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Article

Residue dissipation kinetics, safety evaluation and decontamination of Deltamethrin in tomato and the soil

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Abstract: Tomato powdery mildew, fruit rot, and twig blight are all managed with Deltamethrin. Its residues could still be present in the crops, posing a health risk. The pesticide residue analysis, dissipation rate, and safety assessments were thus examined in green tomatoes. The analytical method for residue analysis was validated according to international standards. Tomato fruits and soil were used to study the dissipation of Deltamethrin 100 EC (11% w/w) at 12.5 g a.i ha^{-1} for the recommended dose (RD) and 25.0 g a.i ha^{-1} for the double of the recommended dose (DD). Ethyl acetate was used to extract residues from tomato fruit, and PSA and magnesium sulphate were used for cleanup. The fruits had recoveries ranging from 83% to 93% and the soil sample from 81.67% to 89.6%, with the limit of detection (LOQ) estimated at 0.01 mg kg^{-1}. The matrix effect (ME) was calculated to be less than 20% for the tomato fruits and the soil. Half-lives for RD and DD were 1.95 and 1.84 days, respectively. All sampling days for both doses had dietary exposures of residues below the maximum permissible intake (MPI) of 0.16 mg $person^{-1}$ day^{-1}. The most effective method of decontaminating tomato residue containing Deltamethrin is blanching.

Keywords: dissipation kinetics; decontamination; safety; Deltamethrin; tomato

1. Introduction

The world's second-most important vegetable crop is the tomato (*Lycopersicon esculentum*), which is regarded as a protective food due to its distinctive nutritional content and extensive production. It is considered to be an important commercial and dietary vegetable crop. Along with potassium, iron, and phosphorus, it is rich in vitamins A, B, and C. Additionally, lycopene is abundant in it [1]. The Indian culinary culture makes extensive use of it since it has been used in salads, a variety of culinary preparations, juices, or processed into purees, concentrates, condiments, and sauces. Andhra Pradesh, Madhya Pradesh, Uttar Pradesh, Karnataka, Odisha, Bihar, and Assam are the main tomato-producing states in India [2]. India ranked second in the world with 18.4 million tonnes of tomato production [3]. According to the estimates for 2017–2018, India has 789 000 hectares of land cultivated with tomatoes, with a production of 19.7 million tonnes and a productivity of 25.0 t/ha per hectare, respectively. Also, India's share of world tomato production is 10.4 percent [4].

The suitable weather for germination and growth is 20 to 25 °C. The fruit is fragile and tender; thus, insect pest damage is one of the severe problems that negatively influence tomato yields in India [5]. Along with other factors contributing to low productivity, fruit borer, which results in a 22%–38% yield loss in marketable fruit yield, also causes substantial damage [6]. One of the most significant pests in India is fruit borer, which affects tomato production and market value. The most detrimental pest to tomatoes in India is the fruit borer.

Helicoverpa armigera (Hubner), also known as the American bollworm, gram pod borer, and fruit borer [7]. To combat the productivity losses brought on by pest infestations, tomato producers regularly employed a variety of pesticides. The use of pesticides increases the likelihood that hazardous residues will remain in tomatoes while also protecting against production reductions. Using microbiological pesticides [8], due to their generally higher level of safety, environmental friendliness, and reduced susceptibility to resistance, research into plant products and insecticides is being conducted [9,10]. In this situation, it is essential to utilize new generation pesticides because they are less environmentally harmful and more selective [11,12]. According to the estimates for 2017–2018, India has 789,000 hectares of land cultivated with tomatoes, with a production of 19.7 million tonnes and a productivity of 25.0 t/ha per hectare, respectively. An artificial pyrethroid insecticide with a high degree of action against a wide range of insects is Deltamethrin. According to studies, Deltamethrin effectively suppressed *Helicoverpa armigera*, the tomato fruit borer, increasing tomato yields [13]. As a result, pesticides are consumed in huge quantities, leaving hazardous residues behind [14,15]. However, its residue might still be present in the soil and crops. Tomatoes being the most important staple vegetable in our households, it becomes essential to analyze their residual pesticide status. The present study was thus conducted because there was no prior assessment of Deltamethrin in tomatoes through GC-μECD analysis.

2. Materials and methods

2.1. Chemical and reagent

We bought glacial acetic acid (Analytical Reagent grade), ethyl acetate, and anhydrous sodium sulphate (Na_2SO_4) from Thomas Baker in Mumbai, India. HPLC grade water was obtained from the Sartorius water purification system (Sartorius AG, Gottingen, Germany). Graphite Carbon Black (GCB), magnesium sulfate, and primary secondary amine (PSA, 75 mg) were purchased from Agilent Technologies in Bangalore, India.

2.2. Apparatus

The following equipment was used in the experiment: centrifuge (Kubota, Germany); microcentrifuge (Eppendorf refrigerated centrifuge, Chennai, Tamil Nadu); mixer and grinder (Bajaj India Pvt. Ltd., Mumbai); precision balance (Vibra, Adair Dutt, and Mumbai, India); vortex mixer (Geni 2T, Imperials Biomedicals, Mumbai, India); and ultrasonic bath (Oscar electronics, Mumbai, India).

2.3. Reference standard

The Dr. Ehrenstorfer GmbH (Augsburg, Germany) supplied the certified reference standards of Deltamethrin with 99.0% purity. 10 ± 0.1 mg of standard reference was dissolved in 10 mL of ethyl acetate, yielding a final concentration of

1000 μg mL^{-1}. The calibration standard solutions at 10, 20, 40, 60, 80, and 100 μg mL^{-1} were prepared from the working standard mixture of 10 μg mL^{-1} that was created by appropriately mixing the individual standard stock solutions and further dilution. The same concentration of matrix standards was prepared using the control of tomato extract that was collected during the sample preparation procedure, in accordance with the description provided regarding sample preparation and analysis.

2.4. Field experiment

Field experiments were performed during the month of January 2023 at ICAR—Indian Institute of Vegetable Research (Varanasi, Uttar Pradesh, India; longitude: 82.52° E and latitude 25.10° N) as per the Food and Agriculture Organization (FAO) guidelines. Three treatments were used in the experiment, each of which was replicated three times, using a randomized block design. The tomato (var. Kashi Aman) was cultivated using the methodical and advised cultivation techniques. Deltamethrin (Decis® 11% w/w EC, Aristo Bio-tech and Life-Science Limited, India) was implemented at the recommended dose (12.5 g a.i. ha^{-1}) and double the recommended dose (25.0 g a.i. $ha^{-1)}$ in tomato [16]. During the initial phase of fruit development, three gusher spray treatments were administered at seven-day intervals. Similarly, in the double dose and recommended dose plots, water was sprayed in the control plot to compare. Insecticides were sprayed in the mid-afternoon when the weather was sunny and windy. During the experimental intervening period the average minimum and maximum temperatures were 18 ℃ and 25 ℃ respectively with an average relative humidity between 50%–85% and during the study no rainfall was observed.

2.5. Sampling

Tomato fruit samples were randomly collected from each replicate after two hours of spraying from the treated and control plots independently following the final spraying, which was completed at regular intervals of 0, 1, 3, 5, 7, 10, and 12 days. The samples were gathered in polythene bags and kept at −4 ℃ until inspection in order to prevent sample deterioration. A random soil sample was taken at a depth of 0 to 15 cm from each plot using a soil auger. The soil samples were air dried and sieved prior to the residue being removed.

2.6. Sample preparation

To facilitate future research, the entire tomato sample for each replica was split into four subsamples, thoroughly mixed, and then arbitrarily chosen. All of the samples were extracted with only minor modifications using the provided methods [17]. All the tomato samples were crushed extensively in a blender without any addition of distilled water. The extraction of 10 g sample occurred with 10 mL of 1% glacial acetic acid in ethyl acetate in the presence of 10 g anhydrous sodium sulphate then vortexing it for 2 min and successionally centrifugation at 5000 rpm for 5 min. An aliquot of the supernatant ethyl acetate layer (1.5 mL) was cleaned by dispersive solid-phase extraction followed by 75 mg PSA, 225 mg $MgSO_4$, and 15 mg of Graphite Carbon Black (GCB). Since tomato possess lycopene abundantly, it's difficult to separate it from the matrix. Thus, extract was cleaned using several combinations of GCB like 8 mg, 12 mg and 15 mg. Satisfactory results were obtained at 15 mg concentration only. Centrifugation of the extract at 5000 rpm for 5min applied, following passed through a 0.2 µm Nylon 6, 6 membrane filter (**Figure 1**). Soil samples were extracted using the same procedure as tomato fruits, with the exception of adding 5 mL of water to 10 g of samples before adding the extracting solvent, ethyl acetate.

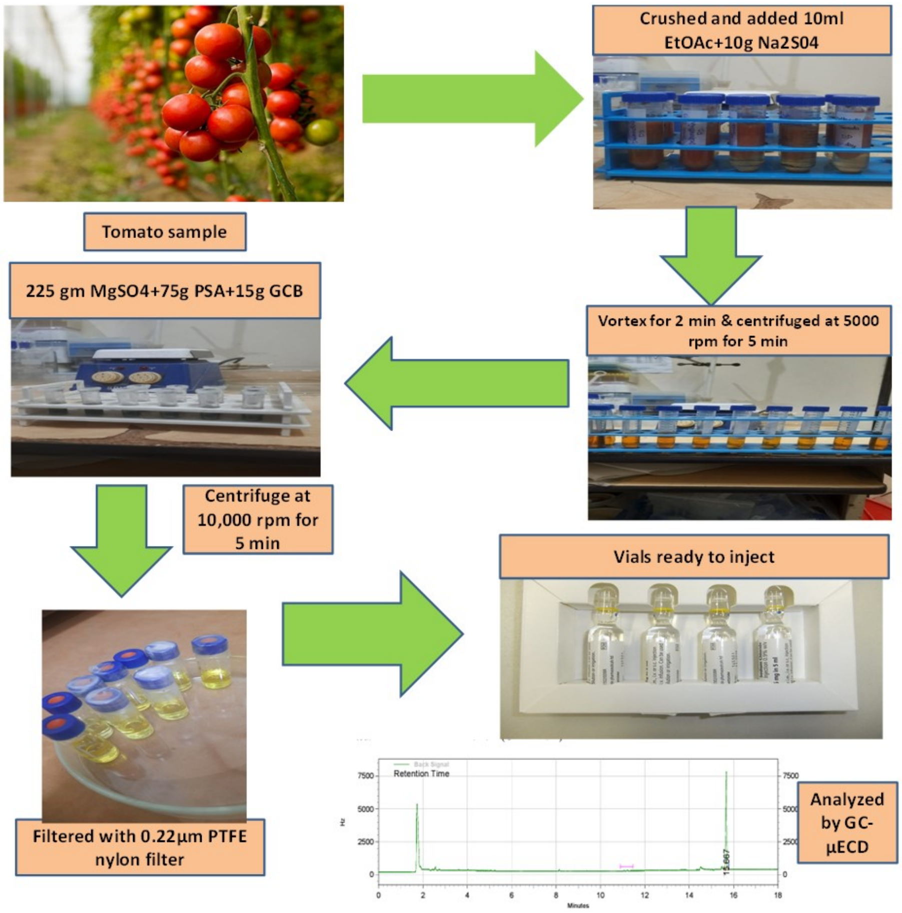

Figure 1. Diagrammatic representation of extraction and cleanup procedure.

3. GC-µECD analysis

Deltamethrin was detected using a gas chromatography system from Agilent (Model 7890B) with an autosampler and electron capture detector (µECD, ^{63}Ni). In the split injection mode, a standard syringe split/split less injector was used at a ratio of 10:1 at 250 °C with a volume of 1 µL. A capillary column HP-5 (30 m length, 320 µm id, 0.25 µm) film with nitrogen gas flowing at 1.5 mL/min thickness was used at a pressure of 9.22 psi for separation. In this experiment, a 300 °C detector was maintained with makeup gas i.e. (N_2) flowed at 30 mL/min. The oven temperature was set to 120 °C for 2 min which was then ramped to 300 °C at 15 °C/min and retained for 4 min. It was found that Deltamethrin exhibited a retention time (RT) of 15.667 min in these conditions (**Figure 2**). It took around 18 min to run the program. In order to detect and quantify insecticide residues, 1 µL liter of clean extract was injected in GC-ECD in split injection mode with a split ratio of 10:1. Comparing the peak areas of the standards with those of the unknown or spiked samples was done under identical experimental conditions in order to determine the residues of insecticide. The following formula was used to compute insecticide residue in mg kg^{-1}:

$$\text{Residue (mg kg}^{-1}\text{)} = (A_1 \times V_1 \times C)/(A_2 \times V_2 \times W)$$

where, A_1 = area of the chromatogram's field sample, A_2 = area of the chromatogram's analytical standard, V_1 = total sample volume measured in mL, V_2 = injected volume in µL, C = analytical standard concentration in mg kg^{-1}, and W = weight of the sample in gm.

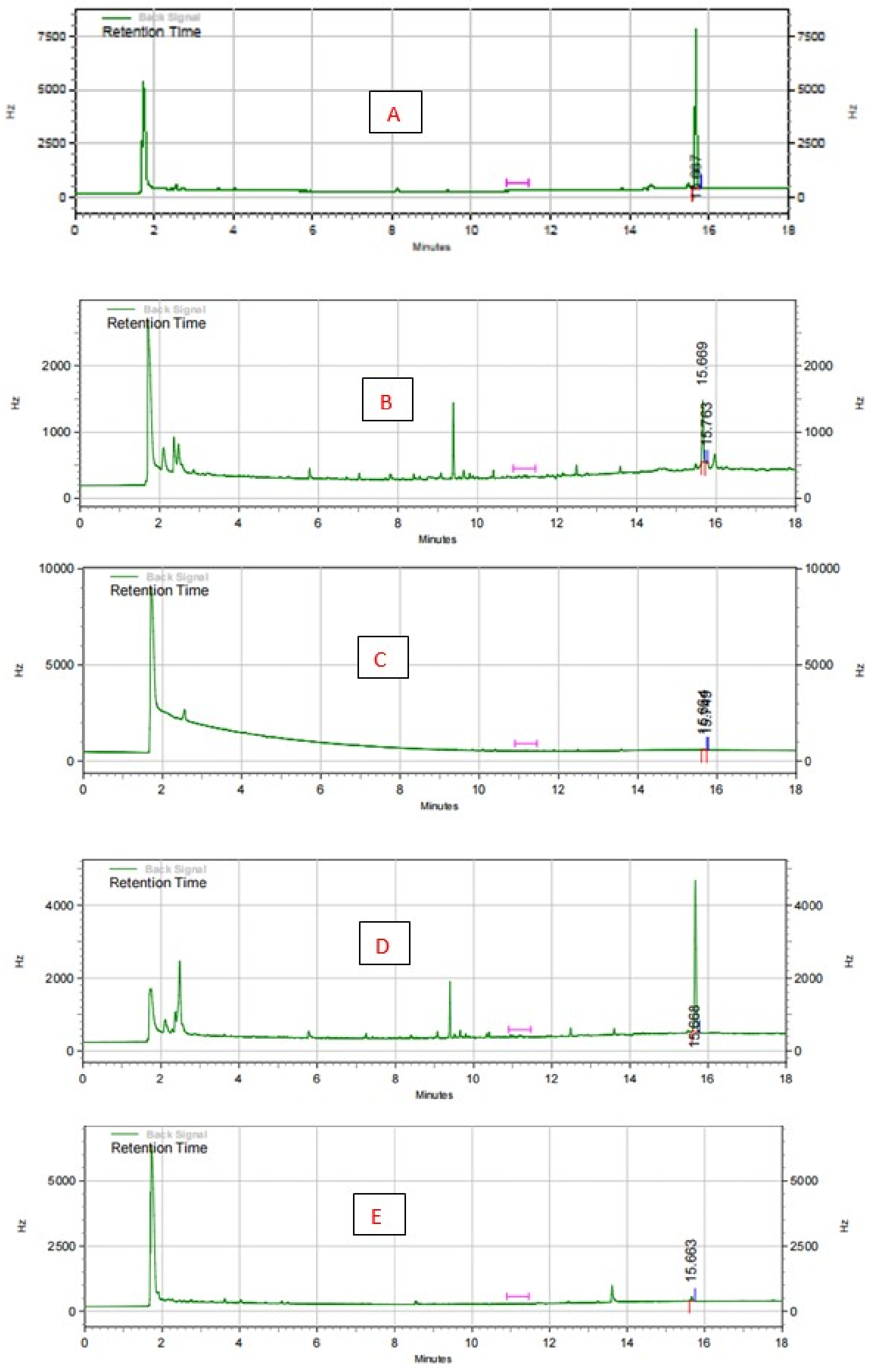

Figure 2. Chromatogram for **(A)** Deltamethrin standard **(B)** tomato sample **(C)** control sample **(D)** matrix matched standard for the soil **(E)** soil sample.

3.1. Method validation

The linearity, quantification limit, matrix effect, accuracy, and recovery of a single laboratory method were all validated in accordance with European Union SANTE/12682/2019 guidelines [18].

3.2. Calibration curves and linearity

By producing six-point calibration curves using calibration standards in the range of 0.01–0.1 µg mL^{-1} accomplished in solvent, i.e., ethyl acetate, and in matrix (control) extract for Deltamethrin, the linear response with regard to concentration gradient was evaluated. Plotting the peak area response versus concentration yielded an accurate calibration curve.

3.3. Limit of quantification and recovery

The smallest measured quantity in the tomato matrix was determined as the limit of quantification (LOQ), respectively, at which the signal to noise ratio (S/N) was 3:1 and 10:1. Calibration linearity (solvent-based standard and matrix-matched standard) was observed within the range of 0.01–0.1 µg mL^{-1}. Recovery studies were conducted with six replicates at concentrations of 0.01, 0.02, 0.05, 0.1 and 0.5 µg mL^{-1} (**Figure 3**).

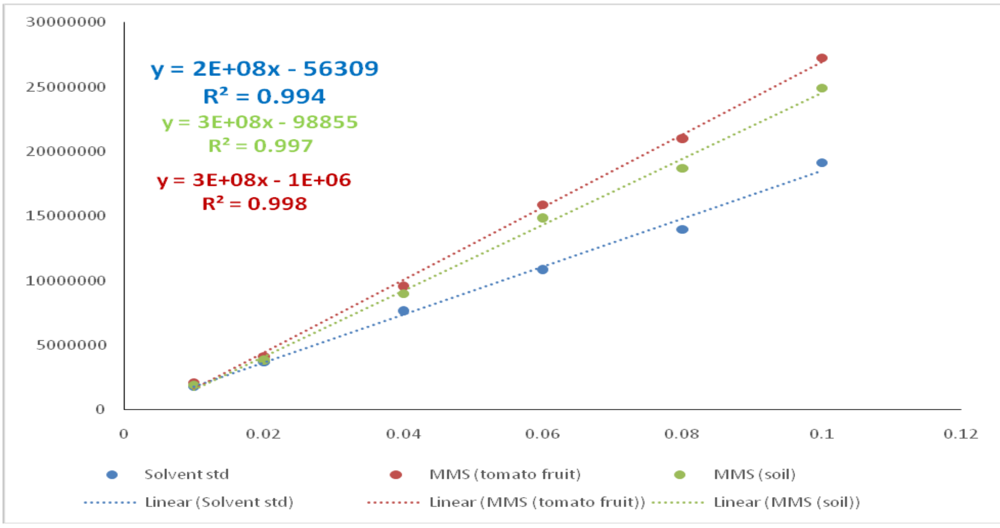

Figure 3. Calibration curve of Deltamethrin in tomato fruits, soil and solvent.

3.4. Matrix effect

The Matrix Effects (ME) was assessed by contrasting the solvent of choice's height vicinity reaction with the matrix-matched standard at 0.1 and 0.01 µg mL^{-1}. Calculation of the matrix effect was done using the equation:

$$ME\ (\%) = (\text{peak area of matrix matched standard} - \text{peak area of solvent standard})/\text{peak area of matrix matched standard} \times 100\%$$

The ME values above 100% indicated induced signal enhancement while, underneath 100% precipitated signal suppressions.

3.5. Dissipation kinetics

Dissipation of Deltamethrin in tomato turned into studied with the aid of subjecting the records to first-order kinetic Equation (1).

$$C_t = C_0 \, e^{-kt} \quad (1)$$

where, C_t is the concentration at time t, C_0 is the initial concentration, k is the rate constant for insecticide dissipation, and t is the time [19].

For calculating half-life ($t_{1/2}$) of the determine compounds, the residue statistics were subjected to statistical analysis as in line with the following Equation (2).

$$t_{1/2} = \ln 2/k \quad (2)$$

3.6. Consumer risk assessment

The food safety of Deltamethrin was evaluated by analysing the dietary exposure TMDI i.e. (Theoretical maximum daily intake) to determine if it is within the Maximum Permissible Intake (MPI). The MPIs were derived by multiplying the ADI by the bodyweight of an average child (16 kg). The ADI of Deltamethrin was 0.01 mg kg^{-1} bodyweight day. The MPI of Deltamethrin was estimated at 0.16 mg $person^{-1}$ day^{-1}. Dietary exposures were calculated by taking into account the residue levels in each sample (mg kg^{-1}) and an average per capita consumption of 0.0179 and 0.025 kg day^{-1} in case of urban and rural people respectively.

3.7. Decontamination of Deltamethrin residues from tomato

The removal or decontamination of the resultant residue impact is impacted by a number of household practices, including boiling water cleaning (blanching), 1% NaCl solution cleaning, tap water, hot water (50 ℃), and 10-minute ultrasonic treatment. Deltamethrin was evaluated in field-sprayed tomato samples harvested on 0 day after spraying. We extracted and analyzed the treated samples by GC-ECD and compared the results with each other to estimate the degree of decontamination. To estimate the degree of decontamination, the residue in the untreated sample was taken as 100% and added to the residue left after the decontamination process [20].

4. Results and discussion

4.1. Sample preparation

Additional extraction was done after the tomato samples were crushed. There was no need to add water for a smooth grinding process. The extraction procedure included only 5 mL of water. With only 75 mg of PSA or without cleanup, Deltamethrin displayed higher matrix induced signal enhancement when ethyl acetate extract of Tomato was used. Cleanup with 75 mg PSA may reduce the matrix effect to less than 25%. Therefore, purification of the 1.5 mL ethyl acetate extract was performed using only 75 mg PSA. A comparable process of preparation was used for the green chili matrices [21].

4.2. Method validation

The coefficient of determination (R^2) was 0.9485 and 0.9823 for RD and DD respectively all tested analytes within the calibration range of 0.1 and 0.01 μg mL^{-1}

for both solvent and matrix-matched standards. The LOQ of Deltamethrin was found 0.01 mg kg^{-1}. Mean recoveries of Deltamethrin at 10, 20, 50, 100 and 500 µg kg^{-1} were greater than 80% (**Table 1**). Matrix-matched calibration was used for accurate quantification, as matrix effects are reflected less than 20% in both the matrix.

Table 1. Percentage recoveries of Deltamethrin in tomato fruits and the soil.

Level of fortification (mg kg^{-1})	% Recovery	% Relative standard deviation (RSD)
Fruit		
0.01	83.00	5.252
0.02	83.33	9.165
0.05	84.00	2.381
0.1	93.00	1.075
0.5	92.67	6.594
Soil		
0.01	82.00	2.112
0.02	81.67	7.070
0.05	85.33	1.353
0.1	89.67	2.807
0.5	88.00	6.818

4.3. Persistence and dissipation

A comparison of Deltamethrin dissipation behavior about the recommended dose (RD) and double the recommended dose (DD) is shown in **Figure 4**. Based on residue dissipation data, in tomato fruits, Deltamethrin's initial concentration in RD was found below detection limit (BDL) and decreased by 98.76% after 12 days in case of DD (**Table 2**). The findings coincide with earlier reports of Deltamethrin reaching below detectable level after 11 days from application [21]. There was no residue detected in soil hence, no dissipation pattern could be observed. The level of residue on or in the substrate dissipates at an overall rate when the substrate (such as leaf surfaces, fruit, plants) is foliar treated with a pesticide. In order for pesticide residues to dissipate, they need to be physically degraded, volatilized, photolyzed, washed off, leached, hydrolyzed and microbially degraded. The rate of degradation kinetics could be pseudo-first, first, or second order depending on rapid [In phase-1: the linear plot with $R^2 > 0.85$] or slow [Phase-2: two or more nonlinear plots with $R^2 \leq 0.85$] dissipation of the pesticide resulting in small or extended half-lives. Similar results were found in case of tebuconazole in tomato [22]. However, regulatory agencies apply first order kinetics to the entire dissipation period, regardless of whether there is any scientific basis for doing so. Biphasic dissipation kinetics of pesticides should be given special consideration in federal guidelines. The Deltamethrin dissipation kinetics in tomato was observed to be first-order, with a correlation of determination (R^2) of 0.9485 and 0.9823 of RD and DD respectively.

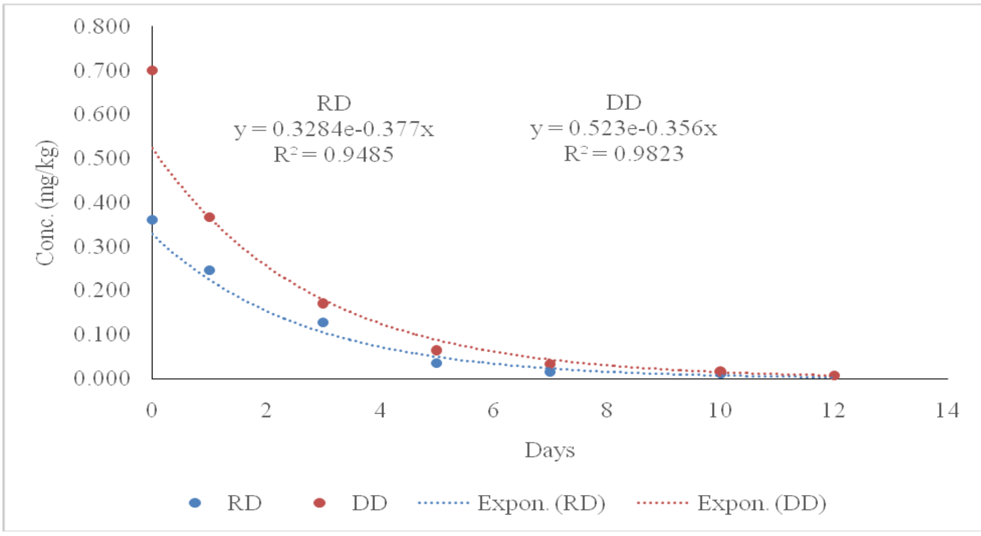

Figure 4. Degradation pattern of Deltamethrin in tomato fruits.

Table 2. Percentage reduction of residue on a different day of sampling of tomato fruits.

Days after spray	**Recommended dose (RD)**		**Double the recommended dose (DD)**	
	Residues (mg kg^{-1})	**% decrease in residue**	**Residues (mg kg^{-1})**	**% decrease in residue**
0	0.361	0.000	0.699	0.000
1	0.246	31.752	0.366	47.598
3	0.127	64.687	0.171	75.509
5	0.036	90.122	0.065	90.659
7	0.015	95.899	0.034	95.110
10	0.012	96.809	0.017	97.564
12	BDL		0.009	98.767

Dissipation of pesticide residues is typically expressed in terms of half-life ($t_{1/2}$), which represents the time required for 50% of the residue(s) to dissipate from their initial concentration. Deltamethrin showed half-lives of 1.84 and 1.95 days at the RD and DD, respectively. Deltamethrin was found to dissipate according to first order kinetics in a related study, with half-lives varying between 3.04 and 3.54 days for two different rates of foliar application [23]. Since it took less time for Deltamethrin to dissipate at both application dosages to reach the EU-MRL value, the chemical may be used safely in tomato crops to control fruit disease.

4.4. Consumer risk assessment

Both dosages of residues showed a similar trend of decline below the standard MRL of 0.07 mg kg^{-1}. The safety of this pesticide residue has to be assessed due to the dearth of information on the usage of Deltamethrin in vegetables. Deltamethrin's recommended daily intake (ADI) is 0.01 mg kg^{-1} of body weight per day. By, multiplying the ADI with the bodyweight of an average child (16 kg), the MPI of Deltamethrin was evaluated as 0.16 mg person^{-1} day^{-1}. On all sample days, for both the single dosage and the double dose, average daily consumption of tomato was

0.0179 and 0.025 kg day^{-1} for urban and rural residents respectively; also the dietary exposures to the residues were lower than the MPI of 0.16 mg $person^{-1}$ day^{-1} (**Table 3**). So, it is determined that when used for pest management in tomatoes, Deltamethrin carries a minimal risk of acute toxicity.

Table 3. Safety evaluation of day wise residues of Deltamethrin in tomato.

Sampling days	Recommended dose			Double the recommended dose		
	Residues (mg kg^{-1})	Dietary exposure (mg $person^{-1}$ day^{-1})		Residues (mg kg^{-1})	Dietary exposure (mg $person^{-1}$ day^{-1})	
		urban	rural		urban	rural
0	0.361	0.00646	0.009093	0.699	0.01251	0.017609
1	0.246	0.00441	0.006206	0.366	0.00655	0.009228
3	0.127	0.00228	0.003211	0.171	0.00306	0.004313
5	0.036	0.00064	0.000898	0.065	0.00117	0.001645
7	0.015	0.00026	0.000373	0.034	0.00061	0.000861
10	0.012	0.00021	0.00029	0.017	0.00030	0.000429
12	BDL			0.009	0.00015	0.000217

4.5. Decontamination of Deltamethrin residues from incurred samples

It was shown in the experiment that blanching (i.e., washing with hot water) decreased the Deltamethrin residues from tomato fruits by 75.36%. However, the residue removal was to the extent of 58.51%–65.93% with all the other treatments (**Table 4**). Deltamethrin is a highly active contact insecticide and a non-systemic compound. By the help of blanching process, the surface layer of insecticide residue may be removed and washed off from tomato. So, it represents as follows that boiling water has a better probability of eliminating Deltamethrin residues from tomato. Similar findings in the study demonstrate that blanching green chilies is a more effective method of removing kresoxim methyl [24].

Table 4. Effect of different household preparations in the removal of Deltamethrin residue from tomato fruits.

Treatment	% Reduction of residue
Washing with running tap water	58.51 ± 2.62
Washing with 1% NaCl solution	65.93 ± 3.48
Warm water (50 °C)	61.46 ± 0.88
Vinegar solution	64.06 ± 2.03
Washing with boiling water (blanching)	75.36 ± 1.46

5. Conclusion

In conclusion, the residue analysis method based on ethyl acetate extraction was quite effective in evaluating Deltamethrin residues in tomatoes by GC-μECD. The half-life of the Deltamethrin residues for the recommended dose and the double-recommended dose, respectively, were 6.3 and 5.3 days. The safety assessment indicates that tomato fruits may be regarded as safe for humans to eat. Deltamethrin was also reported to be safe in terms of application rate and pre-harvest interval in pear because the residue was 30 times lower than the MRL (1 mg kg^{-1}) [25]. Blanching could be employed as a method of decontaminating Deltamethrin.

Author contributions: Investigation and methodology, SM and JP; analysis, writing—original draft, PS, A and SS; method validation, data curation, writing—review & editing, SM, PAD and SGK; supervision, project administration, SM and TKB. All authors have read and agreed to the published version of the manuscript.

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Conflict of interest: The authors declare no conflicts of interest.

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