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DETERMINATION AND DISSIPATION KINETICS OF EMAMECTIN BENZOATE AND DELTAMETHRIN RESIDUES IN FIELD PEA SEEDS USING LC-MS/MS AND GC-µECD

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ABSTRACT

The present study was aimed to establish and validate analytical methods for quantifying emamectin benzoate and deltamethrin residues in field pea seeds under storage conditions. Emamectin benzoate residues were analysed using liquid chromatography-tandem mass spectrometry, while deltamethrin residues were analysed using gas chromatography with micro-electron capture detection. The procedure was validated using the applicable analytical quality-control requirements. Matrix-matched calibration was linear from 0.0025-0.100 µg mL^{-1}. Recoveries from spiking levels of 0.01-0.50 mg kg^{-1} varied from 80 to 95%, with relative standard deviations below 20%, matrix effects within ±20%, and a limit of quantification of 0.01 mg kg^{-1} for both analytes. Pea seed samples were taken 2 h, 90 days, and 180 days after insecticidal seed treatment during storage. Residues of both insecticides dissipated following first-order kinetics. Emamectin benzoate declined from 0.504 mg kg^{-1} at 2 h to 0.155 mg kg^{-1} at 180 days, with a half-life of 99.0 days and a coefficient of determination of 0.896, whereas deltamethrin declined from 0.412 to 0.127 mg kg^{-1} over the same period, with a half-life of 69.3 days and a coefficient of determination of 0.936. These findings characterize the long-term dissipation behaviour of emamectin benzoate and deltamethrin in stored field pea seeds and provide baseline information relevant to residue management during storage.

Keywords: Deltamethrin, emamectin benzoate, gas chromatography-micro-electron capture detection, liquid chromatography-tandem mass spectrometry, pea seeds, pesticide residues, residue dissipation

INTRODUCTION

Pulses are essential nutritional staples in India and in other developing countries, prized for their high protein content and ability to improve soil fertility through biological nitrogen fixation. Among them, field pea (*Pisum sativum* var. *hortense* L.) is a popular winter pulse crop, especially in northern India. However, using insecticides to protect stored seeds from pests like pulse beetles (*Callosobruchus* spp.) has raised concerns about chemical residues and their persistence in food products. Hence, it is

essential to understand the residue dissipation in stored seeds for assessing residue persistence and supporting safe pesticide use.

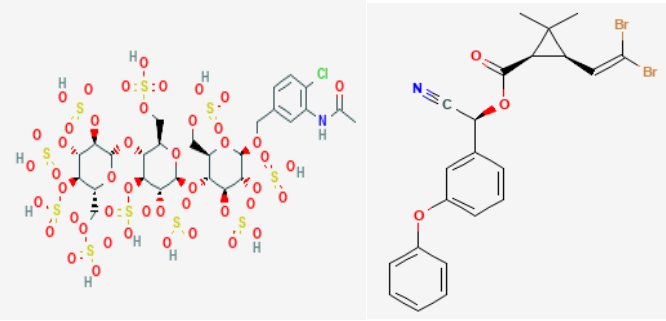
Pesticide dissipation varies depending on the pesticide, crop matrix, application conditions, and commodity characteristics. Patel *et al*. (2025) used LC-MS/MS to study the dissipation and dietary risk of emamectin benzoate and chlorantraniliprole in green cowpea and found first-order dissipation of emamectin benzoate, with residues declining below the limit of quantification within 5 days and a half-life of 1.9 days. Majumder *et al*. (2026) examined deltamethrin residues in cowpea pods, leaves, and soil under field settings and found half-lives of 1.73-3.15 days in cowpea pods. Wang *et al*. (2021) found that emamectin benzoate dissipation in cowpea varied with formulation and pod maturity, with tender cowpea dissipating more quickly than mature cowpea. These studies show that residue behaviour cannot be readily extrapolated to other crops, plant parts, or storage conditions. The physicochemical properties of emamectin benzoate and deltamethrin, including their molecular weight, water solubility, log K_ow, and vapour pressure, differ considerably and may influence their persistence and dissipation behaviour. The information on dissipation of these pesticides in field pea seeds remain scarce. Existing research has focused primarily on cowpea and other vegetable or pulse commodities, generally under field conditions and in green pods or foliage rather than in stored edible seeds. Such findings may not be directly applicable to field pea seeds since residue persistence is affected by commodity-specific factors such as seed composition, moisture content, surface features, pesticide-matrix interactions, and storage conditions. Furthermore, there is a lack of comparison data on the behaviour of emamectin benzoate and deltamethrin within the same field pea seed matrix, making it difficult to assess the persistence of these two chemically distinct insecticides under identical storage conditions.

Reliable residue data is required by regulatory frameworks for pesticide registration, labelling, and food safety evaluation. The Central Insecticides Board and Registration Committee (CIBRC) requires supervised residue trials and terminal residue data for food commodities, whereas All India Coordinated Research Project on Pesticide Residues focuses on systematic pesticide residue monitoring in food commodities, including pulses (CIBRC, 2022; AICRP, 2023). Therefore, the present study was aimed to generate commodity-specific residue dissipation information for emamectin benzoate and deltamethrin in field pea seeds under storage conditions. Emamectin benzoate and deltamethrin residues were quantified using LC-MS/MS and GC-μECD, respectively, and their dissipation kinetics evaluated during storage. This study provides comparative residue-depletion and dissipation-kinetic information for these two insecticides in field pea seeds, addressing a research gap not adequately covered by previous studies on cowpea, green pods, or other crop matrices. The findings provide crop and commodity-specific evidence that may support residue management, food-safety assessment, and regulatory evaluation of insecticide use in field pea.

MATERIALS AND METHODS

Experimental design, seed treatment and storage conditions

The experiment was conducted at the ICAR–Indian Institute of Vegetable Research (ICAR-IIVR), Varanasi, Uttar Pradesh, India, to determine the dissipation kinetics of emamectin benzoate and deltamethrin residues in field pea seeds. The experiment conducted in a completely randomized design (CRD) and comprised of three treatments: emamectin benzoate @ 2 ppm, deltamethrin @ 1.0 ppm, and an untreated control, all replicated three times. The insecticides were applied as seed treatments using their respective commercial formulations. Emamectin benzoate was applied using Proclaim 5 SG (5% a.i.) at 40 mg formulation kg^{-1} seed, equivalent to 2 mg a.i. kg^{-1} seed, while deltamethrin was applied using Deltamethrin 2.8 EC (28 g a.i. L^{-1}) at 0.04 mL formulation kg^{-1} seed, equivalent to 1 mg a.i. kg^{-1} seed. The chemical structure of these two pesticides is given in Fig. 1.



(a) (b)

Fig. 1: Chemical structure of emamectin benzoate (a) and deltamethrin (b)

Following treatment, the seeds were stored under ambient room-temperature conditions to simulate typical on-farm/ godown storage. Samples were collected at 0, 90 and 180 days after treatment and immediately stored at -20°C until extraction to prevent further residue degradation. Residue extraction and analysis were conducted at the Pesticide Residue Analysis Laboratory, ICAR-IIVR, Varanasi, using LC-MS/MS for emamectin benzoate and GC-µECD for deltamethrin. The residue data generated was used to determine dissipation kinetics of both insecticides.

Chemicals and reagents

Pesticide standards and LC-MS/MS grade formic acid were sourced from Sigma-Aldrich Pvt. Ltd., India. Acetonitrile was obtained from Thermo Fischer, Japan. Anhydrous sodium sulfate (Na_2SO_4) and analytical reagent grade acetic acid were procured from Thomas Baker, Mumbai, India. HPLC-grade water was supplied by the Sartorius water purification system (Sartorius AG, Germany). Primary secondary amine (PSA, 40 µm), graphitized carbon black (GCB), and magnesium sulphate ($MgSO_4$) were acquired from Sigma. The equipment used in the study included a precision balance (Vibra, Adair Dutt, India), a vortex mixer (Geni 2T, Imperials Biomedicals, India), a centrifuge (Kubota, Germany), and an ultrasonic bath (Oscar Electronics, Mumbai, India).

Preparation of standard solutions

Standard solutions were prepared by dissolving the accurately weighed 10 (±0.1) mg certified reference materials in 10 mL acetonitrile to achieve a concentration of 1000 µg mL^{-1}. A working solution of 10 µg mL^{-1} concentration was made in acetonitrile by suitably mixing portions of individual stock solutions and performing further dilutions. From this working solution, external calibration standards were prepared at the concentrations of 0.0025, 0.005, 0.01, 0.025, 0.05, and 0.10 µg mL^{-1}. Matrix-matched standards at the same concentrations were prepared using extracts from control matrix samples.

Mass spectrometry (MS) system

The LC-MS/MS analyses were performed using a Shimadzu I-series LC-2050C HPLC coupled with a Shimadzu 8045 mass spectrometer via an electrospray ionization (ESI) interface, achieving pesticide separation on a Shim Pack GIST C18 column (2.1 × 150 mm, 3.0 µm). Argon served as the collision gas and nitrogen as the ion source gas, with LabSolution software managing system control and data analysis. The triple quadrupole MS featured a heated ESI probe for improved sensitivity under various conditions, operating across a mass range of m/z 2-2000 with high mass stability and resolution, rapid scanning up to 30,000 amu sec^{-1}, and swift polarity switching in 5 ms. Sensitivity tests indicated excellent ESI responses, detecting as low as 1 pg reserpine or chloramphenicol with signal-to-noise ratios exceeding 500,000: 1 and instrument detection limits below a few femtograms. The system minimized cross-talk (<0.005%) and efficiently conducted high-speed multiple reaction monitoring (MRM) with up to 555 channels sec^{-1}. The interface parameters were optimized by adjusting heating gas flow (5-10 mL min^{-1}), interface temperature (200-300°C), drying gas, and DL and HB settings, with a chosen injection volume of 3 µL to ensure optimal performance.

Optimization of MS/MS conditions

The optimized analysis in MRM mode was based on identifying the two most prominent transitions used for quantification and confirmation. Individual standard solutions were injected into the tandem mass spectrometer to determine these transitions and to fine-tune the acquisition parameters. All

selected pesticides were analyzed in positive ionization mode. Instrument sensitivity was assessed by establishing the method detection limits (MDLs) for emamectin benzoate (Majumder *et al*., 2024a-d).

High performance liquid chromatography (HPLC) system

An HPLC system equipped with a quaternary gradient pump, autosampler, column oven, and UV detector was employed. The system supports solvent flow rates between 0.001 and 10 mL min^{-1}. Experiments were carried out to evaluate the suitability of two different columns: a) a Shim-pack HPLC column packed with 3 µm C_{18} particles [2.1 × 150 mm], and b) a Shim-pack HPLC column with 5 µm C18 particles [4.6 × 250 mm], to assess the separation efficiency of analytes. The system can withstand pressures exceeding 7000 psi and includes an online membrane degasser with four solvent lines. The autosampler features variable injection volumes ranging from 0.1 to 50 µL and comprises two racks, each accommodating 108 vials of 1.5 mL capacity. The injector provides an injection volume accuracy of ±1% and minimal carryover below 0.0025%. The column oven operates from ambient temperature up to 80°C with a temperature accuracy of ±0.5°C and can house two columns, each up to 300 mm long. Additionally, the system is fitted with a programmable flow-divert valve for automated switching between the LC-MS and waste. In this study, peak shapes and sensitivity for each target analyte were compared at injection volumes of 1, 3, and 5 µL.

Data interpretation (qualitative and quantitative analysis)

The collected data for both standards and samples were analyzed using Lab Solution software. During data processing, the presence of two transitions for each analyte at the same expected retention time was verified. For emamectin benzoate, the retention time was 14.73 min, with the two transitions showing consistent retention-time agreement. The ion ratio between these two transitions was calculated, and it was ensured that this ratio remained within 30% of the ion ratio observed in reference standard (Majumder *et al*., 2024a-c). Once these identification criteria were satisfied, quantification was performed, retention times were checked to fall within ±0.1 min, and it was confirmed that the calibration standards exhibited a response that increased proportionally with concentration. An optimal concentration level was selected for setting up the quantification method. The calibration curve for standards was constructed using a linear equation with a weighting factor of 1/x (Majumder *et al*., 2024a-c).

Sampling

Field pea seed samples (100 g) were collected from each replicate treated with emamectin benzoate and deltamethrin as well as untreated control separately at 0, 90 and 180 days after respective insecticidal application. The samples were collected in polyethene bags and stored at -20°C until analysis to avoid any degradation. Unlike conventional field dissipation studies, in which residues are typically monitored at short intervals following pesticide application (e.g., 2 h, 1, 3, 5, 7 and 10 days), the present experiment was designed to assess the persistence and dissipation of insecticide residues in field pea seeds under storage conditions. Therefore, sampling intervals were selected based on the storage duration rather than rapid initial dissipation phase. Samples were collected at 0, 90 and 180 days after treatment, representing the initial, intermediate and extended storage periods, respectively. The 0-day sample represented the initial residue concentration i.e., 2 h following treatment, while the 90- and 180-day samples were used to assess residue persistence during prolonged storage. This sampling schedule was intended to characterize the overall decline in emamectin benzoate and deltamethrin residues during storage period, and the resulting residue data were used to estimate dissipation rate and half-life using a first-order kinetic model.

Extraction and clean-up

The samples were extracted by using the procedure followed by Aralimarad *et al*. (2025) with minor modifications. The QuEChERS technique outlined by Anastassiades *et al*. (2003) and Gonzalez-Curbelo *et al*. (2014) is widely used for preparing the samples to extract various organic contaminants, including pesticides of differing polarities and volatilities, from diverse matrices. A key adaptation of this method involves adding extra water to dry samples for ensuring adequate moisture and improve

recovery rates (Koesukuiwat *et al*., 2010; Mastovska *et al*., 2010). In this procedure, 10 ± 0.1 g homogenized field pea seed samples were taken in a 50 mL polypropylene centrifuge tube containing 10 mL water and mixed using a vortex, followed by the addition of 20 mL acetonitrile. The mixture was homogenized using a low-volume homogenizer at 12,000 rpm for 2 min. Subsequently, 2 g NaCl was added and the shaken thoroughly, followed by centrifugation at 10,000 rpm for 5 min at 10°C to separate the organic layer. Next, 10 mL upper organic phase (supernatant) was carefully transferred into a test tube containing 5 g anhydrous sodium sulfate to remove residual moisture. Then 7 mL of this extract was transferred to a 15 mL centrifuge tube containing 0.2 g PSA, 1.0 g anhydrous $MgSO_4$, and 25 mg graphitized carbon black for clean-up. The mixture was vortexed for 2 min and centrifuged again at 10,000 rpm for 5 min at 10°C. Finally, 1 mL supernatant was filtered through a 0.22 µm syringe filter into LC vials, and 1 µL purified extract injected into LC-MS/MS system for analysis.

GC-µECD analysis for deltamethrin

The detection and analysis of deltamethrin residues in field pea seeds were carried out using an Agilent 7890B gas chromatograph equipped with an autosampler and a microelectron capture detector (µECD) operating with a ^63Ni source. The instrument was configured with a split/splitless injector working in split mode (10:1 ratio) at 250°C, injecting precisely 1 µL extract each time. Separation of analytes was achieved on an HP-5 capillary column (30 m length, 320 µm internal dia., 0.25 µm film thickness), with nitrogen as carrier gas at a flow rate of 2 mL min^{-1}. The detector temperature was maintained at 300°C, supported by a makeup nitrogen gas flow of 60 mL min^{-1}. The oven temperature program began at 100°C, held for one min, then ramped up at 15°C min^{-1} to 280°C, where it was held for 2 min. Under these optimized conditions, deltamethrin was detected at a retention time of 15.77 min. For quantification, 1 µL clean extract was injected under identical conditions, and pesticide residues estimated by comparing the peak areas of standards to those of unknown or spiked samples (Majumder *et al*., 2022). The residue concentration was calculated using the formula:

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

where A_1 is the chromatographic peak area of sample, A_2 is the peak area of analytical standard, V_1 is the total extract volume, V_2 is the injection volume, C is the concentration of standard, and W is the sample weight (g).

For ensuring the reliability of this analytical method, a comprehensive single-laboratory validation was conducted following the SANTE/11312/2021 guidelines (ECDGHFS, 2021), evaluating the parameters such as linearity, detection and quantification limits, matrix effects, accuracy, and precision. Matrix-matched external calibration was performed at six concentration levels (0.0025, 0.0050, 0.0100, 0.0250, 0.0500, and 0.1000 µg mL^{-1}), and calibration curves constructed. Method linearity was evaluated across the selected concentration range. Matrix effects (ME) were assessed by comparing the slopes of the solvent-based calibration curve with those obtained from the matrix-matched calibration curve. The selectivity and sensitivity of method was established through limits of detection (LOD) and quantification (LOQ), defined as the lowest measurable levels in field pea seed matrix, corresponding to signal-to-noise ratios of 3:1 and 10:1, respectively. The recovery studies were conducted at spiking levels of 0.01, 0.02, 0.05, 0.10 and 0.50 mg Kg^{-1}. Precision was evaluated through both repeatability and reproducibility assessments. Matrix effects (ME) arise when the components of sample matrix other than the analyte of interest interfere with analytical measurements, potentially affecting the accuracy and reliability of quantification. These effects may result from the co-elution of matrix constituents, leading to signal enhancement or suppression (Zhang *et al*., 2014). In present study, matrix effects were evaluated by comparing the slopes of calibration curves prepared in matrix-matched solutions with those obtained from solvent-based standard solutions (Majumder *et al*., 2023). The matrix effect was calculated using the following equation:

$$ME\ (\%) = (M_{matrix} - M_{solvent})/M_{solvent} \times 100$$

where ME represents the matrix effect, M_{matrix} is the slope of the matrix-matched calibration curve, and $M_{solvent}$ is the slope of the solvent-based calibration curve. Matrix effects within the range of −20% to +20% were considered negligible. Values exceeding +20% indicated signal enhancement, whereas

values below −20% indicated signal suppression. Matrix-matched calibration was used to account for potential matrix-related variations and improve the reliability of residue quantification.

The dissipation behaviour of insecticide residues was evaluated using a first-order kinetic model following Hoskins (1961), as represented by Eq. (3):

$$A_t = A_0 e^{-kt} \quad (3)$$

where A_t denotes the residue concentration at time t, A_0 represents the initial residue concentration, k is the first-order dissipation rate constant, and t is the elapsed storage period.

The dissipation half-life ($t_{1/2}$) of insecticides was estimated from residue concentration data using Eq. (4) [Majumder *et al*. 2024a-c]:

$$t_{1/2} = \ln(2)/k \quad (4)$$

where k is the first-order dissipation constant derived from regression analysis of the residue concentration data (Malhat *et al*., 2016; Singh *et al*., 2023).

At each sampling interval (0, 90 and 180 days), one sample from each of the three independent replicates was collected and analyzed. The reported residue concentration represents the mean value of three replicate determinations. This kinetic modeling provides valuable insight into the behaviour of emamectin benzoate residues over time, crucial for determining safe storage intervals and ensuring compliance with regulatory standards for food safety. As agricultural practices evolve, future studies should focus on how changing environmental factors, storage conditions, and integrated pest management strategies might further influence residue behaviour in field pea seeds and other crops.

Method validation

Validation of analytical methods is essential for all analytes to demonstrate that the suitability of method for its intended use. Validation was conducted following the SANTE/2020/12830 guidelines (ECDGHFS, 2020). The method underwent testing to evaluate its sensitivity, mean recovery, precision, and limit of quantification (LOQ). The analytical method was validated for linearity, accuracy, precision, and LOQ. Linearity was assessed using six calibration levels (0.0025, 0.005, 0.01, 0.025, 0.05, and 0.10 µg mL^{-1}) using matrix-matched calibration. Accuracy and repeatability were evaluated through recovery studies by spiking blank matrix samples at 0.01, 0.02, 0.05, 0.1, and 0.5 mg kg^{-1}. The spiked samples were processed according to the sample preparation procedure, and recoveries calculated from the matrix-matched calibration curves. LOQ was defined as the lowest validated concentration meeting the acceptance criteria of 80-120% mean recovery and ≤ 20% RSD. An appropriate set of validation samples included solvent blank, matrix blank, spiked matrix samples, and matrix-matched calibration standards. Analyte concentrations were determined by interpolation of relative peak areas against the corresponding calibration curves (Debnath *et al*., 2024; Majumder *et al*., 2024a-c).

RESULTS AND DISCUSSION

Optimization of MS/MS conditions

Optimizing mass spectrometry parameters is crucial for achieving maximum sensitivity. In this study, standards were prepared as solutions of 1 mg L^{-1} in methanol, and both positive and negative ion scanning modes were simultaneously applied. Initially, a full scan (MS1) was conducted for each individual standard solution of the target analytes to identify parent ions with high and stable signal intensity. Once the parent ion was determined, its fragmentation was optimised in single ion monitoring (SIM) mode. In this mode, the parent ion was introduced into the second stage of mass spectrometer, where it underwent fragmentation or rearrangement to generate specific product ions. In product ion mode, defined collision energy (CE) levels were applied to the parent ions to produce characteristic fragment ions. Finally, in multiple reaction monitoring (MRM) mode, the collision energies for the product ions were fine-tuned to achieve optimal mass spectrometry performance. The

final MRM parameters, including optimised retention times, parent and product (daughter) ions, and collision energies for each target pesticide (Table 1).

Table 1: Multiple reaction monitoring (MRM) parameters, calibration range and limit of quantification (LOQ) for emamectin benzoate determined by LC-MS/MS

Insecticide	M.W. (g mol^{-1})	RT (min)	Precursor ion	Product ion-01	Product ion-02	Q1 pre-bias	Collision energy (CE)	Q3 pre-bias	Calibration range (µg mL^{-1})	LOQ (mg kg^{-1})
Emamectin benzoate	1008.2	14.73	886.40	158.20	82.25	−22	−65	−16	0.0025-0.100	0.01

Optimization of chromatographic conditions

After establishing the optimal MRM parameters, the HPLC method was refined to enable rapid and precise separation of peaks corresponding to the target analytes. A C_{18} column with a 3 µm particle size was selected because it provided better retention of analytes, along with improved peak shape and response. The mobile phases consisted of two reservoirs: Mobile phase A contained 5 mM ammonium formate in water with formic acid, and Mobile phase B contained 5 mM ammonium formate in methanol with formic acid. For dilution and elution purposes, three options were tested: a) pure acetonitrile, b) pure methanol, and c) a 1:1 mixture of mobile phases A and B. The 1:1 mixture yielded best signal response for eluting the target pesticides from column. Under these optimised conditions, the target compound eluted within 7.921 min. Therefore, the optimised instrument conditions significantly shortened run times, ensuring fast and efficient elution of analytes from column (Majumder *et al*., 2024a-c).

Method validation

Matrix-matched calibration curves for emamectin benzoate and deltamethrin were linear over the range 0.0025-0.100 µg mL^{-1}. Pesticide recoveries across five spiking levels (0.01, 0.02, 0.05, 0.10 and 0.50 mg kg^{-1}) ranged from 80 to 95%, with relative standard deviations (RSD) below 20% for both repeatability and reproducibility. These results comply with the performance criteria established under SANTE (11312/2021) guideline (CDGHFS, 2021). The LOQ was set at 0.01 mg kg^{-1}, corresponding to the lowest spiking level at which mean recoveries fell between 80 and 120% with RSD below 20%. To minimize matrix effects, different clean-up sorbents and their combinations were evaluated; matrix effects for both pesticides in pea seed matrix were effectively controlled (within ±20%) through this optimization process. Representative LC-MS/MS chromatograms of emamectin B1a analytical standards are given in Fig. 1. The target analyte produced a distinct chromatographic response at a consistent retention time, with MRM transition of m/z 886.40 → 158.20 used for detection and quantification. The observed chromatographic response increased with increasing concentration of analytical standard, supporting the suitability of developed LC-MS/MS method for quantitative determination of emamectin benzoate residues. The dissipation of both insecticides under storage followed first-order kinetics.

Dissipation kinetics

Under the established chromatographic conditions, the retention times for all pesticides in LC-MS/MS were within an acceptable deviation of ±0.1 min. The matrix effect varied depending on matrix complexity and was calculated by comparing calibration curve slopes obtained in solvent versus matrix-matched standards prepared with control extracts for each pesticide. In this study, pesticide residue analysis was performed using triple quadrupole LC-MS/MS, where two product ions were monitored alongside a precursor ion for each analyte. The ion ratio, calculated as the percentage of peak area of confirmation ion relative to the quantification ion was considered valid if it deviated by no more than ±30% from the standard, ensuring analyte confirmation. Retention time consistency was also crucial, requiring analyte peaks in the samples to match those of matrix-matched standards within a ±0.1-min window. For real sample analysis, 100 g treated and untreated field pea seeds were collected, stored at -20°C, and extracted using a standardized method. The pesticide residues were

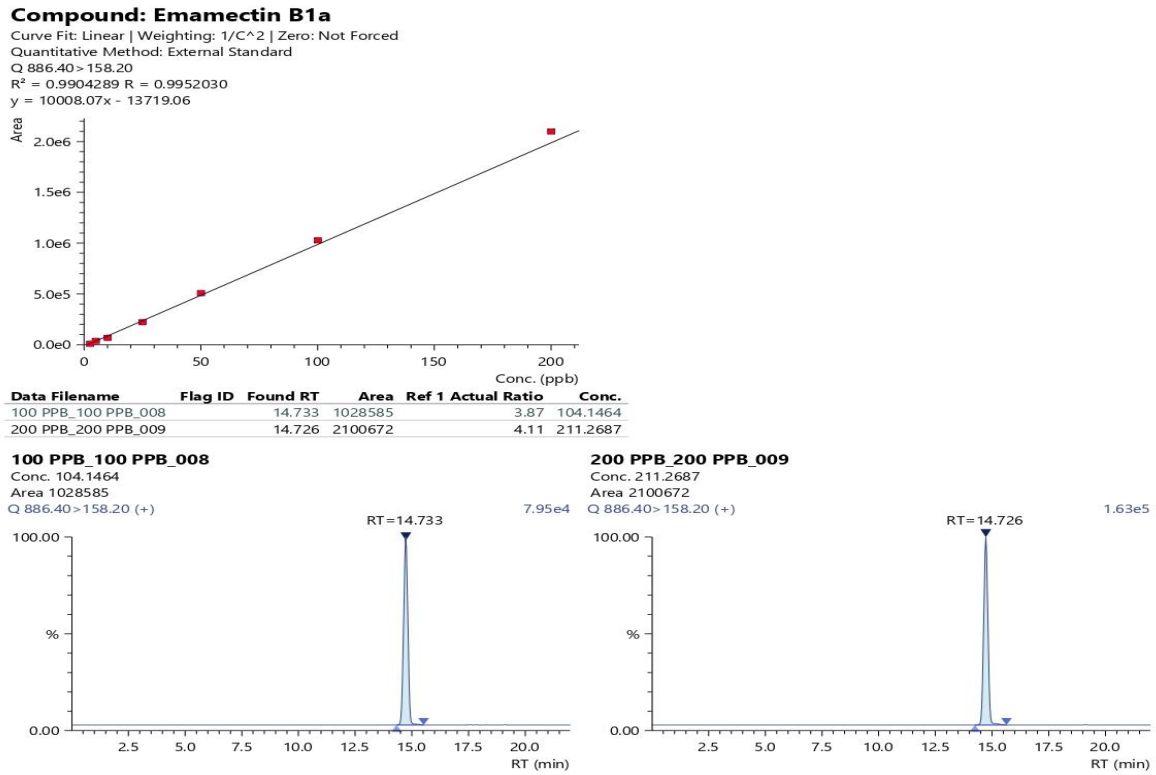


Fig. 1: Representative LC-MS/MS MRM chromatograms of emamectin B1a analytical standards at 100 and 200 ppb, showing the characteristic transition (m/z 886.40 → 158.20)

quantified under validated LC-MS/MS conditions using established formula to compute both sample weight and residue concentration. To understand residue behaviour, the dissipation of deltamethrin and emamectin benzoate was modelled using a first-order kinetic equation ($At = A_0e^{-kt}$), wherein At is the residue at time t, A_0 is initial concentration, and k is rate constant. The half-life ($t_{1/2}$) of pesticides was derived using the formula $t_{1/2} = \ln(2)/k$, providing insight into how quickly each chemical degraded over time.

Pesticide residues present in crops can diminish over time due to various environmental and biological processes, such as microbial degradation, volatilization, photodegradation, and chemical breakdown. These factors collectively contribute to the gradual reduction of pesticide levels, ensuring that residues do not persist indefinitely in the environment or food products. In current study, the dissipation pattern of emamectin benzoate demonstrated an initially rapid decline in residue levels, which later progressively slowed down, indicating a characteristic exponential decay curve. This suggests that the degradation of emamectin benzoate followed simple first-order kinetic behaviour, as evidenced by a high correlation coefficient ($R^2 = 0.896$), confirming the reliability of the kinetic model applied. Specifically, 2 h post-application, the concentration of emamectin benzoate residues in pea seeds was recorded at 0.504 mg kg^{-1}. Over time, this level declined to 0.197 mg kg^{-1} after 90 days and

Table 2: Dissipation and persistence of emamectin benzoate and deltamethrin residues in pea seeds

Insecticide	Days after insecticide application	Residues (mg kg^{-1})	Regression equation	Coefficient of determination (R^2)	Half-lives ($t_{1/2}$) days
Emamectin benzoate	0	0.504			
	90	0.197			
	180	0.155	$y = 448.75e^{-0.007x}$	$R^2 = 0.896$	99.0
Deltamethrin	0	0.412			
	90	0.211			
	180	0.127	$y = 0.7059e^{-0.01x}$	$R^2 = 0.936$	69.3

further decreased to 0.155 mg kg^{-1} after 180 days, reflecting the gradual breakdown of the chemical under storage conditions.

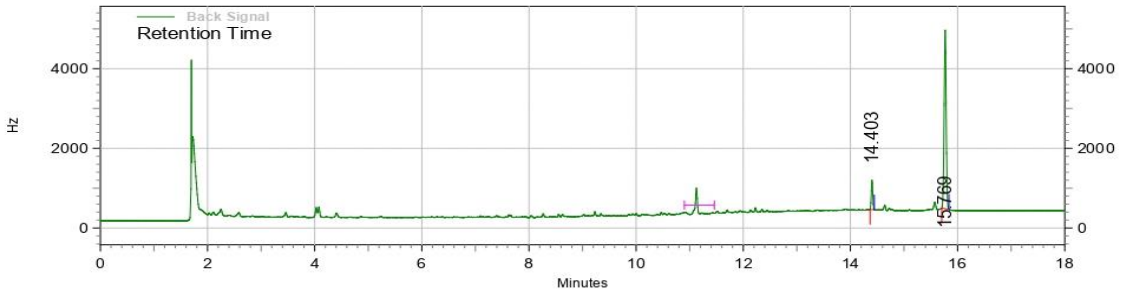
Similarly, in pea seeds, the dissipation of deltamethrin was assessed following seed treatment. The initial residue concentration detected just 2 h after application was 0.412 mg kg^{-1} (Table 2). The dissipation kinetics of deltamethrin in pea seeds also followed a simple first-order pattern, demonstrating the predictable degradation behaviour. The residue levels of deltamethrin decreased to 0.211 mg kg^{-1} after 90 days and further reduced to 0.127 mg kg^{-1} after 180 days (Table 2), indicating substantial degradation over time. These findings underscore the dynamic nature of pesticide residues in agricultural products and highlight the importance of understanding dissipation behavior to ensure food safety and compliance with regulatory standards.

GC-µECD analysis for deltamethrin

During method validation, the method proved highly sensitive, establishing LOD and LOQ for delatmethrin at 0.005 mg kg^{-1} and 0.01 mg kg^{-1}, respectively. Impressively, average recoveries at spiking levels of 0.01-0.50 mg kg^{-1} in field pea seeds consistently surpassed 80-95%, indicating the method's reliability with the retention time of 15.695 min. However, significant matrix effects were observed, being < 20%, necessitating the adoption of matrix-matched calibration curves to ensure precise quantification. These findings underscore the method's robustness and the critical role of accounting for matrix influences in achieving accurate pesticide residue analysis in field pea seeds. A representative chromatogram of deltamethrin analytical standard at 500 ppb is given in Fig. 2. The chromatogram showed a distinct response for analytical standard, with major chromatographic peak observed at 15.77 min. The standard chromatogram provided the reference retention time for identification and quantitative determination of deltamethrin residues in the field pea seed samples.

Area % ECD Reports

Data File: C:\Enterprise\Projects\IIVR\Result\Deltaethrin 2 07082021.rslt\500 ppb.dat
Method: C:\Enterprise\Projects\IIVR\Method\Deltamethrin-2.met
Acquired: 07-08-2021 13:55:47 (GMT +05:30)
Printed: 07-08-2021 14:59:40 (GMT +05:30)



Back Signal
Results

Retention Time	Area	Area %	Height	Height %
14.403	10778448	10.58	5679411	14.16
15.769	91069621	89.42	34421572	85.84
Totals	101848069	100.00	40100983	100.00

Fig. 2: GC chromatogram of deltamethrin analytical standard at 500 ppb, showing characteristic chromatographic peak at a retention time of 15.77 min

Physicochemical properties of insecticides relevant to residue persistence in stored field pea seed

The dissipation of emamectin benzoate and deltamethrin in stored pea seeds, with residues declining from 0.504 to 0.155 mg kg^{-1} and from 0.412 to 0.127 mg kg^{-1}, respectively (Table 2), over 180 days,

is consistent with their physicochemical properties. Emamectin benzoate is a macrocyclic lactone with relatively low water solubility and moderate binding affinity to organic matrices, and is reported to degrade via photolysis, hydrolysis, and microbial action (USEPA, 2010; EFSA, 2012), consistent with its faster relative decline in this study. Deltamethrin, a synthetic pyrethroid, is highly lipophilic and strongly sorptive to organic matter, and has been reported as more persistent in stored products and environmental matrices (Tomlin, 2009; EFSA, 2017; PPDB, 2023), consistent with the somewhat slower relative decline observed here. These properties are drawn from established physicochemical and regulatory databases and were not independently measured in the present study.

The initial residues of emamectin benzoate and deltamethrin in field pea seeds were 0.504 and 0.412 mg kg^{-1}, respectively, at 2 h after treatment (Table 2) which subsequently declined to 0.197 and 0.211 mg kg^{-1} at 90 days and to 0.155 and 0.127 mg kg^{-1} at 180 days after treatment, indicating gradual dissipation of both insecticides during storage (Fig. 3 and 4). The dissipation pattern of emamectin benzoate followed first-order kinetics ($R^2 = 0.896$), while deltamethrin showed a progressive decline throughout the storage period. The persistence observed in present study was markedly longer than that generally reported following foliar application to actively growing crops. This difference is important because the present investigation involved seed treatment followed by prolonged storage, whereas most published residue studies have evaluated foliar applications under field conditions.

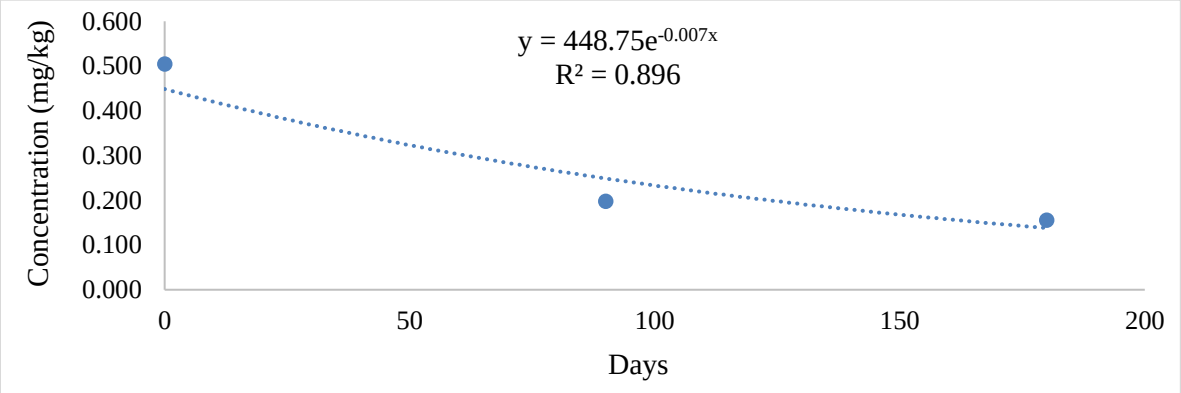


Fig. 3: Degradation pattern of emamectin benzoate in pea seeds

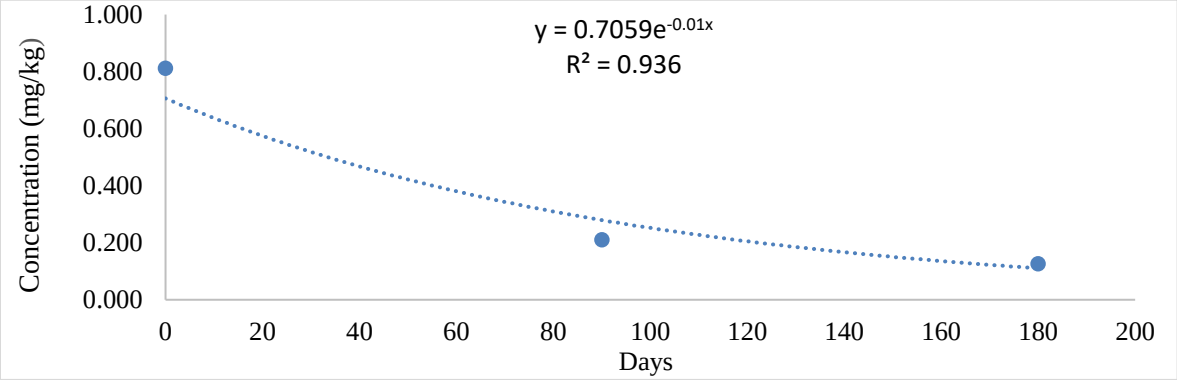


Fig. 4: Degradation pattern of deltamethrin in pea seeds

Several recent studies have reported the dissipation behaviour of emamectin benzoate in different crop matrices. Patel *et al*. (2025) reported first-order dissipation of emamectin benzoate in green cowpea pods following field application, with a half-life of 1.9 days and residues declining below the LOQ of 0.01 mg kg^{-1} by 5 days. Similarly, Katna *et al*. (2026) reported short half-lives of emamectin benzoate in cauliflower and brinjal, ranging from 1.25 to 1.48 days and 1.08 to 1.17 days, respectively, following field application. Vijay *et al*. (2024) also reported first-order dissipation of emamectin benzoate in cauliflower curd, with a half-life of 8.85 days. These studies demonstrate that emamectin

benzoate generally declines within a relatively short period in harvested vegetable commodities following foliar application, although the rate of dissipation varies among crop matrices and application conditions.

The influence of crop matrix and maturity on emamectin benzoate dissipation has been demonstrated by Wang *et al*. (2021), who reported first-order dissipation in tender and old cowpea. The half-lives of emulsifiable concentrate formulation were 1.34-1.39 days in tender cowpea and 1.74-2.31 days in old cowpea, while those of the microemulsion formulation were 1.39-1.51 and 2.08-2.67 days, respectively. The comparatively slower dissipation in old cowpea indicates that residue behaviour varies with commodity characteristics and developmental stage. Balkan *et al*. (2024) reported half-lives of 7.30-8.15 days for emamectin benzoate in grape under field conditions, demonstrating that dissipation rates are matrix- and crop-dependent. Therefore, direct comparison of numerical half-lives across studies should be made cautiously when the crop, plant part, application method and environmental conditions differ.

The deltamethrin results of present study can be compared with recent field-residue studies. Majumder *et al*. (2026) reported first-order dissipation of deltamethrin in cowpea pods, with half-lives of 1.73 days at the recommended dose and 3.15 days at the double dose. Sharma *et al*. (2023) observed that deltamethrin residues in chickpea pods declined below LOQ within 10 and 15 days at the recommended and double recommended doses, respectively. Majumder *et al*. (2024c) reported half-lives of 1.95 and 1.84 days for deltamethrin in tomato at the recommended and double recommended doses, respectively. In contrast, Singh *et al*. (2024) reported dissipation of deltamethrin in bell pepper, further demonstrating that residue decline varies among crop matrices and application conditions. These studies predominantly involved foliar application to growing crops, whereas the present study involved treatment of dry field pea seeds followed by storage; therefore, the substantially longer persistence observed in the present study should not be interpreted as a contradiction of the rapid dissipation reported in field-grown crops. The evidence from post-harvest commodities highlights the influence of storage conditions on pesticide persistence. Recent studies on deltamethrin in stored wheat and pesticide behaviour during cereal-grain drying indicate that storage, processing and temperature can affect residue dissipation (Rekabi *et al*., 2026). Thus, residue decline in stored commodities may differ from dissipation on plant surfaces under field conditions.

Overall, emamectin benzoate and deltamethrin generally show first-order or approximately first-order dissipation, although rates vary with pesticide, crop matrix, formulation, application method and storage conditions. The present study complements existing field-based research by characterizing residue decline of both insecticides in seed-treated field pea during 180 days of storage using LC-MS/MS and GC-μECD. The findings emphasize the need for commodity-specific residue data for treated seeds intended for prolonged storage. The first-order pattern describes residue decline mathematically and does not establish a specific degradation mechanism. Microbial transformation, hydrolysis, photolysis, volatilization and binding to the seed matrix were not independently evaluated. Therefore, the study demonstrates the net decline of measurable residues during storage, while the contribution of individual mechanisms requires further investigation,

Limitations and future scope

The present study revealed dissipation of emamectin benzoate and deltamethrin residues in stored field pea seeds over 180 days; however, the observed decline represents residue dissipation from the seed matrix and does not establish specific degradation pathways or complete environmental fate. Processes such as photolysis, hydrolysis, microbial degradation, sorption, volatilization, and transformation to metabolites were not experimentally studied. Although these processes have been reported for emamectin benzoate and deltamethrin under different environmental conditions (USEPA, 2002; Tomlin, 2009; USEPA, 2011; EFSA, 2017; EFSA, 2022; PPDB, 2023), they should therefore be considered possible explanations rather than mechanisms demonstrated by the present study.

Previous studies have reported first-order or pseudo-first-order dissipation of emamectin benzoate in cowpea, amaranth, mustard, cauliflower and fruits (Van *et al*., 2021; Wang *et al*., 2021;

Xiao *et al*., 2021; Vijay *et al*., 2024), and of deltamethrin in chickpea, tomato and bell pepper (Sharma *et al*., 2023; Majumder *et al*., 2024c; Singh *et al*., 2024). However, differences in crop matrix, storage/environmental conditions, application practices and sampling intervals limit direct comparison of half-lives among these studies. Moreover, the present study included only 0, 90 and 180-day sampling points; hence, the fitted first-order model describes the observed long-term decline but provides limited information on the initial dissipation phase or individual degradation processes. Future studies should therefore incorporate more frequent sampling during the early storage period and investigate transformation products using LC-MS/MS or high-resolution mass spectrometry. Controlled hydrolysis, photolysis, microbial degradation, sorption/desorption and mass-balance studies would further clarify the mechanisms responsible for residue loss. Assessment of transformation products and their persistence, mobility and toxicity would also be necessary before drawing conclusions regarding environmental contamination, food-chain effects or broader safety implications. Thus, the present study provides baseline information on long-term residue dissipation in stored field pea seeds, while the underlying degradation pathways and environmental fate remain subjects for future investigation.

Conclusion: The dissipation of emamectin benzoate and deltamethrin residues in stored field pea seeds followed first-order kinetics, with residues of emamectin benzoate dissipating from 0.504 mg kg^{-1} at 2 h after treatment to 0.155 mg kg^{-1} at 180 days ($R^2 = 0.896$), while deltamethrin dissipated from 0.412 to 0.127 mg kg^{-1} over the same period ($R^2 = 0.936$). The study establishes a benchmark for the long-term dissipation behaviour of both pesticides in stored field pea seeds and indicates the survival of quantifiable residues over prolonged storage. These findings provide useful residue data for storage-related residue management and support the need for further studies under different storage conditions to better understand factors influencing pesticide dissipation.

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Disclosure statement: The authors declare to have no conflicts of interest related to this study.

Data availability statement: The datasets generated and/or analysed during the present study are available from the corresponding author upon reasonable request.

Ethical consent: This study did not involve human subjects or vertebrate animals. All experiments were conducted exclusively on field pea seeds using LC-MS/MS and GC-μECD which are not governed by the regulations of Institutional Review Boards (IRB) or animal ethics committees. Consequently, ethical approval and informed consent were not required. Nevertheless, all experimental research were performed in compliance with established institutional and national guidelines for laboratory, ensuring ethical handling and minimizing unnecessary harm to experimental organisms. The study adheres to the ethical and publication standards of the journal and aligns with internationally accepted norms for responsible scientific conduct.

Author's contributions: ANS & SM - conceptualization, methodology, data curation, analysis, and original draft preparation. MGT, PAD, JH, D, PY, NA, KS, & RK - review and editing. SSD - interpretation, original draft preparation, review and editing. All authors contributed equally in this research and approved the final manuscript procedures.

Declaration: The authors declare that no generative artificial intelligence (AI) or AI-assisted tools were used at any stage of manuscript preparation, data analysis, figure generation, or publication process. All content presented in this manuscript represents original scholarly work carried out entirely by the authors.

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