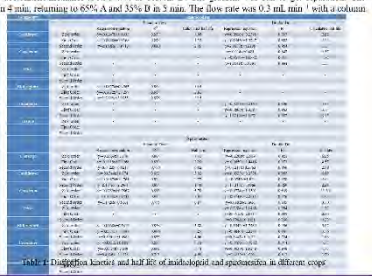

Table 1: Dissipation kinetics and half life of imidacloprid and spiromesifen in different crops

Future prospects

- ❖ As we know the both pesticide is safe for humans but effect of imidacloprid on honeybees are at alarming point so we can find new formulations.
- ❖ Help for studies the other agricultural commodity for Food Safety & Food Security.

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