

## DETERMINATION OF SPIROMESIFEN, QUINOLPHOS, MONOCROTOPHOS, CARBENDAZIM AND ACEPHATE RESIDUE BEHAVIOUR IN OKRA (*ABELMOSHUS ESCULENTUS*) BY LIQUID CHROMATOGRAPHY AND MASS SPECTROPHOTOMETRY AND THEIR DECONTAMINATION USING HOUSE HOLD PROCESSES

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### ABSTRACT

**Objective:** Field experiments were conducted at Agricultural University, Rajendra Nagar, Hyderabad, Telanagana to study the dissipation kinetics of carbendazim, monocrotophos, spiromesifen, acephate and quinalphos in okra fruit. Decontamination study was also conducted to evaluate quality of okra pods by reducing the residues of carbendazim, monocrotophos, spiromesifen, acephate and quinalphos by using different processes such as 2% salt solution, acetic acid, biowash, butter milk, cooking, drying, Formula 1(T<sub>7</sub>), frying, lemon water, sodium bicarbonate, tamarind water and tap water.

**Methods:** All the pesticide residues with one test dose at two spray i.e., first spray at flowering stage and second spray after an interval of ten days was carried out. The samples drawn at specific periods were analyzed by liquid chromatography and mass spectrophotometry (LC-MS/MS).

**Results:** The initial deposit of carbendazim, monocrotophos, spiromesifen, acephate and quinalphos in okra was found to be 2.239, 2.586, 2.401, 1.39, 0.78 mg/kg respectively. More than 98 % of carbendazim, spiromesifen, acephate and quinalphos dissipated after 15 d and monocrotophos was dissipated after 10 d. Sodium bicarbonate and 2% salt solution are the best methods for decontamination after cooking. The decontamination values of frying and formula 1 seems to be almost same. After these two methods biowash thought to be the method of choice.

**Conclusion:** These results are helpful in setting up maximum residual limit (MRLs) of these pesticides in okra in India. From the results, it could be recommended that cooking suits best for almost all of the pesticide residues.

**Keywords:** Okra, Biowash, Tamarind water, Dissipation, Decontamination, Pesticide residues

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### INTRODUCTION

Vegetables supply essential nutrients and vitamins and hence are an important constituent of human diet. Vegetables are often infested by certain insect pest such as Aphids, Thrips, Jassids, Fruit borer, Shoot borer and Leafhopper during their growth [1]. Though the use of Pesticides and Insecticides are recognized as important for food production, their usage might cause potential health risks such as chronic neurotoxicity, endocrine disruption, genotoxicity, mutagenicity and carcinogenesis through consumption of dietary residues [2]. High percentages of pesticide residue levels are present in fruits and vegetables when compared to other foods of plant origin.

The random utilize of pesticides especially at the mature stage and non-adoption of safe waiting time are the due reasons of accumulation of pesticide residues that leads to contamination of vegetables [3, 4]. Inappropriate use of pesticides may lead to public concern on food safety and human health [5-10], environmental contamination [11, 12], insect resistance and resurgence [12-14] though most of the pesticides are effective to control pests in Agriculture. The factors that may influence the dissipation behaviour of pesticides in plants, include the climatic conditions (temperature, humidity, light intensity, etc) [15], the type crop species [16-19] the nature of the chemicals, the formulations, and the application methods [20, 21].

Household processing methods, industrial-scale methods and food plants have been reported to affect, the pesticide level in foods. Many of these food processing techniques decrease residual quantities of pesticides, but some may not due to their higher concentration. Simple household processing techniques such as washing with water, soaking in various solutions, chemicals and cleaners are reported to be much effective in decreasing the level of pesticides [22-24]. For to decrease the levels of pesticide residues, thermal processing treatment such as pasteurization, blanching, boiling, cooking, steaming, canning etc. found to be effective [25-27].

Currently there is an increasing concern about the hazards of pesticides to consumers. Pesticide application pre or post-harvest, leave residues on food products that pose a potential health risk to the consumer. Farmers still believe in the control of pests using pesticide because of their even though they adopt Integrated Pest Management. Monitoring pesticide residues in agricultural commodities has to be given importance and also to standardize simple cost-effective methods which can be practiced by home makers to eliminate pesticide residues.

The uses of insecticides/pesticides such as carbendazim, monocrotophos, spiromesifen, acephate and quinalphos have increased in okra vegetable crop over the years and hence their monitoring is very important. Keeping in view the above facts, an attempt has been made in the present study to estimate the quantity of these residual pesticides in okra at different days of pre-harvest intervals and estimate the effect of traditional processing methods on the reduction of pesticide residues and evaluate their levels present in commercially produced okra. In addition, the study may develop/suggest effective methods through which pesticides can be reduced/removed or even decontaminated.

### MATERIALS AND METHODS

#### Dissipation and decontamination studies

The field experiments were conducted at Agricultural College, Rajendranagar, Hyderabad and Telangana, India. A 30 m<sup>2</sup> experimental plot was chosen and each treatment was carried out in triplicate in randomized block design. Okra crop that was grown through organic farming without pesticide spray serve as control. To investigate the dissipation of quinalphos, carbendazim, monocrotophos, spiromesifen, acephate with one test dose at two spray i.e., first spray at flowering stage and second spray after an interval of ten days was carried out. Okra fruits were collected randomly at 0,1,3,5,7,10 and 15 d after the last spray from all the

plots separately. 2 kg of okra fruits were collected from each plot and packed in poly bags to avoid contamination.

The sample collected at 0 d interval also used for decontamination studies for determining the effect of various processing techniques

(Treatment-1 to T-12) on the level of removal or reduction in pesticide residue contents. The details of all the treatments were listed in table 1. After about 2 h of the spray, okra was harvested and packed in polyethylene bags and brought to the laboratory for further processing.

**Table 1: Recommended dose of pesticides**

| Treatment | Common name of the insecticide | Dosage (g a. i ha <sup>-1</sup> ) |
|-----------|--------------------------------|-----------------------------------|
| 1         | Carbendazim 50% WP             | 80                                |
| 2         | Monocrotophos 36% SL           | 437 ml/ha                         |
| 3         | Acephate 75SP                  | 747g/ha                           |
| 4         | Spiromesifen 240SC             | 625 ml/ha                         |
| 5         | Quinalphos 25% EC              | 1250 ml/ha                        |

### Decontamination methods used for reducing pesticide residues

Okra fruit samples were collected at from various treatments in large quantities as twelve sets in four replicates. One set of the sample from each treatment was checked for initial deposits of the pesticide. From every treatment, the remaining 12 samples were analyzed after various decontamination methods separately. Different traditional processing techniques employed in the present study are simple, cost-effective, less cumbersome cleaning and processing methods. In order to reduce the impact of pesticide

residues, effective methods are to be employed to safeguard the health of consumers. The efficiency of these decontamination methods in reducing pesticide residues from the okra fruit samples was calculated. The lists of risk mitigation methods utilized in the present study are presented in table 2.

### Percent removal of pesticide

$$\text{Per cent removal} = \frac{\text{Initial deposit} - \text{Residues after treatment}}{\text{Initial deposit}} \times 100$$

**Table 2: The various decontamination methods employed in the present study**

|     |                                                                                                                                                                       |
|-----|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| T1  | Dipping in 2% salt solution for 10 min (80 grams of table salt was added to 4L of water, and 2 kg of okra dipped in saltwater for 1 h)                                |
| T2  | Dipping in 4% acetic acid solution for 1 min (160 ml of acetic acid was added to 4 lts of water; 2 kg okra samples dipped in the solution for 10 min)                 |
| T3  | Dipping in biowash for 10 min (biowash was added to 4 lts of water, and 2 kg okra samples is dipped in biowash for 10 min)                                            |
| T4  | Dipping in butter milk for 30 min (sour curd was added to 4 lts of water to prepare buttermilk and 2 kg okra samples is dipped in butter milk for 30 min)             |
| T5  | Cooking in pressure cooker (2 kg okra sample was cooked in pressure cooker for 10 min)                                                                                |
| T6  | Okra dried under sun to remove moisture and the powdered samples are used for analysis.                                                                               |
| T7  | Dipping in (4% acetic acid+0.1%NAHCO <sub>3</sub> +1lemon (1lemon/1lit): 160 ml of acetic acid, 4 gms of sodium bicarbonate, lemon juice of (Formula 1)               |
| T8  | 4 lemons added to 4 lts of water; 2 kg Okra samples dipping in the solution for 10 min)                                                                               |
| T9  | Okra fried in oil to remove moisture and the powdered samples are used for analysis                                                                                   |
| T10 | Dipping in lemon water (1Lemon/1lit) for 10 min (Juice of 4 lemons was added to 4 lts of water, and 2 kg okra samples is dipped in lemon water for 30 min)            |
| T11 | Dipping in 0.1% sodium bicarbonate solution for 10 min (4 grams of sodium bicarbonate was added to 4 lts of water; 2 kg Okra sample is dipped in solution for 10 min) |
| T12 | Dipping in 2% tamarind solution for 10 min (80 grams of tamarind was added to 4 lts of water, and 2 kg okra sample dipped in salt water for 30 min)                   |
|     | Tap water wash                                                                                                                                                        |

### Preparation of sample

#### Extraction of the sample

The modified QuEChERS extraction procedure was employed for the sample clean up step with the addition of GCB (Graphitised Carbon Black). Magnesium sulfate and PSA (Primary Secondary amine Sorbent) were added for removal of residual water, sugar and carbohydrates respectively. This procedure has been validated (in-house) to satisfy the European Union SANCO/12571/2013 guidelines [28].

A representative sample was homogenized and ~15.0 g of the sample has been transferred into a 50 ml tarson tube and appropriate amounts of multi pesticide mix standards were added. Subsequently, 30 ml of acetonitrile (v/v) was added and the mixture was homogenised with homogenizer and vortexed for 1 minute. Then 3.0 g of anhydrous sodium chloride was added and vortexed for 30 seconds and centrifuged at 2500 rpm for 10 min. The organic layer (~16 ml supernatant) was transferred to 50 ml of tarson tube supplemented with anhydrous sodium sulphate (9.0 g) and thoroughly shaken, vortexed for 1 min. The use of sodium sulphate was it will absorb the moisture. The supernatant (8 ml) obtained was transferred to a 15 ml tarson tube containing 1500 mg of magnesium sulphate and 400 mg of PSA and 7.5 mg of GCB. The mixture was vortexed and centrifuged for 10 min at 2500 rpm. An aliquot of 2 ml supernatant eventually was transferred to a Ria vial then evaporated at

45 °C in turbo evaporator. The residue was reconstituted with mobile phase (1 ml), filtered and injected into LC-MS/MS system.

The okra samples without any pesticide interference serves as blank for validation experiments and the same extraction procedure was employed for the samples that are treated with decontamination procedures.

### Reagents and chemicals

All the reagents employed in the present study analytical grade reagents were employed in this study. Analytical standard of pesticides (purity ≥ 99.8 %) was supplied from Sigma Aldrich, Germany, Ehrenstoffer (Japan), acetonitrile and glacial acetic acid (HPLC-grade) purchased from Merck (India). Methanol was purchased from J. T Baker, magnesium sulphate was purchased from Agilent, while sodium acetate purchased from Merck (India) and ammonium acetate was purchased from (Sigma). Formic acid was purchased from Merck (Xalostoc, Mexico). Primary and secondary amine sorbent (PSA) and Graphitised carbon block (GCB) purchased from Agilent, ultrapure water and HPLC water procured from Merck (Xalostoc, Mexico).

The stock solutions were prepared at 1000 ppm in an appropriate solvent, stored at -20±2 °C in a deep freezer. From the above stock solution the working standards were prepared.

## Instrumental conditions

### Chromatography and mass spectrometry

The chromatographic analysis was performed using an UHPLC (equipped with a Quaternary pump (LC20AD), autosampler (SIL20AC), Shim-pack XR-ODSIII column (75 × 2 mm, 1.6 μm) used for separation of the pesticide residues. 10 mM ammonium acetate in water (Mobile phase A) and 10 mmol ammonium acetate in methanol (Mobile phase B) at 0.4 ml/min flow rate was employed for separation of pesticide residues. The elution was programmed as follows: A (40)–B (60) (2 min), A (10)–B (90) (6 min), A (40%)–B (60%) (4 min) with a run time of 12 min and the volume injected was 5 μl. Triple Quadrupole Mass spectrophotometry (Lab ware solutions, Shimadzu, Japan) (Desolvation gas temperature: 250 °C; Heat block temperature: 300 °C; Drying gas flow–15 L/min; Nebulizer gas (N<sub>2</sub>) flow–2 L/min; Dwell time–10 msec<sup>-1</sup>; Interface voltage–4.5–5.0 kv) has been employed for the present study. The retention times, Multiple Reaction Monitoring (MRM) transitions of all the compounds used for the quantitative and qualitative estimation are presented in table 1. Linearity was calculated based on five different concentrations (10 ppb, 50ppb, 100ppb, 250ppb, 500 ppb) of pesticide residues and the recovery was validated by fortifying the untreated okra samples with standards of the mix of 5 compounds. The LOD (limit of detection) for 5 compounds was nearer to 0.005 mg/kg and the limit of quantification (LOQ) being nearer to 0.01 mg/kg.

### Method validation

#### Selectivity and linearity curves

The method selectivity was checked by blank injections. The method is set to be free of any target compounds as there was no signal

observed at retention times of the compounds. Matrix-matched calibration (MMC) was used to minimise the effect of the matrix. Analytical MMC curves were constructed using blank okra extracts with appropriate quantity of pesticide mix standard at different levels such as 10, 50, 100, 250, 500 μg/kg. The obtained data was analysed by Lab Solution Software.

#### LOD, LOQ and measurement of uncertainty

The LOD was the lowest concentration of analyte in a sample which can be detected and not quantified. The LOQ is the lowest spike level meeting the method performance criteria trueness and precision (70–120% and RSD ≤ 15%, respectively). Measurement uncertainty (MU) was assessed following SANCO/12571/2013 guidelines. LOD and LOQ values obtained are presented in table 1 [29–31].

#### Trueness and precision

The trueness of the method was determined based on the values obtained from the recovery assay. The samples were spiked at three different concentrations ie., 50.0, 250, 500 μg/kg (n=6 at each concentration) for three different days by two analysts and analytes recovery was calculated using true value and analysed value. The obtained data were used for determining the precision and measurement of uncertainty (MU). The precision was expressed as relative standard deviation (RSD) and is determined with replication data (n=6) of 3 different days at each concentration level and the results obtained are presented in table 3. The chromatogram showing the total ions (TIC) is given in fig. 1. To get the chromatogram, blank extracts of okra samples were fortified with all the pesticide mix at 500 ppb, the more intense MRM transition for each compound was picked up.

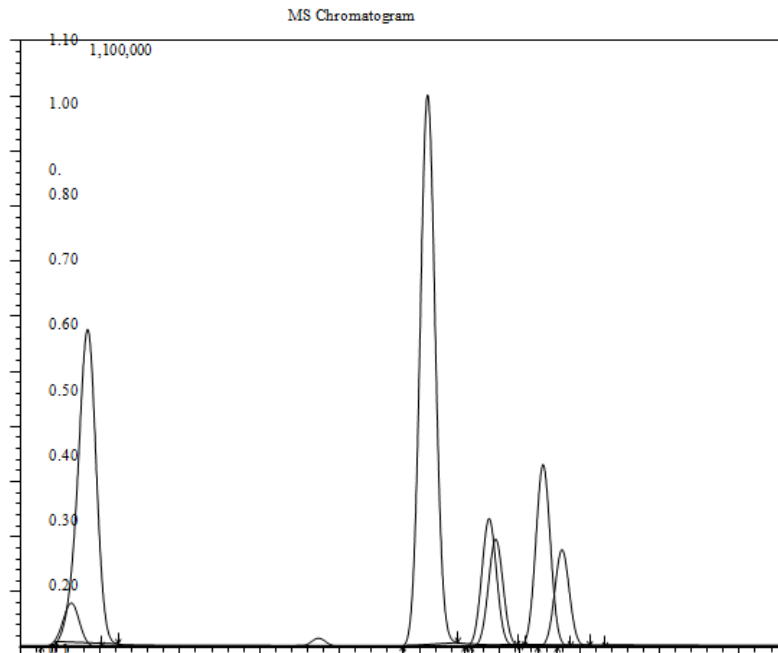


Fig. 1: Total ion chromatogram (TIC) obtained by LC-MS/MS (ESI positive mode) for blank okra samples fortified at 500 ppb

Table 3: Method validation parameters for each of the analyte

| Compound name | Correlation coefficient | Average recovery with %RSD <sup>c</sup> |              |              | Measurement of uncertainty % |          |          | LOD <sup>a</sup> | LOQ <sup>b</sup> |
|---------------|-------------------------|-----------------------------------------|--------------|--------------|------------------------------|----------|----------|------------------|------------------|
|               |                         | 50μg/kg                                 | 250μg/kg     | 500μg/kg     | 50μg/kg                      | 250μg/kg | 500μg/kg |                  |                  |
| Carbendazim   | 0.999                   | 107.77(6.57)                            | 98.56(4.65)  | 99.41(10.37) | 7.49                         | 6.46     | 9.74     | 2.55             | 8.50             |
| Monocrotophos | 0.995                   | 96.06(8.19)                             | 101.01(5.75) | 99.80(8.62)  | 8.49                         | 7.03     | 10.67    | 2.13             | 7.10             |
| Acephate      | 0.999                   | 83.62(2.87)                             | 87.30(6.87)  | 88.16(8.88)  | 5.73                         | 7.66     | 8.93     | 2.93             | 9.79             |
| Spiromesifen  | 0.996                   | 91.80(5.27)                             | 103.87(8.07) | 98.96(9.80)  | 6.77                         | 8.41     | 9.95     | 1.47             | 4.92             |
| Quinalphos    | 0.996                   | 89.56(6.67)                             | 88.67(8.59)  | 91.82(12.40) | 7.55                         | 8.74     | 11.39    | 2.25             | 7.52             |

<sup>c</sup>RSD%, percentage relative standard deviation (n=16); <sup>a</sup>LOD, Limit of detection; <sup>b</sup>LOQ, Limit of quantification

RESULTS AND DISCUSSION

Dissipation of carbendazim, monocrotophos, spiromesifen, acephate and quinalphos

Table 4: Dissipation of carbendazim

| DAY                    | R1             | R2    | R3    | Average | *SD   | %RSD <sup>b</sup> |
|------------------------|----------------|-------|-------|---------|-------|-------------------|
| 0 D                    | 2.010          | 2.685 | 2.021 | 2.239   | 0.386 | 17.263            |
| 1 D                    | 1.245          | 0.999 | 1.287 | 1.177   | 0.155 | 13.193            |
| 3 D                    | 0.916          | 0.821 | 0.901 | 0.880   | 0.051 | 5.827             |
| 5 D                    | 0.516          | 0.729 | 0.760 | 0.668   | 0.133 | 19.885            |
| 7 D                    | 0.515          | 0.536 | 0.544 | 0.532   | 0.015 | 2.809             |
| 10 D                   | 0.198          | 0.255 | 0.220 | 0.224   | 0.029 | 12.782            |
| 15 D                   | 0.097          | 0.103 | 0.092 | 0.097   | 0.005 | 5.425             |
| 20 D                   | *BDL           | BDL   | BDL   | BDL     | BDL   | BDL               |
| Regression equation    | Y=0.115x+1.506 |       |       |         |       |                   |
| Regression coefficient | 0.719          |       |       |         |       |                   |
| Half-life              | 3.544          |       |       |         |       |                   |
| Pre-Harvest interval   | 2.920          |       |       |         |       |                   |

\*BDL-Below Detection Level; \*SD, Standard deviation; <sup>b</sup>RSD%, percentage relative standard deviation (n=3)

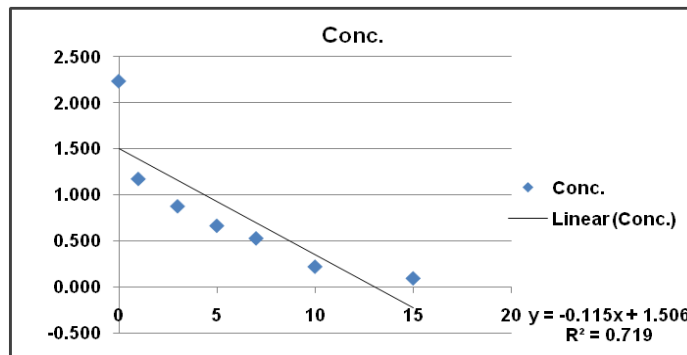


Fig. 2: Semi logarithmic graph showing dissipation pattern of carbendazim in Okra

Table 5: Dissipation of monocrotophos

| DAY                    | R1             | R2    | R3    | Average | *SD   | %RSD <sup>b</sup> |
|------------------------|----------------|-------|-------|---------|-------|-------------------|
| 0 D                    | 2.674          | 2.669 | 2.416 | 2.586   | 0.148 | 5.713             |
| 1 D                    | 1.593          | 1.565 | 2.126 | 1.761   | 0.317 | 17.979            |
| 3 D                    | 1.085          | 0.954 | 1.186 | 1.075   | 0.116 | 10.818            |
| 5 D                    | 0.449          | 0.475 | 0.427 | 0.450   | 0.024 | 5.276             |
| 7 D                    | 0.143          | 0.147 | 0.128 | 0.139   | 0.010 | 7.355             |
| 10 D                   | 0.047          | 0.037 | 0.047 | 0.044   | 0.005 | 12.563            |
| 15 D                   | 0.005          | 0.003 | 0.002 | 0.013   | 0.001 | 9.438             |
| Regression equation    | Y=0.246x+2.077 |       |       |         |       |                   |
| Regression coefficient | 0.857          |       |       |         |       |                   |
| Half life              | 1.718          |       |       |         |       |                   |
| Pre-Harvest interval   | 2.614          |       |       |         |       |                   |

\*SD, Standard deviation; <sup>b</sup>RSD%, percentage relative standard deviation (n=3)

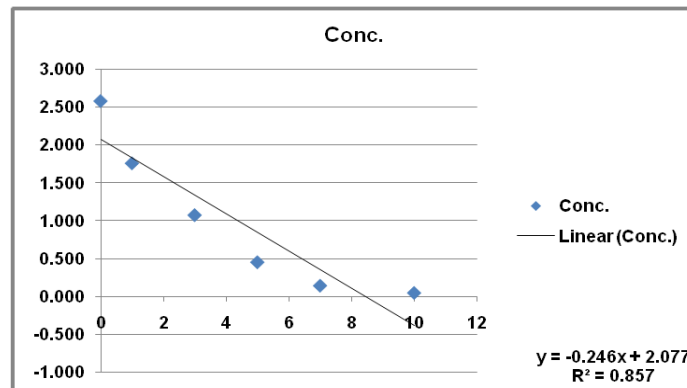


Fig. 3: Semi logarithmic graph showing dissipation pattern of monocrotophos in Okra

Table 6: Dissipation of spiromesifen

| DAY                    | R1             | R2    | R3    | Average | <sup>a</sup> SD | <sup>b</sup> RSD <sup>b</sup> |
|------------------------|----------------|-------|-------|---------|-----------------|-------------------------------|
| 0 D                    | 2.623          | 2.343 | 2.238 | 2.401   | 0.199           | 8.288                         |
| 1 D                    | 1.770          | 1.635 | 1.767 | 1.724   | 0.078           | 4.524                         |
| 3 D                    | 0.898          | 1.166 | 1.094 | 1.053   | 0.139           | 13.200                        |
| 5 D                    | 0.798          | 0.737 | 0.767 | 0.767   | 0.030           | 3.911                         |
| 7 D                    | 0.299          | 0.208 | 0.206 | 0.238   | 0.053           | 22.269                        |
| 10 D                   | 0.062          | 0.068 | 0.057 | 0.062   | 0.006           | 9.677                         |
| 15 D                   | 0.009          | 0.008 | 0.008 | 0.008   | 0.001           | 6.928                         |
| Regression equation    | Y=0.225x+2.016 |       |       |         |                 |                               |
| Regression coefficient | 0.904          |       |       |         |                 |                               |
| Half-life              | 1.943          |       |       |         |                 |                               |
| Pre-Harvest interval   | 2.927          |       |       |         |                 |                               |

<sup>a</sup>SD, Standard deviation; <sup>b</sup>RSD%, percentage relative standard deviation (n=3)

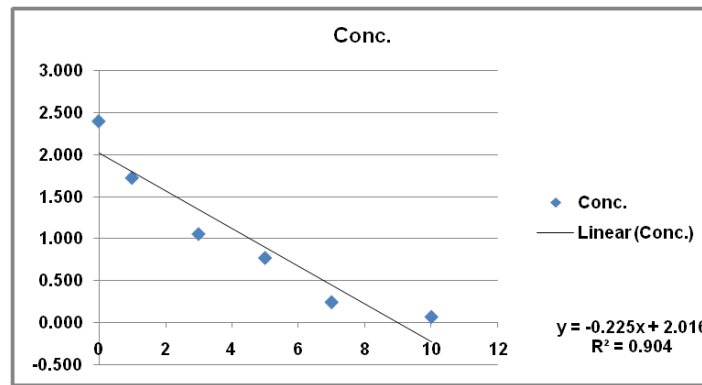


Fig. 4: Semi logarithmic graph showing dissipation pattern of spiromesifen in Okra

Table 7: Dissipation of acephate

| DAY                    | R1             | R2    | R3    | Average | <sup>a</sup> SD | <sup>b</sup> RSD <sup>b</sup> |
|------------------------|----------------|-------|-------|---------|-----------------|-------------------------------|
| 0 D                    | 1.351          | 1.340 | 1.502 | 1.398   | 0.091           | 6.475                         |
| 1 D                    | 1.210          | 1.211 | 1.165 | 1.195   | 0.026           | 2.207                         |
| 3 D                    | 1.009          | 1.073 | 0.836 | 0.973   | 0.122           | 12.594                        |
| 5 D                    | 0.791          | 0.806 | 0.836 | 0.811   | 0.023           | 2.827                         |
| 7 D                    | 0.599          | 0.597 | 0.564 | 0.587   | 0.020           | 3.375                         |
| 10 D                   | 0.138          | 0.139 | 0.121 | 0.133   | 0.010           | 7.839                         |
| 15 D                   | 0.015          | 0.013 | 0.012 | 0.013   | 0.001           | 9.438                         |
| Regression equation    | Y=0.118x+1.364 |       |       |         |                 |                               |
| Regression coefficient | 0.988          |       |       |         |                 |                               |
| Half-life              | 3.290          |       |       |         |                 |                               |
| Pre-Harvest interval   | 2.544          |       |       |         |                 |                               |

<sup>a</sup>SD, Standard deviation; <sup>b</sup>RSD%, percentage relative standard deviation (n=3)

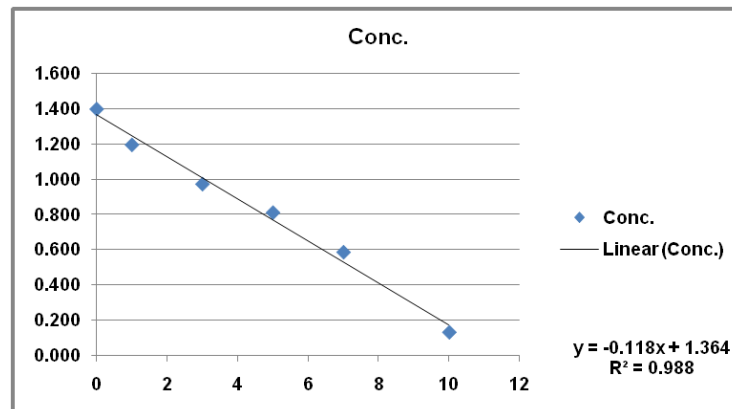


Fig. 5: Semi logarithmic graph showing dissipation pattern of acephate in Okra

Table 8: Dissipation of quinalphos

| DAY                    | R1             | R2    | R3    | Average | <sup>a</sup> SD | %RSD <sup>b</sup> |
|------------------------|----------------|-------|-------|---------|-----------------|-------------------|
| 0 D                    | 0.798          | 0.735 | 0.819 | 0.784   | 0.044           | 5.589             |
| 1 D                    | 0.628          | 0.660 | 0.763 | 0.684   | 0.071           | 10.375            |
| 3 D                    | 0.440          | 0.366 | 0.317 | 0.374   | 0.062           | 16.618            |
| 5 D                    | 0.126          | 0.127 | 0.084 | 0.112   | 0.024           | 21.725            |
| 7 D                    | 0.080          | 0.086 | 0.076 | 0.081   | 0.006           | 6.831             |
| 10 D                   | 0.058          | 0.062 | 0.076 | 0.065   | 0.009           | 13.952            |
| Regression equation    | Y=0.077x+0.685 |       |       |         |                 |                   |
| Regression coefficient | 0.835          |       |       |         |                 |                   |
| Half-life              | 2.471          |       |       |         |                 |                   |
| Pre-Harvest interval   | 1.039          |       |       |         |                 |                   |

<sup>a</sup>SD, Standard deviation; <sup>b</sup>RSD%, percentage relative standard deviation (n=3)

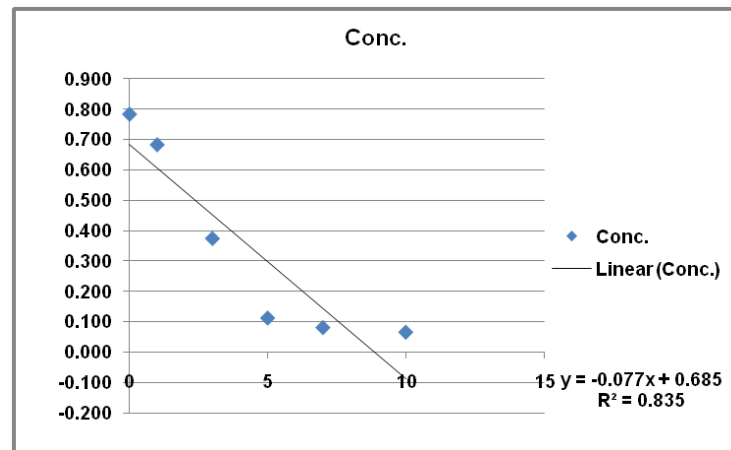


Fig. 6: Semi logarithmic graph showing dissipation pattern of quinalphos in okra

#### Dissipation of carbendazim in okra fruit

The initial deposit of Carbendazim in okra fruits grown in the field was 2.239 mg/kg with a half-life ( $t_{1/2}$ ) of 3.544 d. More than 98 % of this residue had dissipated after 15 d of spraying (table 4 and fig. 2). The various factors that may influence pesticide persistence are climate, physical and chemical properties of pesticide [6]. MRLs have not yet been set by FAO/WHO, US for carbendazim in okra. Maximum residue limits for carbendazim have been set in tomato in the range of 0.5 mg per kg and cucumber 0.05 mg per kg body weight.

#### Dissipation of monocrotophos in okra fruit

The initial deposit of monocrotophos in okra fruits grown in the field was 2.586 mg/kg with a half-life ( $t_{1/2}$ ) of 1.718 d. More than 98 % of this residue had dissipated after 10 d of spraying (table 5 and fig. 3). The various factors that may influence pesticide persistence are climate, physical and chemical properties of pesticide [6]. MRLs (maximum residue limits) have not yet been set by CODEX ALIMENTARIUS [9] for monocrotophos in okra. Maximum residue limits for monocrotophos have been set in some crops in the range of 0.02-1 mg/kg and the acceptable daily take is 0.0006 mg/kg body weight.

#### Dissipation of spiromesifen in okra fruit

The initial deposit of spiromesifen in okra fruits grown in the field was 2.401 mg/kg with a half-life ( $t_{1/2}$ ) of 1.94 d. More than 98 % of this residue had dissipated after 15 d of spraying (table 6 and fig. 4).

#### Dissipation of acephate in okra fruit

The initial deposit of acephate in okra fruits grown in the field was 1.39 mg/kg with a half-life ( $t_{1/2}$ ) of 3.29 d. More than 98 % of this residue had dissipated after 15 d of spraying (table 7 and fig. 5). The various factors that may influence pesticide persistence are climate, physical and chemical properties of pesticide [6]. MRLs (maximum residue limits) have not yet been set by FAO/WHO, US [9] for

acephate in okra. Maximum residue limits for acephate have been set in cabbage and tomato as 2 mg and 1 mg per kg body weight.

#### Dissipation of quinalphos in okra fruit

The initial deposit of quinalphos in okra fruits grown in the field was 0.78 mg/kg with a half-life ( $t_{1/2}$ ) of 2.471 d. 100 % of this residue had dissipated after 15 d of spraying (table 8 and fig. 6). The various factors that may influence pesticide persistence are climate, physical and chemical properties of pesticide [6]. MRLs (maximum residue limits) have not yet been set by FAO/WHO, US [9] for quinalphos in okra.

The results of the decontamination studies were presented in table 9. The results were shown by applying the weight loss effect. Carbendazim residues were reduced upto 75.2% by cooking (T5) and 61.4% by sodium bicarbonate treatment (T10). The residues of acephate were reduced by 65% with cooking. Monocrotophos was reduced by 68.2% by cooking. The residues of quinalphos were reduced by 76.2% by cooking and 68.5% by sodium bicarbonate washing. According to Nagesh and Verma (1997), decontamination through different processes showed that the residues in cabbage were reduced to some extent by various home processing methods like washing and cooking [32]. Cooking did not help much in reducing the residue below the MRLs of 0.25 for quinalphos. Spiromesifen was reduced effectively by 76% with cooking. Among all the different decontamination methods employed, cooking suits best for almost all of the pesticide residues. Sodium bicarbonate and 2% salt solution are the best methods for decontamination after cooking. The decontamination values of Frying and formula 1(T7) seems to be almost the same. After these two methods, bio wash thought to be the method of choice. Nath et al. reported that washing of treated okra with tap water resulted in considerable removal of malathion deposits by 89.15% [33]. Washing decreased the carbaryl deposit by 69.55%. Cypermethrin residues reduced in tomato, okra, bottle gourd and ridge gourd after all processing steps i.e. about 5-14% by washing, 6-26% by blanching, 6-19% by washing in brine solution and 15-33% by cooking Kadian et al., 2001[34].



Table 9: Percent removal of pesticide residues

| Decontamination methods | Carbendazim | Monocrotophos | Acephate | Spiromesifen | Quinalphos |
|-------------------------|-------------|---------------|----------|--------------|------------|
| 2% salt solution        | 51.36       | 55.25         | 54.24    | 58.24        | 56.28      |
| Acetic acid             | 39.25       | 14.24         | 31.25    | 21.35        | 26.54      |
| Biowash                 | 49.21       | 60.34         | 26.54    | 33.26        | 55.21      |
| Butter milk             | 21.24       | 37.24         | 35.24    | 51.24        | 30.31      |
| Cooking                 | 75.24       | 68.21         | 65.21    | 78.24        | 76.21      |
| Drying                  | 36.24       | 31.25         | 38.51    | 41.26        | 39.24      |
| Formula1(T7)            | 54.26       | 24.21         | 45.24    | 39.21        | 16.54      |
| Frying                  | 48.21       | 61.85         | 4.56     | 56.41        | 49.24      |
| Lemon water             | 20.56       | 5.46          | 19.21    | 24.21        | 21.54      |
| Sodium bicarbonate      | 61.44       | 42.24         | 35.86    | 52.44        | 68.51      |
| Tamarind water          | 42.54       | 25.18         | 41.54    | 14.54        | 54.26      |
| Tap Water               | 26.51       | 8.56          | 21.58    | 25.46        | 17.24      |

## CONCLUSION

The initial deposit of carbendazim, monocrotophos, spiromesifen, acephate and quinalphos in okra was found to be 2.239, 2.586, 2.401, 1.39, 0.78 mg/kg, respectively. More than 98 % of caebendizam, spirofesifin, acephate and quinolphos dissipated after 15 d and monocrotophos was dissipated after 10 d. These results are helpful in setting up maximum residual limit (MRLs) of these pesticides in okra in India. Based on the results, it can also be concluded that the pesticide residues such as carbendazim, monocrotophos, spiromesifen, acephate and quinalphos were removed by almost all the decontamination methods employed in the study. Among the various decontamination methods employed almost all the pesticide residues considered for the study were removed effectively from okra fruit with simple household processing methods such as cooking, frying, 2 % salt solution and Biowash. Among all methods, cooking in a pressure cooker proved to be the best, and also economical. Employing Acetic acid, buttermilk, tamarind water was also found to be best methods of choice at household levels to remove pesticide residues. The results can be propagated and popularized among homemakers for removal of pesticides from okra fruits.

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Nil

## AUTHORS CONTRIBUTIONS

All the authors have contributed equally.

## CONFLICT OF INTERESTS

Declared none

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