

Quantitative analysis of synthetic pesticide residues in soil samples from a brinjal field using high-performance liquid chromatography

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Abstract

An experiment was conducted at the main Agricultural Research Field of Lovely Professional University in Phagwara, Punjab, from July 2023 to 2024 to analyze the residues of synthetic insecticides in soil samples from a brinjal field using high-performance liquid chromatography (HPLC). The treatment consisted of synthetic pesticides applied at recommended dosages: T₀: Control (water-spray), T₁: Cartap-hydrochloride (50% SP) at 500 g a.i. ha⁻¹, T₂: Chlorpyrifos (20% EC) at 200 g a.i. ha⁻¹, T₃: Deltamethrin (2.8% EC) at 15 g a.i. ha⁻¹. The results were compared to the FAO and EU maximum residue limits (MRL). The findings indicated that T₁ showed the highest initial deposit (1.15 mg/kg), followed by T₂ (0.62 mg/kg). The percent dissipation rates were 91.08%, 79.60%, and 76.46% on the seventh, fifth, and third days after applying T₁, T₂, and T₃, respectively. The degradation of different insecticidal treatment residues was observed between three to seven days after application. The results concluded that the residues of these synthetic insecticides dissipated quickly, leaving no detectable traces in soil.

Key words: Brinjal, chlorpyrifos, cartap-hydrochloride, deltamethrin, HPLC, residues, soil.

Introduction

Brinjal (*Solanum melongena* L.) is susceptible to various insect pests during the growing season, damaging both the shoot and fruit. The most important vegetable in India is brinjal, which is eaten raw in salads and popular among vegetarians and non-vegetarians (Singh *et al.*, 2024). Since it is consumed raw or unpeeled, it is necessary to determine its residual status. The conventional method of controlling pest attacks is the application of synthetic pesticides, which can potentially cause health issues and environmental contamination. In various regions of India, farmers exceed the recommended limits of pesticide concentrations, making it crucial to evaluate the persistence of pesticides (Mahala and Yadav, 2024). The most devastating insect pest of brinjal is *Leucinodes orbonalis*, which can potentially cause significant yield losses. The main challenge with this pest is that once it penetrates the fruit, it becomes difficult to manage, which is a key reason for the predominant application of pesticides during the growth stages of brinjal. In developing countries like India, where a significant portion of the population lives below the poverty line, food safety requires effective techniques to reduce pesticide contamination.

As soil is a necessary resource for life on Earth, maintaining its quality and health is important, as it takes a long time to renew within a human lifetime (Brinco *et al.*, 2023). As a result, several policy frameworks address the issue of soils. Without considering soil, at least seven of the United Nations Sustainable Development Goals (SDGs) cannot be achieved, including three directly (SDGs 3, 13, and 15) and four indirectly (SDGs 2, 6, 11, and 12) (Lal *et al.*, 2021). Therefore, one of the main causes of the deteriorating condition of soil quality is degradation caused by man-made

pollutants. Pesticides are significant pollutants in agricultural soils due to their excessive use and potential adverse effects on various organisms, including humans (Sefiloglu *et al.*, 2021).

A major drawback of pesticide application is the potential for residue to persist in food at levels exceeding Maximum Residue Limits (MRLs). Analyzing pesticide residues in soil is a crucial aspect of environmental monitoring. The residual presence of certain pesticides in soil post-application and their degradation byproducts constitutes a critical environmental issue. These substances can contaminate the food chain, facilitate biomagnification, induce unfavourable health consequences, disrupt microbial populations, and transfer through processes such as leaching and runoff to other environmental compartments, including water. In humans, acute pesticide toxicity (intense exposure over a short duration) is rarely a concern; however, chronic exposure (minimal dosages over an extended period) is an emerging concern for both agricultural labourers and the general population (Pathak *et al.*, 2022). Pesticide monitoring in soil is crucial for human health to identify contamination sources, track the production and distribution of banned insecticides, and determine the persistence of certain pesticides. Nonetheless, specific pesticide residues may exhibit minimal direct toxicity to humans, yet remain extremely hazardous to environmental systems.

Materials and methods

Chemicals and reagents: The standard chemical compounds cartap hydrochloride, chlorpyrifos, and deltamethrin were acquired from Sigma-Aldrich, Kolkata, India. HPLC grade distilled water, HPLC grade acetonitrile, acetonitrile (AR and HPLC grade), activated charcoal, hexane (AR grade), anhydrous

sodium sulphate (AR grade), and dichloromethane (AR grade) were acquired from Loba Chemie Pvt. Ltd., Mumbai, India. A reagent blank was conducted prior to the sample analysis to evaluate the accuracy of the chemicals and standards.

Details of experiment: A field experiment was conducted at Lovely Professional University, Phagwara, Punjab, from July 2023 to 2024, using Navkiran brinjal seeds. Four treatments were tested: T₀: Control (water-spray), T₁: Cartap-hydrochloride 50% SP (500 g a.i. ha⁻¹), T₂: Chlorpyrifos 20% EC (200 g a.i. ha⁻¹), and T₃: Deltamethrin 2.8% EC (15 g a.i. ha⁻¹) in a Randomized Complete Block Design (RCBD) with three replications. The net area of the single plot was 8.10 m². The cultivation followed recommended agronomic practices with an NPK application of 122.5:122.5:29.5 kg/ha, 25 t/ha FYM, and disease management strategies. The physico-chemical parameters of the experimental field's soil were analyzed following standard procedures. The insecticidal treatments were administered using a knapsack sprayer with a hollow cone nozzle for foliar application. Soil samples from the brinjal field were collected from each insecticidal treatment plot and control plot at 0 (2 hour), 3, 5, 7 and 10 days post-spraying, placed in sampling bags, and transported to the laboratory for further examination. The MRLS were established according to various regulatory bodies, including the Food Safety and Standards Authority of India (FSSAI) and the European Commission (EU). Soil samples were individually collected from 10-15 locations within each treatment plot using a tube auger to a depth of approximately 10-15 cm; the soil from each location was combined and sieved, with extraneous materials, such as stones and pebbles, eliminated. Following comprehensive mixing, a subsample of about 1 kg was extracted from each pooled sample of every treatment plot and transferred to the laboratory.

Physicochemical properties of the experimental field: This study focused on the pooled analysis of 2023 and 2024 for the quantitative assessment of synthetic pesticide residues in soil samples from a brinjal field. The Physicochemical characteristics were assessed to enhance the understanding of soil conditions. The physicochemical properties of the soil are presented in Table 1. The soil pH was measured at 6.7, indicating that it is mildly acidic. The electrical conductivity in the soil of the brinjal field was measured at dSm⁻¹. The soil texture in the experimental field was sandy loam, with sand, silt, and clay percentages recorded at 79%, 12%, and 10.80%, respectively. The organic carbon content in the experimental field was measured at 1.6%, indicating that the soil is rich in organic carbon. The brinjal field's N:P:K content was measured at (439: 49.6: 146 kg ha⁻¹), indicating a

Table 1. Physicochemical properties of the experimental field.

Soil parameters	Value
Soil pH	6.7
Electrical conductivity (dSm ⁻¹)	367
Sand (%)	79
Silt (%)	12
Clay (%)	10.80
Soil Texture	Sandy loamy
Organic Carbon (%)	1.6
Available Nitrogen (kg/ha)	439
Available Phosphorus (P ₂ O ₅) (kg/ha)	49.6
Available Potassium (K ₂ O) (kg/ha)	146

high phosphorus concentration in the soil, while nitrogen and potassium levels were classified as medium.

Laboratory Processing of Samples: The sample preparation for residual analysis was followed the method described by Pandit *et al.* (2016). Fifty grams of soil from each treatment were mixed with 100 mL of acetonitrile-water (4:1 v/v) and shaken for one hour. The mixture was filtered, then extracted again with 50 mL acetonitrile. Activated charcoal (500 mg) was added to the filtrate, shaken for 30 minutes, and filtered through Whatman No. 1 paper. The solution was concentrated to 5 mL using a rotary vacuum evaporator. After transferring to a separating funnel, 50 mL of hexane and 50 mL of NaCl solution were added, and the aqueous layer was collected. This layer was partitioned three times with 50 mL dichloromethane and dried over Na₂SO₄. The extract was evaporated, reconstituted in HPLC-grade acetonitrile, filtered, and adjusted to 5 mL.

Recovery study: The accuracy of the specified analytical method and the efficacy of the extraction and clean-up methods were validated by recovery tests conducted in the laboratory. To 50 g of field soil, 10 mL of analytical insecticide solutions at concentrations of 2.0, 1.0, and 0.5 mg kg⁻¹ were added, mixed thoroughly, and kept for 15 minutes. The pesticide residues in the fortified samples were subsequently removed, purified, and quantified using the aforementioned procedure. The limit of detection was determined by identifying the lowest standard concentration level.

Residual analysis by HPLC: The HPLC analysis was conducted utilizing a Waters system comprising a Waters 515 HPLC pump regulated by the Waters pump control module II, a Waters 2487 dual λ absorbance detector, and a Sunfire C₁₈ 5mm reverse phase column (4.6 x 250 mm). The system was operated with Waters Empower Pro software, with a wavelength of 270 nm, an injection volume of 20 µL and an isocratic solvent system of acetonitrile at a flow rate of 1.0 mL min⁻¹. Cartap-hydrochloride, chlorpyrifos, and deltamethrin had a retention time of 7.80, 7.62, 8.51 minutes under the specific conditions.

Statistical analysis: The data were analysed with Duncan's Multiple Range (DMR) test. The value equivalent to $P < 0.05$ was established as the criterion for statistical significance (Duncan, 1955). All data are presented as mean ± standard deviation (SD).

Results and discussion

Recovery study: In T₁ (Cartap-hydrochloride), the percent recoveries ranged from 75.33% to 88.17%. In T₂ (Chlorpyrifos), the percent recoveries also ranged from 75.33% to 88.17%. Similarly, in T₃ (Deltamethrin), the percent recoveries varied from 75.33% to 88.17% across different substrates, which was thought to be satisfactory (Table 2). The detection limit was determined to be 0.01 mg kg⁻¹.

Residues status of insecticides in the soil samples of brinjal field

Cartap hydrochloride: Data on cartap hydrochloride residues in soil of brinjal field recorded at different time intervals revealed that initial deposit of (1.15 mg kg⁻¹) recorded at 0 day (2 hours after application), which was reduced to (0.62 mg kg⁻¹), (0.18 mg kg⁻¹) and (0.10 mg kg⁻¹) at 3rd, 5th and 7th days after treatments, respectively during 2023 (Table 3 and Fig. 1). The corresponding

Table 2. Recovery study on cartap-hydrochloride, chlorpyrifos and deltamethrin residues from experimental field soil

Treatments	Amount fortified (mg kg ⁻¹)	Percent recovery (Mean ± SD)
T ₁ : Cartap-hydrochloride	0.01	88.17 ^a ± 1.04
	0.10	83.33 ^{ab} ± 2.08
	0.20	75.33 ^{ab} ± 3.06
T ₂ : Chlorpyrifos	0.01	84.33 ^a ± 1.26
	0.10	81.00 ^{ab} ± 4.36
	0.20	76.00 ^{bc} ± 4.00
T ₃ : Deltamethrin	0.01	80.17 ^{ab} ± 1.26
	0.10	78.33 ^{bc} ± 4.73
	0.20	81.33 ^{ab} ± 5.03

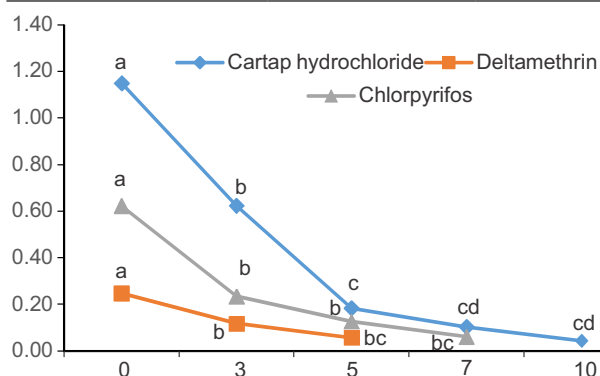


Fig. 1. Residues (mg/kg) of various insecticidal treatments at different days interval.

losses in insecticidal residues were (45.76%), (83.96%) and (91.08%), respectively. Furthermore, the data indicated that cartap hydrochloride, when applied at the recommended dosage, decreased below the determination limit (BDL) by the tenth day post-application. The maximum residue limit (MRL) for cartap hydrochloride residues on brinjal has not been established by either the Prevention of Food Adulteration (PFA) in India or the Codex Alimentarius Commission (FAO/WHO). Nonetheless, the United Kingdom and other European Union nations have suggested a maximum residue limit (MRL) of 0.02 mg/kg. Although cartap hydrochloride undergoes quick photodecomposition after application, it maintains residual activity in the field due to its translaminar action and swift penetration into leaf tissue. It is not only very effective, but as it originates from a biological source, it is a bio-rational product suitable for environmentally sustainable application in integrated pest control systems. Cartap hydrochloride undergoes rapid degradation in soil and is quickly susceptible to photodegradation at the soil surface, with a half-life of 8 hours to 1 day. The degradation of cartap hydrochloride in soils is attributed to microbial activity, as it remains undegraded in sterile soil (Rao *et al.*, 2014). The current investigation similarly found no remains in the soil after seven days. The findings align with certain research conducted on brinjal (Rao *et al.*, 2014).

Chlorpyrifos: Data on chlorpyrifos residues in soil of brinjal field recorded at different time intervals revealed that initial deposit of (0.62 mg kg⁻¹) recoded at 0 day (2 hours after application), which was reduced to (0.23 mg kg⁻¹) and (0.13 mg kg⁻¹) at 3rd and 5th days after treatments, respectively during 2023 (Table 3 and Fig. 1). The corresponding losses in insecticidal residues were (62.44%) and (79.60%), respectively. Further, the data also revealed that chlorpyrifos applied at the recommended dose reached below the determination limit (BDL) at the seventh

day after application. However, the European Commission has proposed a MRL value of 0.01 mg kg⁻¹. The results were in close conformity with the results of (Yeasmin *et al.*, 2019), who observed that the chlorpyrifos residue was not detectable at the seventh day after application.

Table 3. Residues of cartap-hydrochloride, chlorpyrifos and deltamethrin in soil of brinjal field.

Treatment	Days after application	Residue (mg kg ⁻¹)	Percent dissipation
T ₁ : Cartap-hydrochloride	0 (2 h)	1.15 ^a ± 0.05	-
	3	0.62 ^b ± 0.03	45.76 ± 2.44
	5	0.18 ^c ± 0.04	83.96 ± 4.26
	7	0.10 ^{cd} ± 0.03	91.08 ± 2.30
	10	BDL	-
T ₂ : Chlorpyrifos	0 (2 h)	0.62 ^a ± 0.02	-
	3	0.23 ^b ± 0.03	62.44 ± 4.78
	5	0.13 ^b ± 0.02	79.60 ± 3.98
	7	BDL	-
	10	BDL	-
T ₃ : Deltamethrin	0 (2 h)	0.25 ^a ± 0.03	-
	3	0.12 ^b ± 0.02	52.90 ± 4.18
	5	0.06 ^{bc} ± 0.02	76.46 ± 8.51
	7	BDL	-
	10	BDL	-

Deltamethrin: Data on deltamethrin residues in soil of brinjal field recorded at different time intervals revealed that initial deposit of (0.25 mg kg⁻¹) recorded at 0 day (2 hours after application), which was reduced to (0.06 mg kg⁻¹) at 3rd days after treatments, respectively during 2023 (Table 3 and Fig. 1). The corresponding losses in insecticidal residues were (76.46%), respectively. Further, the data also revealed that deltamethrin applied at the recommended dose reached below the determination limit (BDL) five days after application. However, the European Commission has proposed a MRL value of 0.40 mg kg⁻¹. The results were in close conformity with the results of Panickar and Jhala (2006), who observed that the deltamethrin residue was not detectable at 4th days after application.

Half Life kinetics: The dissipation kinetics of the various insecticidal treatments applied to the soil were evaluated by plotting a graph of the logarithm of residues against the time interval in days. The equations of the best-fit curves were estimated using the maximum correlation coefficient (Fig. 2). The persistence of any pesticide is often determined in terms of its half-life (T_{1/2}) or DT₅₀, which refers to the time required

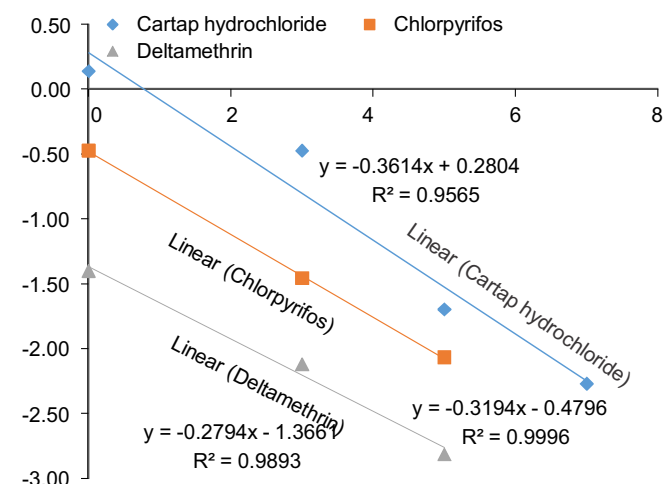


Fig. 2. The degradation kinetics of different insecticide residues in the soil of brinjal field.

for an insecticide to degrade to 50% of its initial amount. The dissipation of T₁ (Cartap-hydrochloride), T₂ (Chlorpyrifos) and T₃ (Deltamethrin) treatments on brinjal follows first-order kinetics. The half-life (T_{1/2}) values of the T₁, T₂ and T₃ treatments were estimated according to Hoskins (1961) and were observed to be 1.93, 2.17, and 2.70 days, respectively, when applied at the recommended dosages (Table 4). Similarly, Lu *et al.* (2014) observed a half-life of 7.79 days for chlorpyrifos in soil. Zhang *et al.* (2012) recorded a half-life of 1.35 days for chlorpyrifos in paddy fields. For cartap hydrochloride, Numan *et al.* (2018) reported half-lives ranging from 2 to 7 days in soil. Rosendahl *et al.* (2009) reported half-lives ranging from 5.8 to 15 days in soil for deltamethrin. Faster dissipation was primarily stimulated by soil moisture, with abiotic factors contributing 77.3% to 85.2% of the chlorpyrifos dissipation in soil (Kumar *et al.*, 2017).

Table 4. Half-life kinetics of different insecticidal treatments in soil of brinjal field

Treatments	Equations	R ² values	Half-life
Cartap-hydrochloride	$y = -0.3446x + 0.2354$	0.9755	1.93
Chlorpyrifos	$y = -0.3313x - 0.4595$	0.9987	2.17
Deltamethrin	$y = -0.2906x - 1.3645$	0.9891	2.70

The findings concluded that synthetic insecticides, including Cartap-hydrochloride, Chlorpyrifos, and Deltamethrin, dissipated quickly in the soil after application. The highest initial pesticide deposit was observed with Cartap-hydrochloride (T₁), followed by Chlorpyrifos (T₂). The dissipation rates of residues were high, with over 90% dissipation observed within a week. The half-life values of different insecticidal treatments ranged from 1.93 to 2.70 days in the soil of the brinjal field. The study indicates that these insecticide residues degrade rapidly, leaving no detectable traces in the soil within a short period, ensuring that the levels remained within acceptable limits set by FAO and EU standards. Further investigation should be conducted on the dissipation pattern of synthetic insecticides in brinjal plants and fruit samples so that farmers can obtain information regarding the half-life and waiting period of insecticides on brinjal plants and fruits.

Conflict of Interest: The contributors hereby affirm that they have no conflicts of interest.

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References

Brinco, J., P. Guedes, M.G. da Silva, E.P. Mateus and A.B. Ribeiro, 2023. Analysis of pesticide residues in soil: A review and comparison of methodologies. *Microchem. J.*, 195: 109465.

- Duncan, D.B. 1955. Multiple range and multiple F tests. *Biometrics*, 11(1): 1-42.
- Hoskins, W.M. 1961. Mathematical Treatment of Loss of Pesticide Residues. *FAO Plant Prot. Bull.*, 9: 163-8.
- Kumar, U., J. Berliner, T. Adak, P.C. Rath, A. Dey, S.S. Pokhare, N.N. Jambhulkar, P. Panneerselvam, A. Kumar and S.D. Mohapatra, 2017. Non-target effect of continuous application of chlorpyrifos on soil microbes, nematodes and its persistence under sub-humid tropical rice-rice cropping system. *Ecotoxicol. Environ. Saf.*, 135: 225-235.
- Lal, R., J. Bouma, E. Brevik, L. Dawson, D. J. Field, B. Glaser, R. Hatano, A.E. Hartemink, T. Kosaki, B. Lascelles and C. Monger, 2021. Soils and sustainable development goals of the United Nations: An International Union of Soil Sciences perspective. *Geoderma Reg.*, 25: e00398.
- Lu, M.X., W.W. Jiang, J.L. Wang, Q. Jian, Y. Shen, X.J. Liu and X.Y. Yu, 2014. Persistence and dissipation of chlorpyrifos in Brassica chinensis, lettuce, celery, asparagus lettuce, eggplant, and pepper in a greenhouse. *PLoS One*, 9(6): p.e100556.
- Mahala, A. and U. Yadav, 2024. A Review on efficacy effect of newer insecticides and biopesticides against brinjal fruit and shoot borer (*Leucinodes orbonalis*) Guenee. *JSRR*, 30(6): 496-501.
- Numan, A., M. Khan, R. Uddin, M. Rahman, M. Bhuiyan and N. Akter, 2018. Risk assessment of commonly used major pesticides for tomato (*Solanum lycopersicum* L.) cultivation in Bangladesh. *Adv in Nutr Fd Sci: ANAFS*-109.
- Pandit, G.K., G.S. Krushna, N. Chowdhury and J. Ghosh, 2016. Dissipation of imidacloprid residues in okra leaves, fruits and soil in the northern region of West Bengal. *Pesticide Research J*, 28(1): 20-24.
- Panickar, B.K. and R.C. Jhala, 2006. Residues of insecticides in/on cowpea pods [*Vigna unguiculata* (Linnaeus)]. *Indian J. Entomol.*, 68(3): 311-312.
- Pathak, V.M., V.K. Verma, B.S. Rawat, B. Kaur, N. Babu, A. Sharma, S. Dewali, M. Yadav, R. Kumari, S. Singh and A. Mohapatra, 2022. Current status of pesticide effects on environment, human health and it's eco-friendly management as bioremediation: A comprehensive review. *Front. Microbiol.*, 13: p.962619.
- Rao, S.V.C.S., S. Swarupa, D. Ravindranth and A.H. Reddy, 2014. Dissipation of cartap hydrochloride in brinjal (*Solanum melongena*). *JMEST*, 1(5): 79-81.
- Rosendahl, I., V. Laabs, C. Atcha-Ahowé, B. James and W. Amelung, 2009. Insecticide dissipation from soil and plant surfaces in tropical horticulture of southern Benin, West Africa. *J. Environ. Monit.*, 11(6): 1157-1164.
- Sefiloglu, F.O., U. Tezel and I.A. Balcioglu, 2021. Validation of an analytical workflow for the analysis of pesticide and emerging organic contaminant residues in paddy soil and rice. *J. Agric. Food Chem.*, 69(11): 3298-3306.
- Singh B., T. Chaubey, R.K. Singh, D.K. Upadhyay, Aastik Jha and S. Pandey, 2024. Genetic variation in phenatic traits of extant varieties of brinjal (*Solanum melongena* L.). *J. Appl. Hort.*, 26(1): 107-111.
- Yeasmin, M., M.A. Rahman, M.A. Islam and M.S. Hossen, 2019. Quantitative analysis of selected pesticide residues in brinjal using gas chromatography. *IJSRST*, 5(4): 159-167.
- Zhang, X., Y. Shen, X.Y. Yu and X.J. Liu, 2012. Dissipation of chlorpyrifos and residue analysis in rice, soil and water under paddy field conditions. *Ecotoxicol. Environ. Saf.*, 78: 276-280.

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