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Understanding the persistence of hexachlorobenzene and its intermediates in agricultural chemical usage areas

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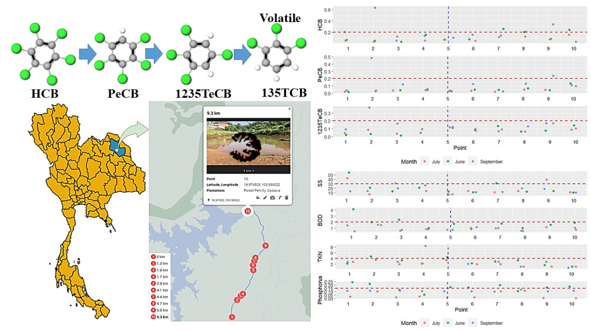
DOI: 10.55674/cs.v16i3.253700

Received: 1 August 2023; **Revised:** 4 July 2024; **Accepted:** 4 July 2024; **Available online:** 1 September 2024

Abstract



This study examined the presence and distribution of Hexachlorobenzene (HCB) and its derivatives in sediment samples. The investigation was conducted in a watershed along the Lum Stream agricultural area during the 2021 rainy season. The results showed HCB persistence despite being banned for a prolonged period. The water quality, indicated by the suspended solids (SS) and organic content, was evaluated at ten different observation points. However, only trace amounts of chemicals were detected in the sediment samples. The concentration levels of HCB, PeCB, and 1235-TeCB in the sediments ranged from 0.032 to 0.631 ppm, 0.018 to 0.485 ppm, and 0.091 to 0.366 ppm, respectively. The relationship between HCB, SS, and organic content was analyzed monthly, revealing interesting patterns. For instance, June and July exhibited similar trends, while HCB and its intermediates were negatively correlated with distance in September. Furthermore, a positive correlation was observed between 1235-PeCB and BOD of 0.48. This implies that as the amount of 1235-PeCB increased in the sediment samples, the BOD of the water also increased, suggesting a possible impact of 1235-PeCB on the water quality.

Keyword: Hexachlorobenzene (HCB); Pesticide; Persistence; Agricultural Chemical

1. Introduction

Thailand is an agricultural country with several provinces engaged in farming practices. Some of the major agricultural provinces in Thailand include Chiang Mai, Chiang Rai, Nakhon Ratchasima, Khon Kaen, Sakon Nakhon, and Surin. These provinces are known for growing rice, corn, sugarcane, fruits, and vegetables. In the meantime, the country heavily relies on pesticides to maintain crop yields and protect crops from pests and diseases. Due to the limited availability of certain types of pesticides, Thailand must import various types from other countries. About 70,000 tons of pesticides comprising 265 active ingredients were imported to Thailand in 2010 [1]. This importation of pesticides poses potential risks to the country's environment and public health. Furthermore, the excessive use of pesticides in agriculture has been linked to negative impacts on soil health, water quality, and human health.

The Thailand Pesticide Alert Network (Thai-PAN) reports that Thailand's annual utilization of pesticides ranks sixth globally among the top 30 countries with significant agricultural land [2]. Additionally, Thailand's consumption of toxic substances also positions it among the leading nations in the world. A few decades ago, numerous pesticide products contained hexachlorobenzene (HCB) and its intermediates, such as pentachlorobenzene (PeCB), 1235-tetrachlorobenzene (TeCB), and 135-trichlorobenzenes (TCB) later listed as hazardous chemicals under the Stockholm Convention on Persistent Organic Pollutants (POPs) many years ago as they possess a high level of toxicity and tend to persist in the environment for extended periods of time [3]. The release of these chemicals into water sources should be strictly avoided as they threaten the aquatic environment and may cause long-term adverse effects.

However, these chemicals were widely used to control various crops' fungal diseases. Thai farmers also relied on these substances to protect their crops from disease and insect damage. Despite the convenience, cost-effectiveness, and efficacy of pesticides, numerous studies indicated that their use can negatively impact the health of the farmers utilizing them. Furthermore, farmers sometimes used a mixture of chemicals under the belief that it was more potent, leading to overuse and improper disposal, causing a detrimental effect on the environment in and around their communities, including land, rivers, and canals. This research examines the endurance and correlations of HCB and its intermediates within the agricultural locale of Sakon Nakhon province, particularly in the vicinity of the Lum stream located in the upstream region of the Phong River during various temporal intervals and distances over the rainy season. This investigation also examines additional parameters such as organic content and nutrients, which may impact the capacity of microorganisms to biodegrade HCB, as well as suspended solids, which may affect the conveyance and destiny of HCB in surface waters. This research can better understand the factors influencing HCB persistence in agricultural chemical usage areas and potentially develop strategies to mitigate its impact on the environment and human health. This study proposes that HCB and its intermediates may persist in sediments surrounding agricultural areas in Thailand despite being banned in the country several decades ago. The following sub-hypotheses were developed to support this hypothesis:

1) Areas closer to the Lum stream, near agricultural activities, may exhibit higher concentrations of HCB and its intermediates due to runoff and leaching during the rainy season.

2) Higher suspended solids in the Lum stream during the rainy season may correspond to a greater conveyance of HCB and its intermediates downstream, potentially leading to more severe impacts on downstream ecosystems and human populations reliant on the stream for water or other resources.

3) Organic content measurements could yield important insights into the persistence of HCB and its intermediates since elevated levels of organic content may promote the growth of microorganisms, leading to increased biodegradation rates of HCB and its intermediates.

2. Materials and Methods

This section comprehensively examines the materials and techniques utilized in this research.

Sampling Area

The investigation was conducted in a watershed along the Lum Stream agricultural area, situated in a primary upstream catchment of the Phong River, and served as the area for sediment collection for the experiment. The stream runs through Nong Han Lake, Sakon Nakhon Province, in the North-Eastern Region of Thailand, and spans approximately 9.3 km. The stream is surrounded by a diverse landscape of long-term agricultural activities, including forestry plantations, mixed field crops, villages, forest fertility, eucalyptus forests, rice fields, mixed fruit fields, sugar cane fields, cassava fields, rubber tree fields, water resources, short-term vegetable fields, and cone fields. In addition, ten sampling points were established along the stream, numbered from 1 to 10, at distances of 0, 1.3, 1.6, 1.7, 3.8, 4.1, 4.4, 4.7, 5.8

and 9.3 km, respectively. A total of 10.31 km^2 of land use was investigated, separated into ten points, with areas of 1.61, 0.87, 0.13, 0.35, 0.90, 1.65, 0.15, 0.50, 1.95 and 2.20 km^2. Most land use at point 1 is a forestry plantation (0.81 km^2), whereas points 2, 3, and 5 to 9 are reserved for cassava fields at 0.47, 0.09, 0.47, 1.46, 0.05, 0.37, and 1.00 km^2, respectively. Point 4 is primarily used for mixed field crops at 0.12 km^2 and point 10 is designated as forest fertility with an area of 1.97 km^2. Fig 1 presents a graphical representation of the sampling area of this study.

Experimental Period

The experiments were carried out during the rainy season in Thailand, between June, July, and September of 2021, after the initiation of agricultural activities in May, including the application of fertilizers and land preparation additives. As the research investigated the persistence of HCB and its intermediates concerning organic contents during the rainy season in agricultural areas, it was crucial to account for the surface runoff flow rate at each sampling point, which could facilitate the transport of pollutants from agricultural sources. The relevant details concerning the surface runoff flow rate, stream distances, highest monthly rainfall, and average daily rainfall in the sampling area are presented in Fig 2, illustrating that points 1, 9, and 10 have significant flow rates, with values of 2.47, 2.45, and 2.78 m^3/H respectively. The surface runoff flow rate (Q) was used as a rational method to analyze the surface runoff, considering the small watersheds (<4 km^2). The average daily and maximum monthly rainfall during the rainy season show variability, ranging from 9.17 to 16.77 and 37.70 to 104.00 mm, respectively. June recorded the highest average daily rainfall and highest monthly rainfall, while September, as the late rainy season, recorded the lowest daily and monthly rainfall.

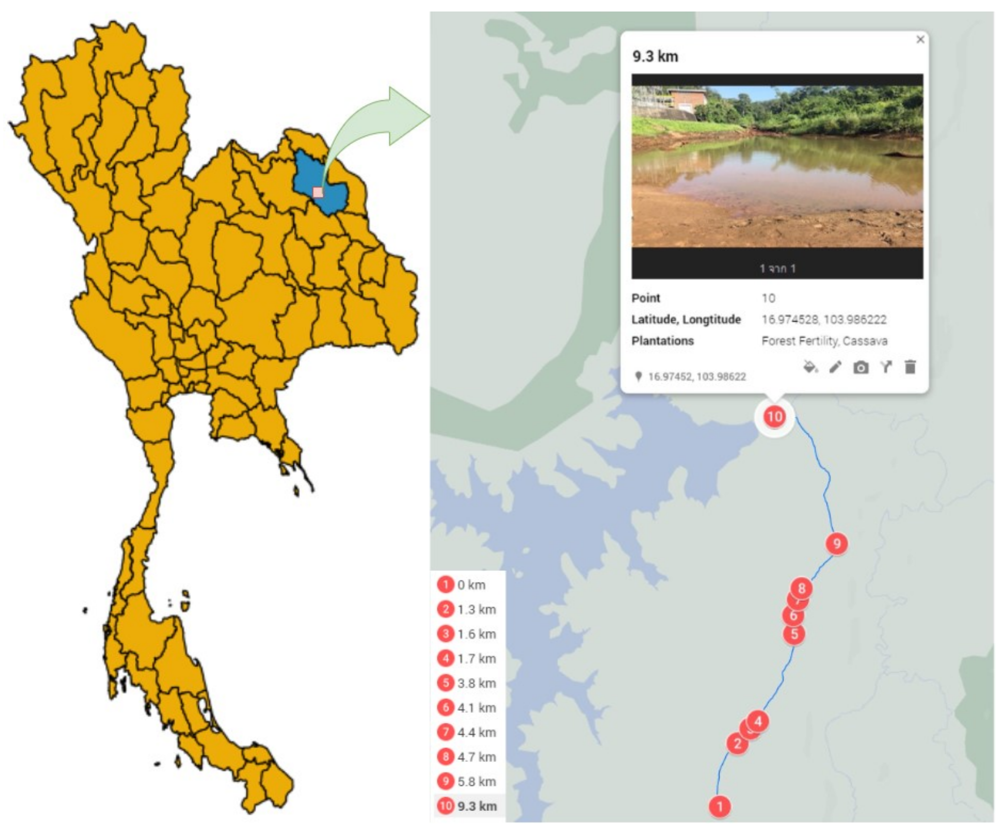


Fig. 1 The utilization of agricultural land in the vicinity of the upstream region.

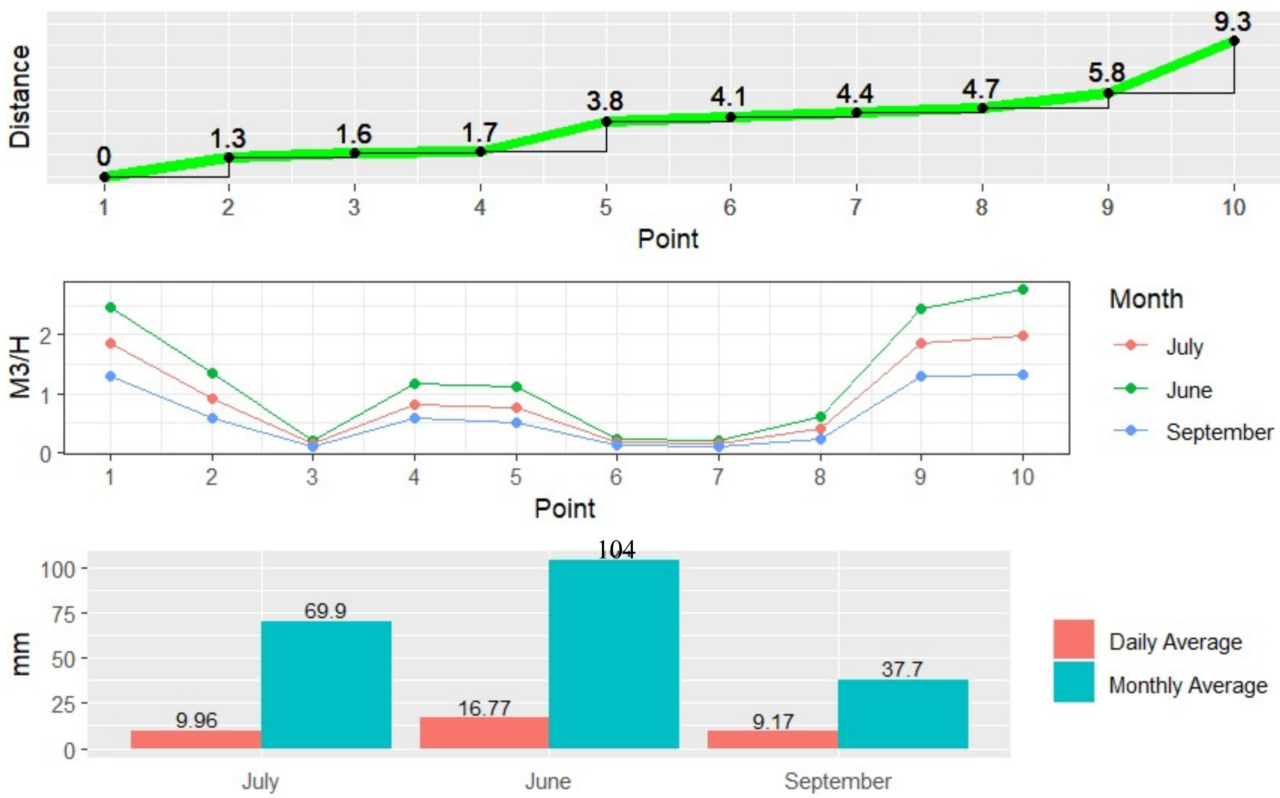


Fig. 2 The utilization of agricultural land in the vicinity of the upstream region

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Sediment Sampling and Chemicals

Sediment samples were collected from three points along the waterfront, at a depth of 15-30 cm. The cone and quarter methods were utilized with shovels to remove the soil surface, and the collected soil was equally divided into four parts. Two parts were placed opposite each other and kept in a plastic bag. Additionally, three-point sediment samples were collected under shallow water using shovels to remove the sediment surface, and the collected samples were mixed and stored in a plastic bag. The chemicals used in this research were of reagent grade quality and included Chlorinated benzene congeners (CBs) such as 1235-TeCB, PeCB, and HCB, which were obtained from Sigma-Aldrich. Stock standard solutions were stored in 1.5-ml vials, sealed with butyl rubber stoppers, capped with alumina cups, and stored in the refrigerator until use.

Sediment Extraction

The ultrasonic extraction procedure (USE), modified from the EPA 3550C method, extracts sediment and soil separately. Two grams of soil or sediment are weighed into the extraction tube in triplicate and then subjected to the extraction process. To initiate the extraction, 2 milliliters of a 1:1 mixture of hexane and acetone and 0.2 ml of 6N NaOH are added to the tube. The mixture is then agitated using a vortex mixer, followed by 10 minutes of ultrasonication. The vial is centrifuged at 3,500 rpm for 10 minutes to separate the extracted components. The upper layer of the extracting solvent is carefully removed and transferred to a new tube. This process is repeated twice, with the third extraction used for analysis. Before analysis, a small amount of sodium sulfate is added to remove moisture. The external chemical 2345-CBP is a spiked chemical for quality control measurement [5].

Chemical Analysis

The Shimadzu Gas Chromatography System (GC-2014) is a precise analytical instrument that utilizes a high-performance and dependable capillary column (Rtx-1) with an electron capture detector for chemical analysis. The column has a length of 30 m, a film thickness of 0.25 µm, and an inner diameter of 0.25 mm. The oven temperature is maintained at 80 °C for 5 minutes and increased at a rate of 3 °C per minute until it reaches the final temperature of 140 °C. The temperature is then increased at a rate of 10 °C per minute to the absolute temperature of 240 °C and maintained for 8 minutes. The detector and injector temperatures are set at 240 °C and 280 °C, respectively. Helium and nitrogen are used as the carrier and makeup gases, with average linear flow rates of 20 and 60 ml/min, respectively, and a split ratio 10:1.

In addition, the analysis of water quality parameters, such as biological oxygen demand (BOD), suspended solids (SS), total Kjeldahl nitrogen (TKN), and phosphorus, is conducted following standard methods for examining water and wastewater. BOD measures the organic content present in surface runoff, where intermediates of HCB can adsorb inorganic particles. SS represents the solid particles suspended in surface runoff from agricultural land use. TKN and phosphorus are analyzed as they serve as indicators of fertilizer applied in agriculture. TKN reflects the total concentration of organic nitrogen and ammonia, and phosphorus is used to assess water quality. Excessive amounts of these nutrients can lead to a reduction in dissolved oxygen levels in water bodies.

Data Analysis

Based on ten points with different distances classified into three experimental months, namely June, July, and September, the experimental results of the persistence of HCB and its intermediates and organic contents were analyzed. Data visualization has become critical in environmental science and related disciplines [16], and the results were visualized in various patterns. RStudio was utilized in this research to present chemical quantities and related constituents based on ten sampling points, using the ‘ggplot2’ package for data visualization and statistical analysis. The software also depicted the correlation between the chemicals and constituents analyzed. The relationships among elements categorized into three months in this study were numerically visualized using the ‘corrplot’ package in R with the ‘number’ method.

3. Results and Discussion

This section discusses experimental results and provides a comprehensive description and visual representation of the experimental results regarding HCB and its intermediates and organic contents.

HCB and its Intermediates

Fig 3 illustrates the persistence of HCB and its intermediates in sediments, including PeCB, and 1235-TeCB. The persistence of HCB and its intermediates is explained and discussed below.

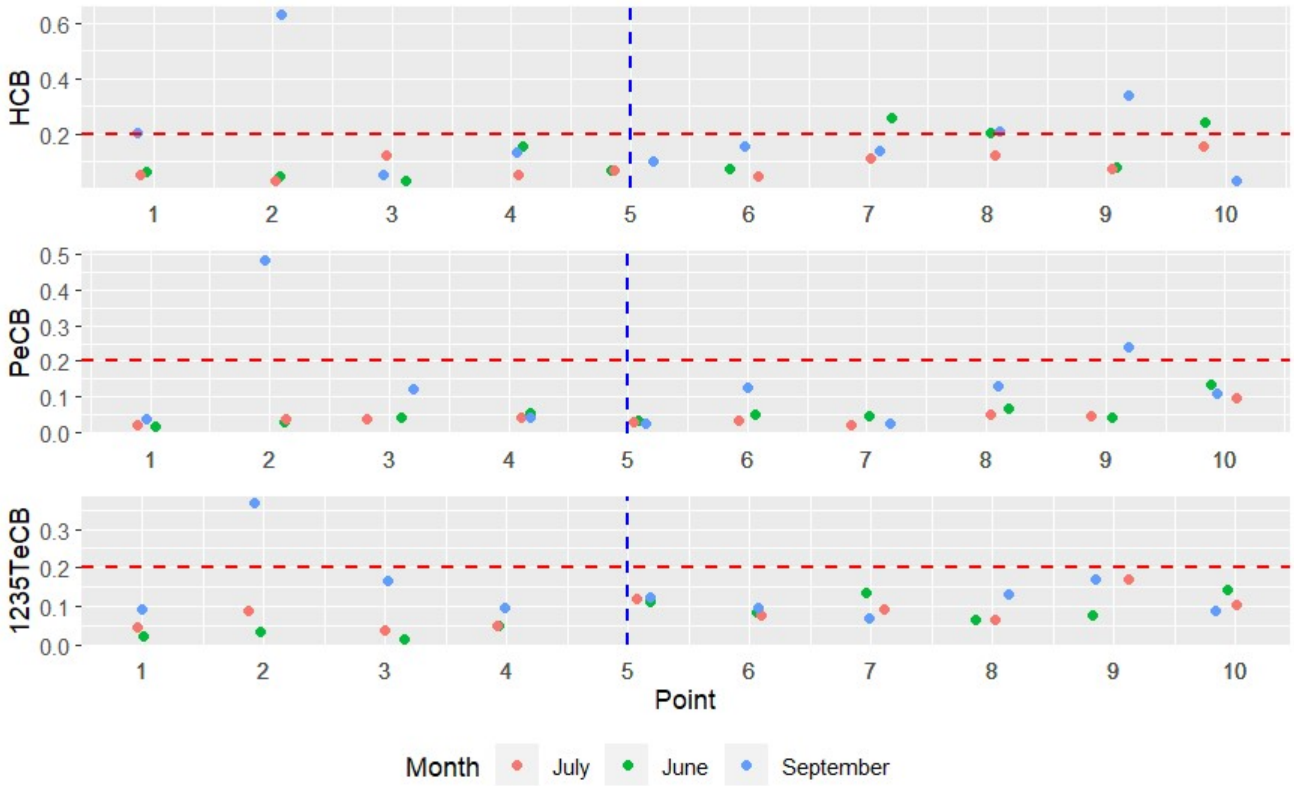


Fig. 3 HCB and its intermediates in sediment.

HCB Persistence

The concentration of HCB in sediments was observed to range from 0.032 to 0.631 ppm, with a peak of 38%. The HCB concentration in sediments collected in June fluctuated between 0.07 ppm and 0.032 ppm at point 3. The HCB levels then alternated between 0.08 and 0.28 ppm until reaching 0.27 ppm at point 10. The HCB concentration in sediments collected in July ranged from 0.06 to 1.6 ppm from point 1 to point 10, except for point 3, which was 1.1 ppm, higher than the HCB levels observed in June. In September, HCB concentration in sediments increased from 0.2 ppm at point 1 to 0.631 ppm at point 2, then decreased to 0.6 ppm at point 3. Interestingly, these levels of HCB were observed to increase gradually from 0.05 ppm at the starting point to 0.33 ppm at point 9 before declining to 0.2 ppm at the last point. These levels were higher than the previously reported HCB values of 0.15 and 0.26 mg/kg in sediments collected from a canal that receives wastewater from the Samut Prakan Industrial Estate [5].

PeCB Persistence

There was consistency in the concentration patterns from points 1 to 10 in June and July. The PeCB levels in June varied between 0.018 and 0.13 ppm, while in July, they were between 0.019 and 0.09 ppm. It is worth noting that detected PeCBs had a range of 0.14-0.2 ppm. PeCBs in September showed an upward trend, starting from 0.04 ppm at point 1 and reaching a peak of 0.485 ppm at point 2, before rapidly declining to 0.14 ppm at point 3. There was a steady decline to 0.03 ppm at point 5, followed by a slight fluctuation until point 9 (4.4 km), where the concentration increased to 0.24 ppm. Finally, the PeCB concentration decreased to 0.11 ppm at point 10 (9.3 km).

1235-TeCB Persistence

The concentration patterns from points 1 to 10 in June and July were similarly stable, with levels ranging between 0.091 and 0.18 ppm. In September, the concentration started at 0.091 ppm at point 1, rose to 0.366 ppm at point 2, and dropped to 0.18 ppm at point 3. From there, the concentration steadily decreased to 0.1 ppm at point 5 and remained between 0.09 and 0.18 ppm, mirroring the trends observed in June and July, until the final point.

The study findings showed that the maximum concentration of HCB and its intermediates was observed at point 2 during September, an area dedicated to cassava, mixed fruit, and mixed crop cultivation. PeCB and 1235-TeCB were found to be between 0.018-0.485 ppm and 0.091 - 0.366 ppm, respectively, with a contamination rate of 26% and 4%. While the highest levels of both HCB intermediates were detected at 1.30 km in September, their concentrations decreased rapidly at 1.6 km and rose again at 5.8 km, reaching 0.239 ppm for PeCB and 0.169 ppm for 1235- TeCB. The concentration of both HCB intermediates continued to decrease consecutively until the last sampling point at 9.3 km. Additionally, the sediment accumulation at point 1 (1.30 km away) was observed to flow into the cultivation areas of cassava, mixed fruit, and crop fields. The results showed that HCB and PeCB were present only in the rainy season, which coincided with the lowest monthly rainfall and the highest concentration of HCB and its intermediates. These findings suggest limited dechlorination of HCB in the environment and its sedimentation at the bottom of the stream during the dry season, which can resuspend after water turbulence during the rainy season.

Organic Contents

Fig 4 depicts the quantification of organic contents, such as SS, BOD, TKN, and phosphorus, observed at intervals ranging from point 1 to point 10 in June, July, and September. This study assessed the impact of agricultural land use on the presence of organic substances in surface water. The results of these organic contents are discussed below.

SS

The SS concentration in June began at 55 mg/l and experienced a sharp drop to 20 mg/l at Point 2. Subsequently, the concentration fluctuated until Point 7 (4.4 km), suddenly rising to 31 mg/l. Afterward, the concentration decreased steadily to reach 11 mg/l at the final Point (9.3 km). In July, the SS concentration started at 42 mg/l at Point 1 and experienced a significant decrease to 8 mg/l at Point 2. The concentration then increased to 31 mg/l at Point 3, before experiencing another sharp decrease to 7 mg/l at Point 5. From Point 5 to Point 8, the concentration remained relatively stable, before experiencing a sudden rise to 40 mg/l at Point 9. Finally, the concentration plummeted rapidly to 11 mg/l at the last Point. In contrast, September saw the concentration at 13 mg/l at Point 1, gradually increasing to 28 mg/l at Point 4. However, there was a sharp drop to 7 mg/l at Point 5, before remaining relatively stable at 11 mg/l at the final Point.

BOD

The stream's organic content was analyzed using the BOD method, which yielded values ranging from 0.9 to 4.15 mg/l in June, 0.5 to 1.5 mg/l in July, and 0.6 to 2 mg/l in September. The highest concentration was observed in June at Point 1, with a value of 4.15 mg/l, while the highest in July and September were 1.5 mg/l and 2 mg/l, respectively. These findings suggest that the organic content of the stream is highly variable and influenced by factors such as seasonal fluctuations and agricultural activities in the surrounding areas. The BOD results obtained from the stream water samples can be used to assess the impact of agricultural activities on water quality in the area. The high BOD values observed in June may be due to the high fertilizers applied during planting. The low BOD values in July and September may indicate less organic matter in the water at these times, possibly due to lower fertilizer use or increased microbial degradation.

TKN

The TKN results indicate high nitrogen levels and other nutrients in the water samples, particularly in June and July. This may be attributed to the heavy use of agricultural fertilizers during this period, which can lead to the leaching of nutrients into the water sources. The presence of high TKN concentrations in the stream during September, particularly at point 1.7 km where mixed field crops are planted, may also be related to the use of fertilizers and other factors such as irrigation practices and soil erosion. The high TKN levels in the water can support the growth of aquatic plants, leading to eutrophication and environmental degradation. The recommended amount of TKN in surface water is 2.5 mg/l, and the detected value of 8.40 mg/l is 3.4 times higher, indicating an extended period of nutrient contamination. This underscores the importance of proper agricultural nutrient management practices to minimize the negative impacts on water quality and the environment.

Phosphorus

The presence of phosphorus residues was detected within a range of 0.009 to 0.240 mg/l, which exceeds the proposed natural allowance of 0.10 mg/l. The highest concentration of phosphorus, 0.240 mg/l, was recorded at Point 1 in June and subsequently decreased drastically to 0.06 at Point 4. However, concentrations higher than 0.15 mg/l were recorded at Points 5-7 and 9-10. The lowest phosphorus concentration was observed in July, ranging from 0.009 to 0.12 mg/l across the 10 sampling points. The highest concentrations in this month were recorded at Points 2, 7, and 8, with values of 0.10, 0.11, and 0.12 mg/l, respectively. In September, the phosphorus concentration varied from the previous months, starting at 0.112 mg/l and fluctuating between 0.1 and 0.126 mg/l until the last sampling point. The results show that phosphorus residues in the stream indicate using fertilizers in the surrounding agricultural areas. Phosphorus is a common nutrient in fertilizers that promotes plant growth, and its presence in water bodies can cause eutrophication, resulting in the excessive growth of aquatic plants and algae. This process can affect water quality, deplete oxygen levels, and lead to ecological damage. High phosphorus levels detected in the stream, particularly in June and September, suggest that agricultural activities in the area may contribute to nutrient loading. It is, therefore, necessary to implement appropriate management practices to minimize nutrient release into the stream and prevent potential ecological and health risks associated with nutrient pollution.

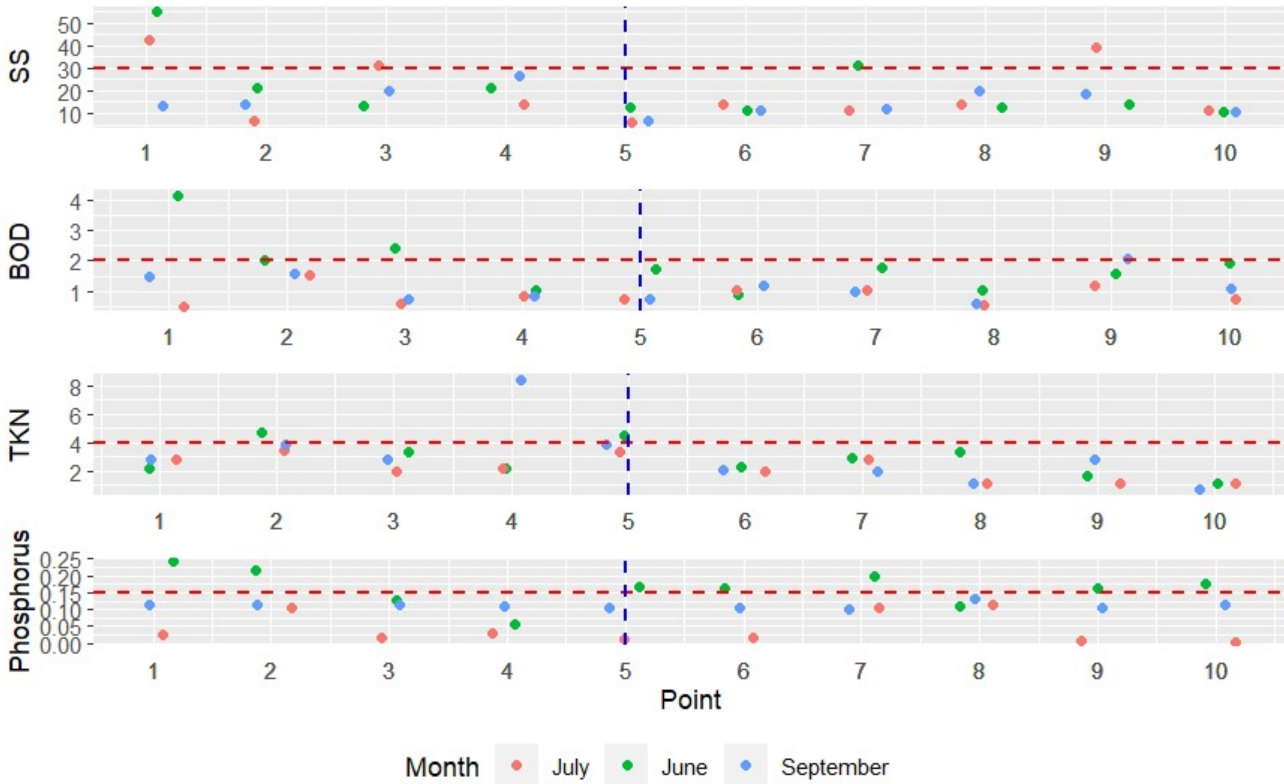
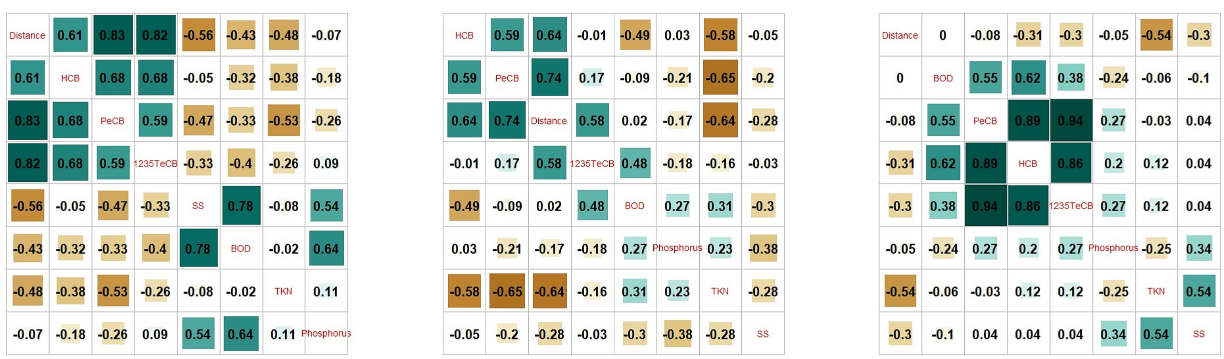


Fig. 4 Organic contents in sediment.

HCB Products and Organic Contents

Following a thorough investigation into the relationship between HCB and its intermediate contaminations and the environment, this section visually represents the correlations between these chemicals and organic contents in Fig 5.



(a) (b) (c)

Fig. 5 Correlations among HCB chemicals and organic contents in sediments: (a) June, (b) July, and (c) September.

June

As shown in Fig 5a, significant correlations exist between HCB chemicals and other studied variables in June. The HCB and its intermediates, PeCB, and 1235-TeCB, display positive correlations with each other, with the highest correlation between PeCB and HCB at 0.68. Additionally, these chemicals demonstrate a positive correlation with distance, with HCB, PeCB, and 1235-TeCB having coefficients of 0.61, 0.83, and 0.82, respectively. Conversely, negative correlations are observed between HCB and its intermediates with organic contents. HCB has negative correlations with BOD (-0.32), TKN (-0.38), and phosphorus (-0.18). Similarly, PeCB and 1235-TeCB show negative correlations with SS and organic contents, with PeCB having negative correlations with SS (-0.47), BOD (-0.33), TKN (-0.53), and phosphorus (-0.26). 1235-TeCB also displays negative correlations with SS (-0.33), BOD (-0.40), and TKN (-0.26). On the other hand, organic contents and SS exhibit positive correlations with each other, but negative correlations with distance. Distance has negative correlations with SS (-0.56), BOD (-0.43), and TKN (-0.38). These findings highlight the complex relationships between these chemicals and their environmental impact.

July

In July, Fig 5b demonstrates positive correlations between HCB and its intermediates. Furthermore, there are significant positive relationships between distance and HCB (0.64), PeCB (0.74), and 1235-TeCB (0.58). Most HCB chemicals and organic contents correlate negatively with SS and organic contents. However, one positive correlation was observed between 1235-TeCB and BOD at 0.48. The correlations between organic contents and SS are both positive and negative. Although SS has negative correlations with all organic contents (BOD = -0.30, TKN = -0.28, phosphorus = -0.38), there are positive relationships between BOD and TKN (0.31) and phosphorus (0.27). Additionally, TKN is positively correlated with phosphorus at 0.23. Similar to June, there are negative correlations between distance and SS (-0.28), TKN (-0.64), and phosphorus (-0.17), and SS and organic contents are negatively correlated with distance.

September

Fig 5c illustrates the correlation between HCB chemicals and the experimental variables. A strong positive relationship is observed between HCB and its intermediates (PeCB = 0.89, 1235-TeCB = 0.86). Additionally, there is a significant positive correlation between PeCB and 1235-TeCB, with a value of 0.94. However, it is noted that the relationship between distance and HCB chemicals differs in September, exhibiting negative correlations as opposed to the positive correlations observed in June and July. Regarding the correlations between SS and organic contents, a positive relationship is seen between SS and TKN (0.54) and phosphorus (0.34). Conversely, small negative correlations exist among organic contents, with the strongest negative correlations observed between phosphorus and BOD (-0.24) and TKN (-0.25). In addition, negative correlations are observed between distance and SS (-0.30) and TKN (0.54). The findings from this analysis indicate significant correlations between HCB and its intermediates in June, July, and September. However, it is important to note a marked difference in the relationship between distance and HCB chemicals in September, as all relationships are negative, contrasting the positive correlations observed in June and July.

4. Conclusion

We investigated the persistence of HCB and its intermediates concerning agricultural land use along a stream in Northeastern Thailand. Most of the land in the region is used for cassava cultivation. Even with solid and organic substances in the water, the environment's ability to purify itself is restricted. The macronutrients from water plants can seep into the stream and contribute to environmental degradation. The concentrations of HCB were found to range from 0.032 to 0.631 ppm, while PeCB and 1235-TeCB were detected in the range of 0.018–0.485 ppm and 0.091–0.366 ppm, respectively. Interestingly, the highest concentrations of HCB and its intermediates were detected at point 2 in September. Additionally, the correlations between HCB and its intermediates with SS and organic content in sediments were negative, indicating a relationship between contamination of HCB in sediment and organic content. This is despite the discontinuation of agricultural use in the region for more than 30 years. The findings suggest that the presence of HCB and its intermediates in sediments is due to the long-term adsorption of HCB in organic soil particles and the re- suspension sediment phenomenon or leaching of residual chemicals into the canal via surface runoff. These results provide insight into why HCB and its intermediates are present in sediments and the limitations of environmental degradation caused by these chemicals.

5. Suggestions

Further research should be conducted to validate the findings of this study on the correlations between HCB and its intermediates, SS, and organic contents in different agricultural areas surrounding river streams. This is crucial to provide a more comprehensive understanding of these relationships and ensure the consistency of results across various environmental conditions. Additionally, experimenting in different seasons may reveal interesting and valuable insights, particularly as environmental factors such as temperature and precipitation can significantly influence chemical behaviors and interactions. This could provide a more nuanced understanding of how seasonal variations affect the persistence and movement of these chemicals in agricultural settings. Moreover, increasing the sample size for the experiment will enhance the credibility of the results, allowing for more robust statistical analyses and generalizations. It is also recommended to include more diverse agricultural practices and crop types in the study to assess the potential variations in chemical persistence due to different agricultural practices. This holistic approach will help develop more targeted and effective environmental management and remediation strategies.

6. Acknowledgement

We thank Sakon Nakhon Rajabhat University and Phuket Rajabhat University for their direct or indirect support.

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