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Research Article

Occurrence and distribution of pesticide residues in coffee growing soil at Lam Ha district, Lam Dong province

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Abstract: Thirty soil samples from 4 communes of Lam Ha district were collected in May and November 2023. The pH, moisture content, organic carbon and mechanical composition were investigated. Two group of pesticides were analyzed including Organophosphorus (Diazinon, Chlorpyrifos, Profenofos, Fenitrothion, Ethoprophos, Glyphosate) and Carbamate (Carbaryl, Mancozeb, Carbosulfan). Maximum concentration of Chlorpyrifos, Profenofos and Carbosulfan founded in soil samples were 391, 63 and 41 μg/kg, respectively. The risk quotient (RQ) of pesticides in coffee growing soils were evaluated for ecological risk assessment. In this study the order from very low to high risk in ascending order from Phi To, Nam Ha, Dong Thanh and Me Linh, respectively.

Keywords: Pesticides in soil; Soil pollutions; Organophosphorus; Carbamates; Risk assessment.

1. Introduction

Lam Dong is a province with favorable natural conditions for growing coffee. Currently, the whole province has a coffee growing area of 172,000 hectares and an output of 515,000 tons. Although it is the second province in the country to accumulate coffee, Lam Dong coffee has the highest average capacity and product in the country. Lam Dong is developing 5 large coffee cultivation areas in the districts of Di Linh, Lam Ha, Bao Lam, Duc Trong, Da Lat city and Lac Duong to form large-scale raw material areas for processing and export.

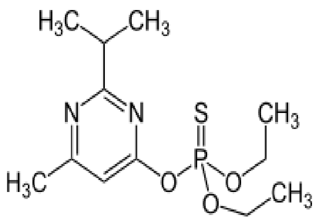
The coffee area of Lam Ha district is currently about 40,000 hectares, the harvest output is about 135,000 tons and the average harvest yield is 3.45 tons/ha. Lam Ha district has been converting new varieties of plants, grafting and improving robusta coffee... to help coffee trees increase productivity. Increasingly intensive farming measures are the main cause affecting the emergence and development of harmful pests and diseases. In coffee farming, farmers regularly treat soil before planting coffee and use plant protection chemicals when pests are detected. Most coffee producing households use chemical plant protection chemicals to prevent pests on coffee plants with a frequency of use of 1 to 4 times a year [1].

Carbamate insecticides included in this research are Carbaryl, Mancozeb and Carbosulfan. The functional group inactivating enzyme acetylcholinesterase causing toxicity to kill insects. Organophosphate group also affect cholinergic poisoning [2–4].

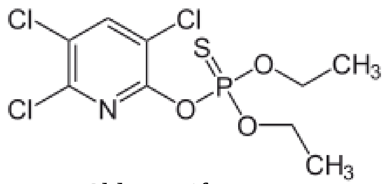
Organophosphorus pesticides poison insects by phosphorylation of the acetylcholinesterase enzyme (AChE) at nerve endings. The result is a loss of available AChE so that the effector organ becomes overstimulated by the excess acetylcholine [2–6]. From field surveys and previous research, six Organophosphorus pesticides including Diazinon, Chlorpyrifos, Profenofos, Fenitrothion, Ethoprophos and Glyphosate were investigated.

Organophosphorus and Carbamate pesticides are widely used in coffee growing areas in Lam Ha district showed in Table 1 [1]. They are highly toxic, persistent in the environment, have high bioaccumulation in humans and organisms. Thus, ethoprophos has been banned from use since March 2018 and Diazinon since March 2018. Pesticides containing Chlorpyrifos must not be produced or imported since October 2019, can only be sold and used until February 12, 2021; Glyphosate only be sold and used until June 30, 2021 in Vietnam [7].

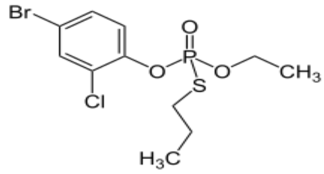
Table 1. Structure of pesticides.



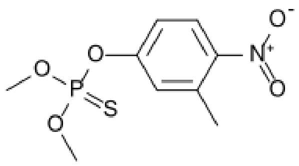
Diazinon



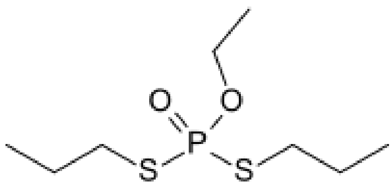
Chlorpyrifos



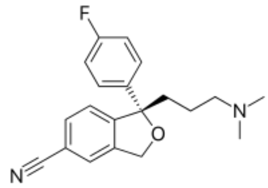
Profenofos



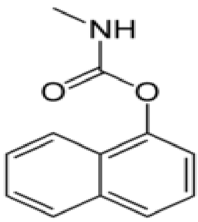
Fenitrothion



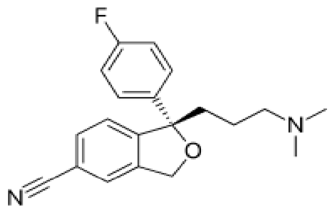
Ethoprophos



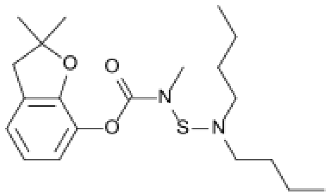
Glyphosate



Carbaryl



Mancozeb



Carbosulfan

Even though there are specific instructions in the agricultural industry, many farmers still use pesticides incorrectly and at higher doses than allowed, leading to them penetrating and remaining in coffee soil, causing risks and long-term harmful effects on human health [7]. Therefore, assessing the level of environmental risks of pesticides residue in coffee growing soil is very necessary.

The study objectives are: (1) Analysis of physicochemical properties of soil samples; (2) Analysis of six organophosphorus and three carbamate pesticides concentration; (3) Evaluate ecological risk of investigated pesticides.

2. Materials and methods

2.1. Study structure

In this research, thirty soil samples were collected and analyzed for physicochemical properties such as pH, moisture contend, organic carbon and the mechanical composition. Concentration of six organophosphorus and three carbamate pesticides were investigated to assess ecological risk. Figure 1 shown the details of study structure.

2.2. Study area

Samples were taken in May 2023 (rainy season) and November 2023 (dry season), each time 15 surface soil samples with a depth of 0-15cm were taken in Lam Ha district as shown in Figure 2: 3 from Phi To, 5 from Nam Ha, 4 from Me Linh and 3 from Dong Thanh communes.

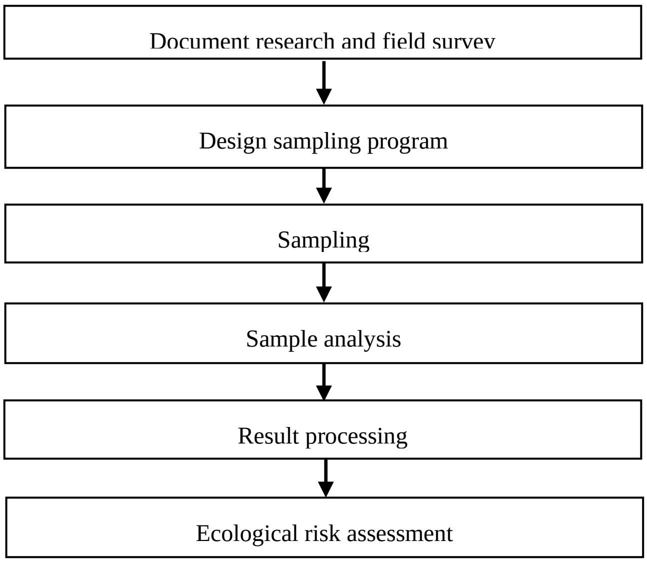


Figure 1. Research diagram.

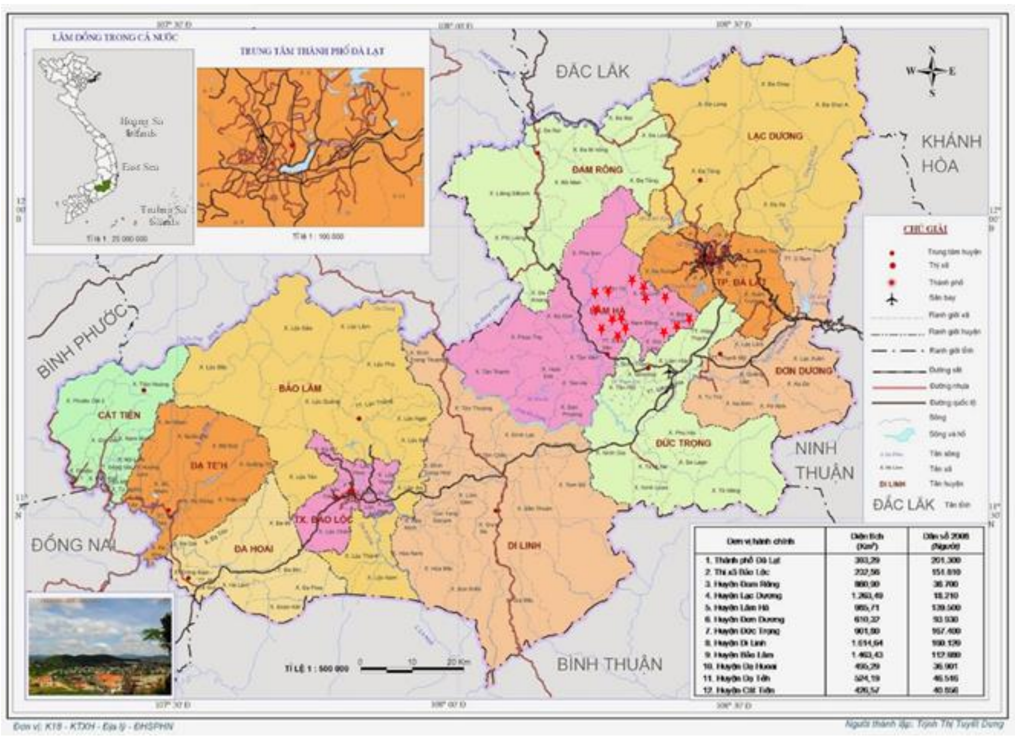


Figure 2. Sampling locations.

2.3. Sampling methods

As a previous research, each coffee garden will take 1 bulk sample with a depth of 0-15cm and a weight of about 1kg. This soil sample is combined from about 5-10 subsamples taken at different locations of the garden to ensure uniformity. These subsamples after removed gravel, leaves, roots were mix well and then followed by the quartering method. Depending on the shape and area of the garden, choose an appropriate sampling method such as the diagonal or zigzag method [8].

2.4. Analysis methods

pH, moisture, organic carbon and mechanical composition of soil samples were determined by Vietnamese standards TCVN 5979:2021, TCVN 4048:2011, TCVN 8941:2011 and TCVN 8567:2010, respectively [8].

There are several extraction methods (shaking, soxhlet, ultrasonic, pressurized liquid extraction, microwave-assisted extraction, solid-phase microextraction and QuEChERS) [10-13], cleaning techniques (SPE, GPC and SFC) [14], and analysis (GC, LC) [15–18] of pesticides in soil, this study chose the soxhlet extraction method, cleaning with Florisil and analysis by GC-MS.

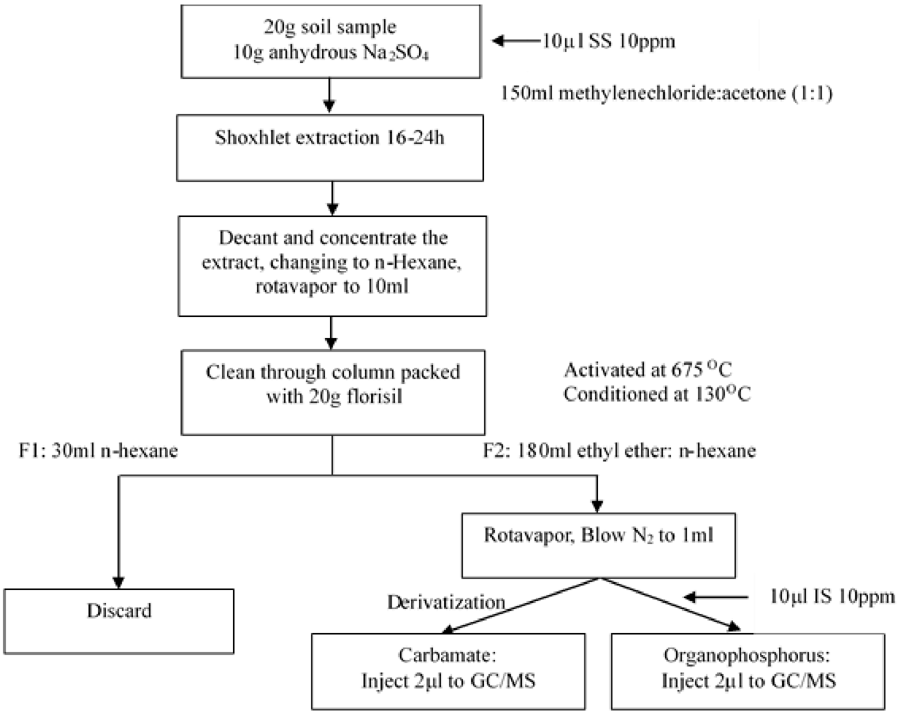


Figure 3. Schematic diagram of pesticides analysis.

Figure 3 describes the diagram of analysis of organophosphorus and carbamate pesticide. 20g of soil samples were extracted with dichloromethane: acetone mixture solvents for 16-24 hours. These extracts were evaporated and transferred to n-Hexane. After cleaned by Florisil column, the eluates were concentrated to 1ml and analyzed by GC-MS.

Derivatization is required for Carbamates before performing analysis. Pesticide concentration of the soil sample is calculated from calibration curve, surrogate standard and internal standard in µg/kg. Soil samples were analyzed according to EPA methods at the chemical and environmental laboratory. The testing laboratory has many years of experience in the field of analysis and has been accredited with ISO/IEC 17025 by the Ministry of Science and Technology and Vimcerts designation by the Ministry of Natural Resources and Environment.

2.5. Ecological risk assessment

To assess the ecological risks of pesticides, in this study, the risk quotient (RQ) is used and calculated according to the following formula:

$$RQ = C/ PNEC \quad (1)$$

where C is the concentration of the pollutant in the sample; PNEC is the concentration value predicted to have no effect on the organism.

$$PNEC = NOEC/ AF \quad (2)$$

where NOEC is the concentration with no effect observed; assessment factor (AF) - factor take into account potential chronic risks.

Assess the levels of ecological risk based on the values of RQ: RQ < 0.01: very low risk; 0.01 ≤ RQ < 0.1: low risk; 0.1 ≤ RQ < 1.0: moderate risk; RQ ≥ 1.0: high risk [8, 19–23].

3. Results and discussion

3.1. Physicochemical properties of soil samples

The pH of 30 soil samples in Lam Ha district in both May and November 2023 was acidic. In general, pH in May (rainy season) is lower than in November (dry season) with lowest in Phi To commune, ranging from 3.52-3.85; then go to Nam Ha commune from 3.90-4.25; Next is Me Linh commune from 4.11-4.36 and the highest is Dong Thanh commune from 4.36-4.58.

Soil moisture in the May was higher than in the November and was not too different between sampling points in the same period. Specifically, the humidity at Phi To ranges from 25.3-34.7%; Nam Ha from 23.7-34.5%; Me Linh from 24.5-38.4% and Dong Thanh from 23.4-33.2%.

Rainwater contains NH_3 which causes the soil to release H^+ and wash away alkaline metals, leading to a decrease in pH. pH and humidity will affect the ability to decompose pesticides in the soil. Organic carbon of 4 communes were ranged of 2.3-3.7%. Phi To ranges from 2.4-2.9%; Nam Ha from 2.3-3.1%; Me Linh from 2.8-3.3% and Dong Thanh from 3.2-3.7%.

Table 2. Physicochemical properties of the soil.

Sample type	pH	Moisture content	OCs	Mechanical components (%)			
				Coarse sand (2-0.2mm)	Fine sand (0.2-0.02mm)	Limon (0.02-0.002mm)	Clay (< 0.002mm)
Rainy seasons	4.05	32.37	2.92	2.85	13.43	28.39	55.33
Dry seasons	4.18	25.37	2.99	2.88	14.23	28.65	54.25

Soil samples in Phi To and Me Linh are clay-rich with the percentage of clay higher than 60% while in Nam Ha and Dong Thanh are mix-clay. There is no significant difference in soil mechanical composition between rainy and dry seasons (Table 2). The average percentages of coarse sand are similar but fine sand, limon and clay are 9.75%, 22.40%, 65.07% in Phi To and Me Linh, and 16.62, 33.67, 46.79% in Nam Ha and Dong Thanh, respectively.

3.2. Pesticide concentration of soil samples

Concentration of Organophosphorus and Carbamate pesticides in soil samples are showed at Table 3 and Table 4.

Table 3. Organophosphorus pesticide concentration.

Sampling time	Sample location	Chlorpyrifos (µg/kg)			Profenofos (µg/kg)		
		Min	Average	Max	Min	Average	Max
May-2023	Phi To	N.D	32.5	61	N.D	N.D	N.D
	Nam Ha	N.D	74.7	158	N.D	N.D	N.D
	Me Linh	119	231	391	N.D	17.3	42.5
	Dong Thanh	129	187	261	N.D	21.0	63
November-2023	Phi To	N.D	29.7	57.5	N.D	9.7	29.0
	Nam Ha	N.D	66.4	129	N.D	N.D	N.D
	Me Linh	78.5	169	294	N.D	7.0	28.0
	Dong Thanh	107	148	195	N.D	17.5	52.5

N.D: Not detected

Table 4. Carbamate pesticide concentration.

Sampling time	Sample location	Carbosulfan (µg/kg)		
		Min	**Average**	**Max**
May-2023	Phi To	N.D	N.D	N.D
	Nam Ha	N.D	3.0	15.0
	Me Linh	N.D	14.0	32.0
	Dong Thanh	N.D	12.3	37.0
November-2023	Phi To	N.D	N.D	N.D
	Nam Ha	N.D	4.4	22.0
	Me Linh	N.D	14.8	41.0
	Dong Thanh	N.D	11.0	33.0

N.D: Not detected

In 30 soil samples, 3 pesticides were detected including Chlorpyrifos and Profenofos of the Organophosphorus group and Carbosulfan of the Carbamate group. From Table 3, we found that soil samples in Phi To commune had the lowest Chlorpyrifos concentration with maximum concentration in May and November 2023 were 61 and 57.5 µg/kg, respectively. Nam Ha Commune had the maximum concentration were 158 and 129 µg/kg. The Chlorpyrifos concentrations of soil samples in Dong Thanh communes higher with maximum concentration were 261 and 195 µg/kg and highest in Me Linh with maximum concentration in May and November 2023 were 391 and 294 µg/kg.

Average Chlorpyrifos concentrations in soil samples gradually increased as 32.5, 74.7, 187, and 231 µg/kg in Phi To, Nam Ha, Dong Thanh, and Me Linh in May and 29.7, 66.4, 149, and 169 µg/kg in November. Pesticide concentrations were detected in the rainy season are higher than in the dry season, possibly because there are more pests and diseases in the rainy season, so the amount of pesticides used leads to high residue in the soil.

The highest concentrations of Profenofos were detected in 2 communes Me Linh and Dong Thanh in May at 42.5 and 63.0 µg/kg and in 3 communes Phi To, Me Linh and Dong Thanh in November at 29.0, 28.0 and 52.5 µg/kg respectively. The average concentrations in May were generally higher than in November.

There are 8 soil samples where Carbosulfan was detected in 3 communes: Nam Ha, Me Linh and Dong Thanh, divided equally between two sampling periods in May and November 2023. The highest concentration in Nam Ha, Me Linh and Dong Thanh in May were 15.0, 24.0 and 37.0 µg/kg and the November were 22.0, 41.0 and 33.0 µg/kg, respectively. There is no significant difference of average Carbosulfan concentration in the rainy and dry seasons, suggesting that this may just be the remaining residue in the soil. Chlorpyrifos concentration is showed and compared in Table 5. It was lower than in Lam Ha, February 2023 [8], Iran [23] and Malaysia [16] but higher than others.

Table 5. Chlorpyrifos concentration.

Sample Locations	Sample type	Sampling time	Chlorpyrifos concentration (µg/kg)	Reference
Lam Ha	Soil	2023	N.D - 391	This study
Lam Ha	Soil	2023	N.D - 954	Lam D.V. [8]
Mekong Delta	Land	2019	3.51 - 291	PhD thesis summary
Nepal	Soil	2021	32.5 - 177	Govinda Bhandari [21]
Iran	Soil	2021	240 - 510	Mohsen Hesami Arani [23]
Egypt	Sediment	2022	119 - 241	Eissa [22]
Ghana	Soil	2016	10 - 40	Fosu-Mensah [6]
Malaysia	Land	2010	20 - 2240	Norhayati Mohd Tahir [16]

N.D: Not detected

Table 6 compared Profenofos concentration with previous studies. The results obtained from this research were higher than in Nepal and Ghana. Table 7 showed the Carbosulfan concentration alittle bit lower than in Indonesia.

Table 6. Profenofos concentration.

Sample Locations	Sample type	Sampling time	Profenofos concentration (µg/kg)	Reference
Lam Ha	Soil	2023	N.D – 63.0	This study
Nepal	Soil	2020	1.75	Govinda Bhandari [20]
Ghana	Soil	2016	20-40	Fosu-Mensah [6]

N.D: Not detected

Table 7. Carbosulfan concentration.

Sample Locations	Sample type	Sampling time	Carbosulfan concentration (µg/kg)	Reference
Lam Ha	Soil	2023	N.D – 41.0	This study
Indonesia	Soil	2021	58.2-307.2	Ardiwinata [24]

N.D: Not detected

In the rainy season, soil pH decreases, moisture increases while organic carbon content and soil mechanical composition change little. The decomposition ability of pesticides increases with moderate humidity, mildly acidic to neutral pH, and high organic carbon content [25]. Soil mechanical composition also affects the decomposition ability of pesticides, however analysis results show that there is no obvious difference between soil samples in the dry season and the rainy season. Currently, no clear correlation has been found between the physicochemical properties of soil and the concentrations of pesticides. This may be because these pesticides are partly residual and partly used to kill pests during cultivation.

3.3. Ecological risk assessment

With AF = 10 and NOEC = 65 (µg/kg) for Chlorpyrifos, AF = 50 and NOEC = 50 (µg/kg) for Profenofos [19] and PNEC= 2410 (µg/kg) of Carbosulfan in soil [26], risk index RQ were calculated in Table 8.

Table 8. Risk assessment.

Sampling time	Sample location	RQ of Chlorpyrifos	RQ of Profenofos	RQ of Carbosulfan	Risk assessment
May-2023	Phi To	< 0.01-9.4	< 0.01	< 0.01	Very low risk - high risk
	Nam Ha	< 0.01-24.2	< 0.01	< 0.01	Very low risk - high risk
	Me Linh	18.2-60.0	< 0.01-42.5	0.013	high risk
	Dong Thanh	19.8-40.1	< 0.01-63	0.015	high risk
November-2023	Phi To	< 0.01-8.8	< 0.01	< 0.01	Very low risk - high risk
	Nam Ha	< 0.01-19.8	< 0.01	< 0.01	Very low risk - high risk
	Me Linh	12.1-45.2	< 0.01-28.0	0.017	high risk
	Dong Thanh	16.5-30.0	< 0.01-52.5	0.014	high risk

Table 8 summarizes the results of RQ coefficient in 30 soil samples in 4 communes of Lam Ha district. In general, the ecological risk coefficient is at a very low to high level. Samples detected Chlorpyrifos all had high risk because it is highly toxic and persistent. This is a specific pesticide that was widely used before being banned in 2021. Maybe because people continue to use it, soil samples have RQ coefficients from < 0.01 to 60.0, however it is lower than the research in February 2023. Profenofos has RQ from < 0.01 to 63.0 and was found in 6/30 soil samples. Other Organophosphorus pesticides were not detected with a detection limit of 1.0 µg/kg and RQ < 0.01. Carbosulfan was found in 8/30 soil samples, however, due to lower toxicity than Chlorpyrifos and Profenofos, the RQ index was ranged from < 0.01 to 0.017. Other Carbamate pesticides were not found and had RQ < 0.01. RQ coefficient is only an initial preliminary assessment of the correlation between residual concentrations and toxicity thresholds. In the following studies, more appropriate evaluation tools will be used to make the most accurate conclusions.

With an ecological risk level < 1.0, no remedial measures are needed. However, with high levels > 1.0, it is necessary to issue warnings and take action to minimize impacts on humans and the environment. Some action that can be taken include propagating and instructing people about the toxicity and usage of some common pesticides, limiting the use of highly toxic and persistent pesticides, and using Personal protective equipment when spraying and cleaning after used to reduce contact.

4. Conclusions

The study was conducted 30 samples of coffee growing surface soil in 4 communes of Lam Ha district: Phi To, Nam Ha, Me Linh, and Dong Thanh. The physicochemical properties and two groups of pesticides, Organophosphorus and Carbamate, were selected for research. Basically, soil in the rainy season has lower pH and higher moisture than in the dry season. Organic carbon content and mechanical composition do not change much with the seasons. Among total 9 Organophosphorus and Carbamate pesticides, 3 were found: Chlorpyrifos, Profenofos and Carbosulfan of a total 30 soil samples. The highest concentrations of Chlorpyrifos, Profenofos and Carbosulfan were 391, 63 and 41 µg/kg, respectively. Using the RQ coefficient, the ecological risk level is assessed with risk results from very low to high and in ascending order from Phi To, Nam Ha, Dong Thanh and Me Linh.

Authors contribution: Constructing research idea: D.V.L., V.D.T.; Select research methods: D.V.L., V.D.T.; Data collection: D.V.L., V.D.T.; Data processing: D.V.L., V.D.T.; Writing original draft preparation: D.V.L.; Writing review and editing: D.V.L., V.D.T.

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Conflicts of interest: The authors declare that this article was the work of the authors, has not been published elsewhere, has not been copied from previous research; there was no conflict of interest within the author group.

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