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Research Paper

INVESTIGATION OF ORGANOCHLORINE PESTICIDES IN SEDIMENT IN CAU HAI LAGOON, CENTRAL VIETNAM

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ABSTRACT

Cau Hai lagoon, located to the south of Phu Loc District, Thua Thien Hue province and the part of the Tam Giang - Cau Hai lagoon system is one of the largest lagoon systems in Southeast Asia. Cau Hai lagoon is a waterbody receiving overflowing water from the inland rivers and streams, so it is possible to accumulate organochlorine pesticides (chlorinated organic groups) in sediments. This study used Gas Chromatography-Mass Spectrometry (GC/MS) method to investigate the content of some organochlorine pesticides in sediment samples. In general, the results showed the presence of some organochlorine pesticides in the sample such as α-HCH, β-HCH, δ-HCH, Heptachlor, Aldrin, Heptachlor epoxide and Endosulfan. Some toxic chemicals had concentration less than the limit of detection of the analytical method. The results of this research showed the scientific and practical significance, which is an important database for concentration of organochlorine pesticides in sediment of Cau Hai lagoon, Thua Thien Hue province, Central Vietnam.

Keywords: *Sediments, organochlorine pesticides, Central Vietnam, Cai Hai, lagoon.*

1. Introduction

Crop protection chemicals penetrate into the water body from various sources such as wastewater, dry condensation, rainwater, runoff, erosion, etc These chemicals are sparingly soluble in water and capaple of accumulating in animal organisims' fat tissue or being adsorbed by suspended solid particles. After settling on the bottom, they will accumulate in the bottom layer organisms and enter the food chain, eventually entering the human body (Connell, 1994). Sediment pollution has adverse impacts on ecosystems and poses potential risks to benthos (Nhan et al., 2001). Estuarine sediments are frequently polluted with hydrocarbons (e.g. polycyclic aromatic hydrocarbons) from some reasons such as fuel spills and industrial wastes (Bach et al., 2005). Due to persistent characteristics in the environment and toxicity risks, the compounds of organicchlorine pesticides (OCPs) in sediments have been extensively researched (Xue et al., 2006; Wang et al., 2007; Darko et al., 2008;

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Farshid, 2015; Wang et al., 2016; Kampire and Rubidge, 2017). In Vietnam, there are also many works interested in plant protection chemicals in water, sediments or aquatic species (Toan, 2015; Doan et al., 2018; Trinh et al., 2019). Pesticide contamination considered shows impacts on organisms, ecosystems and the environment (Jinglan et al., 2011; Cheng et al., 2013; Jayaraj et al., 2016). In nature, the rotation cycle of pesticides is very complicated and has long-lasting characteristics. In addition to the role of human impacts, pesticides enter the environment due to the impact of many factors such as physics, chemistry, geography, climate, hydrology, migration and even biological characteristic of plants and animals (Nguyen, 2005). In particular, after settling to the bottom in the form of sediments, they will accumulate in the bottom layer organisms and enter the food chain, eventually entering the human body (Nhan et al., 2001). The distribution and residual of chemical pesticides in water or sediment cause problems for health as well as ecological environment (Wang et al., 2016; Unyimadu et al., 2019).

Meanwhile, Cau Hai is one of the lagoon belonging to Tam Giang - Cau Hai lagoon system. Tam Giang - Cau Hai system is the largest lagoon in Southeast Asia, located about 7 km northeast of Hue city, at geographical coordinates of 16°14’ to 16°42’ North latitude and from 107°22’ to 107°57’ East longitude (IMOLA, 2010). The total length of 68 km, Tam Giang-Cau Hai lagoon has an area of nearly 22,000 ha, is located on the territory of 33 communes of Phong Dien, Quang Dien, Phu Vang, Phu Loc and Huong Thuy towns (Thua Thien Hue Provincial People’s Committee, 2008; Nguyen and Nguyen, 2014). Cau Hai lagoon has a basin shape, the average depth of the lagoon is 1.4 m. The surface of the water is about 104 km2 and it connects to the East Sea through Tu Hien estuary (IMOLA, 2010). The hydrological conditions of Cau Hai lagoon are influenced by hydrological factors of rivers and the sea. Cau Hai lagoon is facing many critical issues such as oil pollution, eutrophication, coliform contamination and anthropogenic chemicals (Frignani et al., 2004). Zoning of water quality in Cau Hai lagoon showed phosphate, ammonia contents were not suitable for aquaculture and total coliform were higher than the standard (Truong et al., 2018). The study of pesticide residues accumulated in sediment samples in Cau Hai lagoon is very necessary. Previously, a number of studies identified OCPs in the Tam Giang-Cau Hai lagoon system but there has not been any study to assess the cumulative levels in sediment samples of Cau Hai lagoon (Doan et al., 2018). Therefore, we conducted the study “Investigation of organochlorine pesticides in sediment in Cau Hai lagoon, Central Vietnam” with the aim of providing basic information about the concentration of pesticides in sediment in the Cau Hai lagoon, Thua Thien Hue province. The study focused on the survey of 10 types of pesticides in sediments including α-HCH, β-HCH, δ-HCH, γ-HCH, Heptachlor, Aldrin, Heptachlor epoxide, Endosulfan, p,p’-DDE, m,p’-DDD. These are active ingredients with common characteristics such as environmental sustainability, good solubility in fatty acids, adipose tissue, low solubility in water and high toxicity. At the same time, they have the ability to accumulate permanently, especially it causes acute and chronic toxicity to animals and humans through the food chain.

2. Methods

2.1 Research subjects

Subjects of the study: OCPs in sediments include α-HCH, β-HCH, δ-HCH, γ-HCH, Heptachlor, Aldrin, Heptachlor epoxide, Endosulfan, p,p’-DDE, m,p’-DDD in Cau Hai lagoon, Thua Thien Hue Province, Central Vietnam.

Sampling location: Samples were collected at 07 locations (S1 to S7) as shown in Fig. 1 and Table 1.

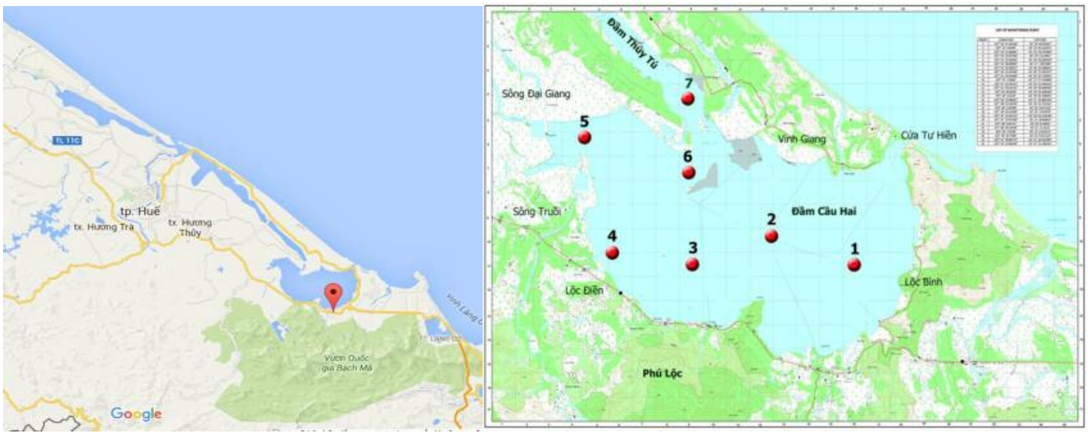

Fig. 1. Cau Hai lagoon system and sampling area

Table 1. Location of the sampling sites

Symbol	Number of samples (n)	Coordinate		Sampling period	Weather
		Latitude	Longitude		
S1	3	$107^0$53`30.469”	$16^0$18`30.00953”	June - September 2015	Sunny Cloudless sky Light winds T = 27.5-30.5^0C
S2	3	$107^0$51`32.352”	$16^0$19`11.24612”		
S3	3	$107^0$49`33.618”	$16^0$18`55.76839”		
S4	3	$107^0$48`02.870”	$16^0$19`10.57116”		
S5	3	$107^0$47`33.976”	$16^0$21`38.64816”		
S6	3	$107^0$50`01.034”	$16^0$21`06.24157”		
S7	3	$107^0$49`29.758”	$16^0$22`53.61114”		

2.2. Methods

2.2.1. Sampling method

In this study, the sampling procedure was carried out using the standard method of TCVN 6663-15:2004 (ISO 5667-15:1999). The sediment samples were taken using a special sampling drill at a depth of 0-10 cm. The collected sample is wrapped in aluminum foil and transferred to the laboratory, allowed to dry naturally at room temperature. The sample after natural drying, rough grinding and sieving through a 2 mm diameter was sieved to remove stones, grit, roots, etc. Then, the sample is continuously finely ground, and sifted with a pore size < 0.1 mm, stored in aluminum foil and kept in a clean, dry plastic bag at -20^0C.

Samples after pretreatment were extracted by Soxhlet extraction method. Soxhlet extraction method for 10 hours and 3-4 cycles/hour, extraction solvent is a mixture of 300 mL n-hexane: acetone with a volume ratio of 1:1 (Nhan et al., 1999; ATSDR, 2007; Pham et al., 2011). The extract sample is concentrated to 2-5 mL on a rotary evaporator. Concentrated extracts are treated with concentrated H2SO4 and continue to be washed with water until no more acid is present. Next, shake with the activated copper chip to remove the sulfide compounds. The extract after removal of the sulfide compounds is transferred to a florisil column for cleaning. Elute the florisil column with 50 mL of n-hexane: acetone mixture with a corresponding volume ratio of 9:1, elution rate of 4-5 mL/minute. The eluate is vacuum-rotated at 40-50°C to a volume of 2-5 mL and transferred to a 15 mL glass tube with a small amount of n-hexane. The solution is further evaporated by solvent flow of nitrogen to a volume of less than 1 mL, adding internal standard phenanthrene-d10 and making up to volume of 1 mL for analysis by the chromatographic system GC - MS.

Fig. 2. Pre-treated sediment sample

2.2.2. Analytical method

The qualitative and quantitative analysis of pesticides was conducted on gas Chromatography - Mass spectrometry GC - MS (Agilent Technologies 7890A System). Based on the study Pham et al. (2010) the working conditions and temperature program of GC-MS device are established. In particular, Separation column: capillary column Rtx®-CLPesticides (column length 30 m, internal diameter 0.25 mm, thickness of the static phase layer of 0.25 μm); Gas bearing He (purity 99.9995 %); Sample pump room temperature: 250 oC; Column oven temperature: 85 oC; Model of pump: splitless, the sample is automatically pumped with a volume of 3 mL; Flow rate: 1 mL/minute. The working conditions of the mass spectrometer are as follows: Ionizing source: EI 70 EV source; Ionizing source temperature: 230 oC; Detector voltage: 1447 V; Interface temperature: 250 oC; Cutting solvent time: 9 minutes; Scan mode (SCAN) and fragment selection analysis mode (SIM); Post run temperature: 285 oC.

2.2.3. Assessing reliability

Proceed to extract OCPs in a sediment sample several times with a mixture of n-hexane: acetone solvent with a volume ratio of 1:1. By the time the extract was no longer detectable OCPs when analyzed on the GC - MS. This pattern is called the background pattern. Add 10 μL of the OCPs standard solution with a concentration of each substance of 40 ng/g to the background sample. Conduct a recovery survey of the analytes of interest on the matrix to assess the reliability of the analytical procedure. The recovery (Rev) is calculated by the formula (1):

$$Rev(\%)=\frac{(C_{m+c} - C_m)}{C_c}.100 \quad (1)$$

where C_{m+c} is the concentration of organochlorine pesticides in the standardized sample (ng/g); C_m is the concentration of organochlorine pesticides in the background sample (ng/g); C_c is the concentration of organochlorine pesticides in the standard sample (ng/g). In addition, the repeatability of the method is assessed through relative standard deviations (RSD) by comparing the calculated RSD to the value $RSD_{Horwitz}$:

$$RSD_{Horwitz} = 2^{(1-0,5lgC)} \quad (2)$$

If $RSD \le \frac{1}{2} RSD_{Horwitz}$ is accepted (Miller and Miller, 1988; Pham, 2006).

2.2.4. Statistical methods

The results are processed by software Aligent G1701EA (GC/MSD ChemStation). Experimental results are processed by statistical methods and using software Excel 2013 and SPSS 13.0 with significance level of 5%. ANOVA (analysis of variance) test was employed to exam the difference of pesticides levels in different categories of sampling sites. The Pearson correlation coefficient is used to evaluate the relationship between pesticides levels.

3. Results and discussion

3.1. The reliability of the analytical methods

Assessing the reliability of the analytical procedure, the process of surveying the recovery of analyte substances on the base sample is shown in Table 2.

The average recovery efficiency of 10 pesticides needed to be analyzed is relatively high, reaching values ranging from 74 to 104 %. In particular, the recovery efficiency of analytes such as β-HCH, Heptachlor, m, p'-DDD with high results respectively 101, 104 and 103 %. At the same time, the RSD ensures a good repeata-

bility, with a small range of 1.0 to 9.7 % (RSD <13 %). Thus, this procedure is suitable for analyzing the concentrations of pesticides in the experimental sample which is sediment - a sample with a sulfide content and the sample background contains many complex components.

Table 2. Recovery of pesticides in the spiked background sample 40 ng/g

No.	Chemistry	Rev_1 (%)	Rev_2 (%)	Rev_3 (%)	Rev_{tb} (%)	RSD (%)
1	α-HCH	79	83	76	79	4.4
2	β-HCH	99	101	102	101	1.5
3	δ-HCH	98	100	96	98	2.0
4	γ-HCH	89	90	97	92	4.7
5	Heptachlor	103	105	104	104	1.0
6	Aldrin	80	79	91	83	8.0
7	Heptachlor epoxide	87	97	80	88	9.7
8	Endosulfan	99	103	94	99	4.6
9	p,p'-DDE	71	76	74	74	3.4
10	m,p'-DDD	104	101	103	103	1.5

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3.2. Investigation of fluctuations of pesticides concentration in Cau Hai lagoon sediments

In general, the former study showed that indicating potential health risks for fish consumers related to the trace elements in Cau Hai lagoon (Tran et al., 2018). They are mainly associated with lithogenic matter and also is most likely the result of erosion process in the lagoon. The trends of lagoon contamination (e.g. sediment samples) indicated that Cau Hai lagoon is still slightly increasing (Frignani et al., 2007). The study of Duong et al. (2018) determined three toxic elements (Pb, As, Hg) in the surface sediment and to assess the ecological risk in this area. With the selected analytical process, the study analyzes the pesticides in sediment samples of Cau Hai lagoon in Thua Thien Hue province, Central Vietnam. The results of analyzing residues of pesticides in the samples are shown in Table 3. Active ingredients α- HCH were found at the monitoring locations S2, S3, S5 and S6. The ANOVA analysis presented the statistically significant differences among α-HCH levels in the monitoring sites ($F = 21.090$, $p = 0.001$). The distribution of HCHs in developing countries may explain the use of pesticides in agriculture in the past (Li et al., 2012). The content of δ –HCH, Heptachlor and Endosulfan was detected at all sampling points in sediments in Cau Hai lagoon. Results of the ANOVA test showed the statistically significant differences of β-HCH ($F = 47.985$, $p = 0.001$) and δ-HCH ($F = 17.805$, $p = 0.001$). Particularly, the content of γ-HCH, p,p'-DDE and m,p'-DDD showed no findings in the analytical samples. Comparing results of research on dioxins content in sediments in Iran shows that the content is equivalent to 8.66 ng/g (Farshid, 2015). Similarly, the content of dioxins analyzed for surface sediments of Lake Honghu (China) is valued at 9.19 ng/g and has an ecological risk (Yun et al., 2014).

Table 3. Concentration of organochlorine pesticides in the sediment in Cau Hai lagoon (ng/g)

No.	Chemistry	S1	S2	S3	S4	S5	S6	S7
1	α-HCH	-	0.23±0.12	0.02±0.01	-	1.12±1.02	0.17±0.03	-
2	β-HCH	-	0.24±0.32	1.80±0.22	0.02±0.02	1.03±1.06	0.17±0.02	-
3	δ-HCH	1.06±0.21	1.24±0.76	0.67±0.23	0.50±0.27	1.23±0.74	1.97±0.25	1.76±0.34
4	γ-HCH	-	-	-	-	-	-	-
	∑HCH	1.06±0.62	1.71±0.45	2.49±1.21	0.52±0.22	3.38±2.10	2.31±1.03	1.76±0.23
5	Heptachlor	13.50±2.34	2.98±0.23	9.98±3.43	8.74±2.37	5.93±1.29	27.56±4.56	14.87±3.71
6	Aldrin	2.00±1.02	-	0.16±3.45	-	-	0.11±0.03	-
7	Heptachlor epoxide	-	-	0.02±0.01	-	-	0.04±0.02	-
8	Endosulfan	15.15±3.42	15.30±5.67	17.22±2.43	15.74±4.53	14.16±2.32	21.12±3.49	21.30±2.39
9	p,p'-DDE	-	-	-	-	-	-	-
10	m,p'-DDD	-	-	-	-	-	-	-

Note: Mean ± SD, n=3; "-":Not detected.

The area around the Cau Hai lagoon is at risk due to flooding, water pollution and over-exploitation (IMOLA, 2010). Regarding to the water quality, in the study of Dang et al. (2019) indicated that the nutrient availability and abundance of the phytoplankton community in lagoon. Besides, in the sediment samples taken from Cau Hai lagoon showed that is only slightly contaminated by trace elements (heavy metals) and these distributions were mainly influenced by river inputs (Giuliani et al., 2011). In this study, the results of analyzing pesticides in the sediment of Cau Hai lagoon show ∑HCH oscillate 0.52±0.22 to 3.38±2.10 ng/g. ∑HCH content in sediment at all points exceeded the impact threshold value TELs. There are 6 points that exceed the PELs impact value level according to Canadian environmental quality guidelines (Canadian Council of Ministers of the Environment, 2002) and no γ-HCH was detected in all sediment samples. This is explained by the nature of the HCH isomers in descending order: β-HCH> δ-HCH, α-HCH> γ-HCH. Therefore, it is possible that the γ-HCH active substance in the sediment has decomposed (Connell, 1994).

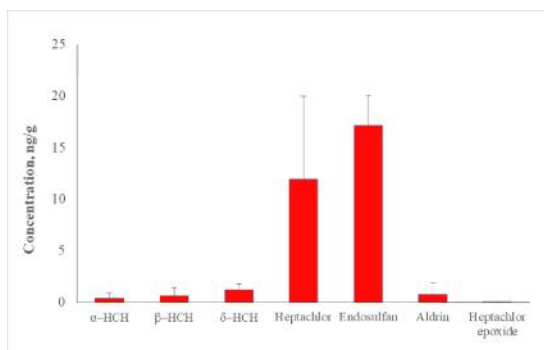

Fig. 3. Concentration of pesticides in sediment

Through the ANOVA test, there were statistically significant differences between the Heptachlor levels (F = 209.529, p = 0.001) as well as the Endosulfan levels (F = 14.812, p = 0.001) of monitoring sites. The combined concentration of organchlorine pesticides in sediments in Cau Hai lagoon is shown in Fig. 3. Comparison of results with studies in China showed high levels of HCHs residues with levels 1.02 ng/g (Hongxuan and Weiguo, 2016). Meanwhile, the concentration of pesticides in the sediment at the study sites is shown through the relatively high levels for Heptachlor and Endosulfan. This can be explained by the use of pesticides in agricultural production and through the erosion process, with the contribution of rain as one of the causes of pesticides accumulation in sediments (Bian et al., 2010). Due to the development of agricultural and aquacultural activities is increasing the pressure on the environment by transfer fertilizers and pesticides into the lagoon system (Nguyen and de Vries, 2009). The economic activities is an important reason impact on sediment and water quality of the lagoon. In addition, active ingredients such as Endosulfan are derived from their use in rat-killing activities during animal husbandry or agricultural production (Carriger et al., 2011; De Roma et al., 2017). In the Cau Hai lagoon, fresh water is coming into the lagoon via the rivers. The rivers flow through urban areas and carry a lot of sediment to the lagoon which may be contained the compounds of OCPs. The sediment transported into the lagoon happens mostly in the flooding. The total sediment transport from the rivers through the Thuan An inlet is 1.43 mm^3/year during the flood season (Nghiem et al., 2008). Moreover, to evaluate the relationship between the parameters of pesticides in sediments in Cau Hai lagoon, correlation analysis results are shown in Table 4.

Table 4. Analysis of correlation among parameters of pesticides in sediment

	α- HCH	β-HCH	δ-HCH	Heptachlor	Endosulfan
α-HCH	1.000				
β-HCH	0.253*	1.000			
δ-HCH	0.515*	-0.312*	1.000		
Heptachlor	-0.345	-0.219	0.633	1.000	
Endosulfan	-0.612*	-0.161	0.690*	0.782*	1.000

**Correlation is significant at the 0.05 level (2-tailed); n=21.*

Correlation analysis of parameters of pesticides parameters in sediments was conducted in previous studies to find a link between them (Sun et al., 2008; Dirbaba et al., 2018). In this study, the results of correlation analysis showed the relationship among α- HCH and, β-HCH, δ-HCH and Endosulfan ($p<0.05$). In particular, the correlation between α-HCH <> β-HCH, α-HCH <> δ-HCH and α-HCH <> Endosulfan were 0.253, 0.515 and -0.612, respectively. The content of β-HCH in sediments and δ-HCH shows a negative correlation with the corresponding coefficient of -0.312 ($p < 0.05$). As for the Endosulfan parameter, there is also a good relationship with δ-HCH and Heptachlor and Pearson correlation is quite high, respectively 0.690, 0.782 ($p < 0.05$). Comparison with another study showed a positive correlation between the content of ∑HCHs <> Heptachlor ($r = 0.560$, $p = 0.01$), Heptachlor <> Heptachlor epoxide ($r = 0.536$, $p = 0.01$) and that indicates the distribution of pesticides related to potential sources of pollution (Dirbaba et al., 2018).

3.3. Risk assessment of pesticides in sediments

OCPs is known as an ecological toxin, persistent and highly bioaccumulating (Bai et al., 2015; Ahmed et al., 2015). Exposure to active ingredients such as HCHs and DDTs can cause serious health effects such as endocrine disorders, cancer and neurological and immune problems (Ize-Iyamu et al., 2007). To assess risks, the study uses Canadian sediment quality guidelines to consider the ecological risks of Cau Hai lagoon (Canadian Council of Ministers of the Environment, 2002). Accordingly, the threshold value of TELs impact of DDD, DDE and DDT respectively 1.22, 2.07, 1.19 ng/g, while the results of sediment analysis in Cau Hai are below the detection threshold. The results showed that the content of some pesticides in the sediment was very low and ecologically safe. However, the analysis results showed the presence of other active substances such as HCHs, Heptachlor, Aldrin, Endosulfan. In recent priods the water dynamics is decreasing again and the sediments is recorded as an increase of porosity and a decrease of the content of sand (Frignani et al., 2007). Therefore, they can be stored and bioaccumulated through food chains, affecting human health as well as affecting ecosystems. The interventions to reduce the erosion are needed to protect the lagoon and the people living in Cau Hai lagoon (Eriksson and Persson, 2014). The erosion might cause great problems for local public around the lagoon and lead to the risk, changed sediment transport and water quality.

Table 5. Acceptable limits of pesticides in sediments

No.	Parameter	Canadian regulations on sediment quality (ng/g)	
		Temporary impact threshold (ISQG/TELs)	Affect level (PELs)
1	Aldrin and Dieldrin		
2	Dieldrin	0.71	4.3
3	Endrin	2.67	62.4
4	DDD	1.22	7.81
5	DDE	2.07	374
6	DDT	1.19	4.77
7	DDT, DDD and DDE		
8	Heptachlor (Heptachlor epoxide)	0.6	2.74
9	Heptachlor and Heptachlor epoxide		
10	HCH	0.32	0.99

Note: <TELs: Rarely harmful; PELs ≤ and ≥ TELs: Some cases may be harmful; >PELs: Constantly harmful.

In general, sediments in ponds and lakes are considered as places for sustainable storage of organic matter such as pesticides and will affect human health through bioaccumulation (Farshid, 2015; Unyimadu et al., 2018). Because of the rapid industrial development, the risks of pollution levels need to alarm for local community around lagoon areas (Thanh et al., 2004). The results show that the concentration of Heptachlor ranges from 2.98 ± 0.23 to 27.56 ± 4.56 ng/g. Heptachlor content in sediment at all monitoring points S1-7 exceeded the PELs influence level (>2.74 ng/g) according to Canadian environmental quality guidelines. In particular, the positions S1, S6, S7 with Heptachlor amount exceeds the value of PELs many times. The remaining content values such as Aldrin fluctuated from 0.11 ± 0.03 to 2.00 ± 1.02 ng/g; Heptachlor epoxide approx. 0.02 ± 0.01 to 0.04 ± 0.02 ng/g; and Endosulfan reach the threshold of 14.16 ± 2.32 to 21.30 ± 2.39 ng/g. In addition, p,p'-DDE, m,p'-DDD values were not detected within the method limit of analysis.

4. Conclusion

Investigation of fluctuations of pesticides concentration by the analyzing process in Cau Hai lagoon area has good recovery and repeatability. The analytical results show that in Cau Hai sediment contains a concentration of some pesticides with high concentrations such as α-HCH, β-HCH, δ-HCH, Heptachlor, Aldrin. The correlation analysis process showed the relationship of some pesticides, such as α-HCH, β-HCH, δ-HCH and Endosulfan ($p < 0.05$). The results have shown some risks of pesticide contamination in sediments affecting bottom organisms of lagoon ecosystems as well as public health issues. Thus, the research is a premise and basis for providing useful scientific information to local communities in the consumption of some benthic species in Cau Hai lagoon, Central Vietnam. In the future, research should be conducted with a comprehensive assessment of the accumulation mechanism of these chemicals in relation to sediment samples and benthic organisms in the lagoon ecosystem.

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