Research Inventy: International Journal of Engineering And Science
Vol.15, Issue 7 (July 2025), PP 57-60
Issn (e): 2278-4721, Issn (p):2319-6483, www.researchinventy.com

Development of a High-Performance Liquid Chromatography (HPLC) Method for Determining Pesticide Residues in Agricultural Soil: A Case Study in the Tea-Growing Region of Thai Nguyen, Vietnam

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Abstract
This study developed an HPLC method to detect and measure pesticide residues in soil from tea-growing areas in Thai Nguyen, Vietnam. The method was used to analyze common pesticides such as chlorpyrifos, which is an organophosphate, and cypermethrin, which is a pyrethroid. The method was improved to be highly sensitive with low detection limits (0. 02–0. 03 µg/g) and a recovery rate of 85–95%. The results showed that chlorpyrifos was found in 70% of the soil samples with an average concentration of 0. 15 µg/g, while cypermethrin was found in 50% of samples at 0. 08 µg/g. Higher levels of these pesticides were found near industrial areas. This study provides important information for environmental management and suggests alternatives to chemical pesticides, like biological methods and organic farming.

Date of Submission: 25-07-2025 Date of acceptance: 03-08-2025

I. Introduction

Tea farming is a major part of agriculture in Thai Nguyen, Vietnam, which is known as the country's "tea capital". The region has more than 23,000 hectares of tea farms and produces about 250,000 tons of tea each year (General Statistics Office of Vietnam, 2023). Thai Nguyen plays an important role in the national agricultural economy and in the export of tea. However, farmers often use pesticides, like chlorpyrifos (an organophosphate) and cypermethrin (a pyrethroid), to protect tea plants from pests and weeds. While these chemicals help in growing crops, they can accumulate in the soil and cause environmental issues such as pollution, soil damage, and food safety problems through the food chain. Pesticide residues in soil not only harm the environment but can also affect human health, especially if they get into water sources or are found in tea products.

Checking pesticide levels in soil is important for sustainable farming and meeting international environmental standards like the World Health Organization’s (WHO) maximum residue limits (MRLs). Although previous studies have examined pesticide pollution in areas like the Mekong Delta and the Red River Delta, there has been little research on soil in Thai Nguyen’s tea-growing areas. Most of the earlier studies focused on water or food products, and soil, where these chemicals often remain, has not been given enough attention. In addition, Vietnamese labs usually use methods like gas chromatography (GC) or liquid chromatography-mass spectrometry (LC-MS), which require expensive equipment that is not easily available in local labs.

High-performance liquid chromatography (HPLC) with a UV-Vis detector is a good method for finding organic chemicals like pesticides. It is better than LC-MS because it is more sensitive, gives the same results each time, and is less expensive. HPLC works by separating different chemicals in a sample based on how they interact with two parts: a fixed part, like a C18 column, and a moving part, such as solvents like acetonitrile or methanol. The UV-Vis detector checks for chemicals by measuring how much light they absorb. This helps count the amount of chemicals like chlorpyrifos, which is measured at 230 nm, and cypermethrin, which is measured at 254 nm. Compared to GC, HPLC is better for chemicals that don't evaporate easily or can't be heated, like some organophosphate pesticides. However, using HPLC on soil samples needs extra steps to clean up the sample and remove natural materials in the soil that might interfere with the results.

Earlier research in Vietnam found chlorpyrifos and cypermethrin in agricultural soil, with amounts ranging from 0.01 to 0. 5 µg/g. But many of these studies didn’t provide information on how well the methods work or how low the levels of chemicals can be detected, making it hard to trust the results or compare them. Also, there’s no study in Thai Nguyen that has worked on improving HPLC methods for tea-growing soil. This area has different soil types and farming practices that might affect the results. Another thing missing is information on how pesticide levels spread across tea-growing areas, especially near factories where pollution might spread.

This study fills these gaps by doing three things: (1) creating a better HPLC method for tea-growing soil in Thai Nguyen with improved steps for cleaning and extracting; (2) finding out how much chlorpyrifos and cypermethrin are in the soil, how much contamination there is, and how the levels change in different areas; and (3) giving a base set of data to help manage soil quality and set rules for how pesticides are used.
Using HPLC with a UV-Vis detector makes sure the results are accurate and detailed, and it fits with local labs in Vietnam. The results will help improve environmental management and keep the tea from Thai Nguyen safe to sell both locally and abroad.

II. Materials and methods

2.1. Sample collection

Study Area: Ten locations where tea is grown in Thai Nguyen, including Tan Cuong, Phuc Xuan, and nearby areas.
Soil Sampling: Soil samples were taken from the top layer, 0 to 20 cm deep, in tea fields.
Each sample weighed about 500 grams and was collected during the dry season, which was November 2024, to keep things consistent.
Sample Preservation: The samples were kept in the fridge at 4°C and tested within 48 hours.

2.2. Sample preparation

Extraction: The soil samples were mixed with a solution of acetonitrile and water in a 80:20 ratio.
This mixture was then used to extract the samples using ultrasonic energy for 30 minutes.
Cleanup: To remove any unwanted chemicals, the extracts were passed through C18 cartridges, which are part of a solid-phase extraction method.
Concentration: The extracts were made thicker using a nitrogen evaporator before they were ready for HPLC analysis.

2.3. HPLC analysis

Equipment: The analysis was done on an Agilent 1260 Infinity II HPLC system that uses a UV-Vis detector to identify chemicals.
Chromatographic Column: A C18 column with dimensions 250 mm by 4.6 mm and a particle size of 5 micrometers was used.
Mobile Phase: A mixture of acetonitrile and water in a 70:30 ratio was used, and it flowed through the column at 1 mL per minute.
Detection Wavelengths: The detector looked for chlorpyrifos at 230 nm and cypermethrin at 254 nm.
Retention Times: Chlorpyrifos appeared at about 6.5 minutes, and cypermethrin appeared at around 8. 2 minutes.
Calibration Curve: Standard solutions were made with pesticide concentrations ranging from 0.
01 to 10 micrograms per milliliter to create a straight line that helps with accurate measurement.

2.4. Method validation

Limit of Detection (LOD): This was determined by looking at the signal-to-noise ratio, where a ratio of 3 was used as the cutoff.
Limit of Quantification (LOQ): This was found by using a signal-to-noise ratio of 10.
Reproducibility: To check how reliable the method was, each sample was tested five times, and the relative standard deviation (RSD) was calculated.
Recovery Efficiency: This was tested by adding known amounts of pesticide standards to soil samples at three levels: 0.1, 1, and 5 micrograms per gram.

III. Results and discussion

3.1. Method performance

The HPLC method we developed worked well for testing pesticide levels in soil.
We found that we could detect chlorpyrifos at 0. 02 µg/g and cypermethrin at 0. 03 µg/g. For accurate measurement, the lowest amount we could reliably measure was 0. 06 µg/g for chlorpyrifos and 0. 09 µg/g for cypermethrin. The method was consistent, with the amount of variation (RSD) being under 5% for both pesticides, which shows it is stable and dependable. When we checked how well the method recovered the pesticides from soil samples, it worked well across different amounts tested (0. 1, 1, and 5 µg/g), with recovery rates between 85 and 95%. This is better than a previous method that used gas chromatography (GC), which had recovery rates of 70 to 80% for chlorpyrifos. These results show that using ultrasound to help extract the pesticides along with a C18 cleanup step helps reduce the effect of natural soil materials found in tea-growing areas.

3.2. Analysis of real samples

Table 1 shows the results of testing pesticide levels in 10 soil samples taken from tea-growing areas in Thai Nguyen.

Table 1: Concentrations of pesticide residues in tea-growing soils of Thai Nguyen

Location	Chlorpyrifos (μg/g)	Cypermethrin (μg/g)	Notes
Tan Cuong 1	0.18	0.07	Near industrial zone
Tan Cuong 2	0.14	0.05	Near industrial zone
Phuc Xuan 1	0.09	ND	Rural area, far from industrial zone
Phuc Xuan 2	0.07	ND	Rural area, far from industrial zone
Dai Tu 1	0.22	0.12	Near industrial zone
Dai Tu 2	0.16	0.09	Near industrial zone
Phu Luong 1	ND	ND	Rural area, far from industrial zone
Phu Luong 2	ND	ND	Rural area, far from industrial zone
Dong Hy 1	0.13	0.06	Near industrial zone
Dong Hy 2	0.10	ND	Near industrial zone

Notes: ND = Not detected (below LOD).

Chlorpyrifos was found in 70% of the samples (7 out of 10), with an average level of 0.15 μg/g, and it was found at levels between 0. 07 and 0. 22 μg/g. Samples taken near industrial areas like Tan Cuong, Dai Tu, and Dong Hy had higher levels, with the highest level at Dai Tu 1 being 0. 22 μg/g, which is above the WHO safety level of 0. 1 μg/g. Samples from rural areas that are far from industrial zones, such as Phu Luong and Phuc Xuan, had no detectable levels or much lower levels. Cypermethrin was found in 50% of the samples (5 out of 10), with an average of 0.08 μg/g, and the levels were between 0. 05 and 0. 12 μg/g. Like chlorpyrifos, higher amounts were found near industrial zones, while rural samples had no detectable levels.

Comparison with Standards: At three sites (Tan Cuong 1, Dai Tu 1, and Dong Hy 1), chlorpyrifos levels were above the WHO safety standard, but cypermethrin levels were below the threshold (0.2 μg/g). Compared with a study inMekong Delta, pesticide levels in Thai Nguyen were lower on average (0. 3 μg/g for chlorpyrifos), but there are still concerns in areas close to industrial zones. The HPLC method created was very sensitive and accurate, with lower limits for detecting and measuring pesticides compared to earlier GC methods used in Vietnam. The method had a recovery rate of 85–95%, meaning the steps for collecting and cleaning soil samples worked well in tea-growing areas. These areas often have a lot of natural organic matter, which can sometimes cause problems with test results. The analysis found that Thai Nguyen has high levels of pesticide contamination, especially near industrial zones. This suggests that more attention is needed. In some areas, the amount of chlorpyrifos was higher than the WHO limit, showing that pesticide use may not be properly controlled. This could be due to overuse or mixing with chemicals from nearby industries. Cypermethrin was also found near industrial areas, though at lower levels. This indicates that these pesticides may build up in the soil over time.

Compared to previous studies, pesticide levels in Thai Nguyen were lower than those in the Mekong Delta, where chlorpyrifos levels reached up to 0. 5 μg/g in rice soils. This might be because tea farming typically uses fewer pesticides than rice farming, and Thai Nguyen has different soil conditions. However, higher contamination near industrial zones suggests possible pollution from these areas or improper pesticide use. This study provides new information on how pesticides are spread across Thai Nguyen, an area that hasn't been widely studied before.

To reduce the impact of pesticides, the study recommends that farmers and local officials in Thai Nguyen use safer and more environmentally friendly methods. One idea is to promote organic farming, which uses natural fertilizers and pest control methods, such as using animals like wasps or ladybugs to keep pests away from crops instead of chemical pesticides. A study in India found that using natural predators can reduce pesticide use in tea farming by up to 70%. Another option is to use physical tools like pheromone traps or insect nets to control pests without harming the soil. Farmers should also be trained on how to use pesticides correctly, and natural products like neem oil or Bacillus thuringiensis (Bt) should be encouraged.

These are better for the environment and people's health. Local officials should also monitor pesticide use more closely, especially around industrial areas, and carry out regular checks on soil and water quality. Developments in clean tea standards and organic certification can add value to Thai Nguyen tea and protect natural environments and public health. These steps will not only help reduce pesticide pollution but also support sustainable agriculture worldwide.

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