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Research article

Distribution of heavy metals and persistent organic pollutants in sediment from coastal areas of northern and middle Viet Nam

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ABSTRACT

Heavy metals and persistent organic pollutants (POPs) in surface and core sediments can reflect the status and trend of pollution in the study area and help to assess negative impacts on the ecosystem. However, comprehensive studies dealing with multiple classes of inorganic and organic contaminants, such as toxic heavy metals and POPs, in sediment core samples in Vietnamese coastal areas have been relatively limited in recent years. In this study, the concentrations of heavy metals and several groups of POPs, including organochlorine pesticides (OCPs), polychlorinated biphenyls (PCBs), and polybrominated diphenyl ethers (PBDEs), in surface and core sediment samples collected from estuarine areas in northern and central Vietnam were determined to evaluate their bioaccumulation characteristics in both special and temporal terms. The accumulation pattern of POPs in surface sediments in some estuaries along central coasts was in the order of PCBs > PBDEs > DDTs > HCHs. The concentrations of PCBs and PBDEs in surface sediments showed more obvious variations than did those of DDTs and HCHs. The residue concentrations of OCPs reached their highest values in the sediment layers corresponding to the late 1960s and early 1980s, and the peak concentrations of PCBs in the sediment layer occurred in the 1970s. The concentrations of total PBDEs, BDE-209, and several heavy metals, such as Cd, Cr, Cu, Pb, and Zn, in the core sediments of the Day River have tended to increase gradually in recent years. Our results of POP and metal accumulation in sediment core layers generally reflect the pattern of usage in Vietnam, such as the effect of the ban of OCPs and the recent input of emerging industrial chemical PBDEs into coastal areas.

Key words: Heavy metals, OCPs, PCBs, PBDEs, Sediment, Vietnam

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INTRODUCTION

Organochlorine pesticides (OCPs), polychlorinated biphenyls (PCBs) and polybrominated diphenyl ethers (PBDEs) are persistent organic pollutants (POPs) of major concern in marine environments due to their toxic impacts on wildlife and humans. These contaminants have high bioaccumulation potential in marine environments and living organisms. Elevated residue concentrations of POPs and toxic heavy metals in sediments and tissues can exert adverse effects on aquatic organisms, such as impaired reproduction, immune suppression, and endocrine disruption [1,2].

Sediments are important environmental components that are often studied to assess the pollution level and ecological risks of heavy metals and organic substances in aquatic environments [3,4]. Surface sediments can reflect the current status of pollution, while core sediment samples can provide useful information about the temporal trend of the accumulation of POPs and heavy metals [5].

Earlier studies have reported heavy metal pollution in surface sediments in several river basins and estuaries in Vietnam [6,7]. Tong et al. (2023) reported that the concentrations of heavy metals in sediments in the Saigon River basin, City Ho Chi Minh City, during the rainy season (Zn: 691–2002; Cr: 250–552; Cu: 78.0–435; Pb: 10.0–342 μg/g) were significantly greater than those during the dry season (Zn: 247–725; Cr: not detected–145; Cu: not detected–88 μg/g; Pb: not detected) [6]. The concentrations of these metals were significantly lower in sediment samples taken from the Ganh Rai Bay area, Vung Tau city (Zn: 20.2–77.1; Cr: 21.0–64.4; Cu: 3.9–29.7; Pb: 8.5–27.7; Cd: 0.04–0.21 μg/g) [7]. A decreasing trend in pollution levels from river areas to estuaries and bays was reported for organic substances such as polychlorinated biphenyls (PCBs) and organochlorine pesticides (OCPs) [8]. The simultaneous examination of OCPs, PCBs, and PBDEs was also conducted on surface and core sediment samples collected in the coastal areas of central Vietnam [9,10]. Nevertheless, comprehensive investigations on the spatial and vertical distributions of multiple

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classes of environmental contaminants, such as POPs and heavy metals, in sediment samples from a wide range of coastal areas of northern and central Vietnam are still limited.

In this study, the residue concentrations of several heavy metals (Cd, Cr, Cu, Pb, and Zn) and three groups of POPs (PCBs, OCPs, and PBDEs) were determined in surface and sediment core samples collected from different coastal areas in northern and central Vietnam to understand their accumulation characteristics and trends of accumulation in both spatial and temporal terms. OCPs such as hexachlorocyclohexanes (HCHs) and dichlorodiphenyltrichloroethane and its metabolites (DDTs) have been extensively used in many developing countries, including Vietnam, for agricultural and public health purposes, and the contamination status of these chemicals in different environmental samples in Vietnam has been previously reported, as reviewed by Minh et al. (2008)[11]. Moreover, PCBs are an important group of industrial chemicals that are widely used as dielectric and coolant fluids in electrical equipment and as additives in many industrial and consumer products. PBDEs, important brominated flame retardants, are used as additive flame retardants in various polymers and textiles and exist in electrical and electronic appliances, building materials, and automobiles. These pollutants have already been added to the list of chemicals that need to be banned or regulated under the Stockholm Convention[11,12].

METHODOLOGY

Collection and preparation of sediment samples

Surface sediment samples (0 - 20 cm) were collected in several central coastal areas, including Cua Hoi (n = 7), Nhat Le (n = 8), Cua Viet (n = 6), Cua Dai (n = 8), Lang Co (n = 5), and the Han River (n = 6) (for POP analysis), using a sampled Peterson grab (Figure 1). Core sediment samples were taken from the Nhat Le Estuary (n = 9), Quang Binh Province (n = 8), Cua Dai Estuary, Quang Nam Province (n = 7) (for POP analysis) and Day Estuary, Ha Nam Province (n = 8) (for heavy metal analysis) using gravity tube sampling. These are important estuaries and beaches in central Vietnam with extensive tourism and business activities. The surface sediment samples were transferred to glass containers, stored in a cooler, and transported to the laboratory. The samples were dried to a constant weight at room temperature in the dark in the laboratory, crushed in a ceramic mortar, passed through a 2 mm sieve, and stored in a glass jar at -20°C until analysis. For core sediment samples, the age of the sediment layers was determined by geochemical methods using the ^{210}Pb isotope, as described in detail in Cuesta et al. (2022)[13]. Briefly, the ^{210}Pb isotope was determined by an alpha-particle spectrometric method though the daughter ion ^{209}Po. The sample was extracted with tributyl phosphate. Po isotopes were deposited in a HCl acid solution onto silver disks, and ascorbic acid was added with gentle agitation. After deposition, the disks were washed with distilled water and counted by alpha spectrometry. The constant initial concentration (CIC) model was used to assess the accumulation rate and dating of the cores.

Heavy metal analysis

The concentrations of heavy metals (Cd, Cr, Cu, Pb, Zn) in the samples were determined using the standard method 3050B of the United States Environmental Protection Agency (US EPA). Approximately 1.0 g of sediment was digested with 10 mL of nitric acid solution (1:1) at 95°C until no brown gas was released. The sample mixture was then cooled to room temperature, and a 30% hydrogen peroxide solution was added. The mixture was then heated at 95°C for 30 minutes. The sample solution was filtered through a 0.45 μm filter and filled with 2% nitric acid solution to 50 mL. This sample solution was analyzed on an inductively coupled plasma–mass spectrometry system (ICP/MS).

POP analysis

For PCBs and OCPs, approximately 20 g of sediment sample was Soxhlet extracted with an acetone/hexane mixture (1:1, v/v) for 16 h. The extract was then concentrated, and the colored organic compounds and sulfur were removed by concentrated sulfuric acid, metallic copper, gel permeation chromatography, and a column containing Florisil adsorbent. The PCBs (CB-28, -52, -101, -138, -153 and -180) and OCPs, including the hexachlorocyclohexane group (α-HCH, β-HCH, γ-HCH, δ-HCH) and the dichlorodiphenyltrichloroethane group (4,4'-DDT, 4,4'-DDD, 4,4'-DDE), were analyzed on a gas chromatography system coupled to an electron capture detector (GC-2010 ECD-2010, Shimadzu, Japan) with a DB-5 capillary column (30 m $\times$ 0.25 mm $\times$ 0.25 μm, Agilent Technologies).

For PBDEs, approximately 20 g of sediment sample was Soxhlet extracted with an acetone/hexane mixture (1:1, v/v) for 16 h. The extract was then concentrated and cleaned with concentrated sulfuric acid,

Figure 1: **Map showing sampling locations for sediment samples.**

metallic copper, and a multilayer column containing silica gel, silica gel impregnated with 44% H_2SO_4, silica gel impregnated with 2% KOH, and silica gel impregnated with 10% $AgNO_3$. PBDEs (BDE-28, BDE-47, BDE-99, BDE-100, BDE-153, BDE-154, BDE-183, and BDE-209) were analyzed on a gas chromatography coupled mass spectrometry detector (GC 7890B MS 5977A, Agilent Technologies) with an Rtx-1614 capillary column (15 m × 0.25 mm × 0.1 μm, Restek).

RESULTS AND DISCUSSION

Heavy metals in sediment core samples

The heavy metal concentrations in the sediment core samples from the Day River are shown in Figure 2. The accumulation patterns (mean and range concentration, mg/g dry wt.) were in the order of Zn (89,2; 35,7–215) > Cr (65,8; 43,3–80,2) > Pb (38,9; 18,5–52,6) > Cu (34,2; 23,0–46,9) > Cd (0,40; 0,19–0,61). The concentrations of Zn, Cr, Pb, Cu, and Cd exhibited peaks in the layers corresponding to the mid-1970s, 1990s, and 2010s. Generally, residue levels tend to increase toward the upper sediment layers, which may be related to rapid industrial and economic growth in recent years. An interesting result was observed for Cd, which gradually increased from the mid-1980s to the early 2010s. This result is similar to that found in the Thi Vai estuary, southern Vietnam, where the residue concentrations of Cd increased during the 2002-2012 period [14].

The concentrations of heavy metals in sediment core samples from the Day River were generally lower than those from the Saigon River, Ho Chi Minh City [6], and higher than those from Vung Tau City [7]. The relatively high heavy metal concentrations in the Saigon River area reflect the discharge of inadequately treated wastewater from industrial zones and residential areas into the environment. The concentrations of Zn, Cr, Cu and Pb in the wet season were greater than those in the dry season, probably due to the impacts of more extensive human activities in the rainy season. Our previous study also revealed greater levels of OCP and PCB residues in the rainy season in the Cua Dai Estuary, which could be due to the rapid increase in tourism activities in Cua Dai – Hoi An Port during the summer [9]. In the present study, heavy metal concentrations in sediment from the Day River were moderate or low. In particular, the concentrations of Zn, Cu, Pb, Cr, and Cd were still lower than the guideline values of the Vietnamese Government in the National Technical Regulation on Sediment Quality (QCVN 43:2017/BTNMT). The low accumulation of heavy metals in coastal sediments is probably due to

the deposition or dilution of these substances during transport from inland sources.

The concentrations of heavy metals in the sediment core samples from the Day River generally tended to increase gradually to the upper layers, reflecting that the pollution tended to increase over time. A gradual increase in heavy metal concentrations over three decades (from approximately 1985 to 2015) was more clearly observed for Zn and Cd, followed by Cr and Cu. Pb is the metal with the slightest fluctuation. A similar trend was also observed in sediment cores from Shuanglong, China [15], and the Pasur River, Bangladesh [16]. The increase in heavy metal concentrations in the sediment cores over time can be explained by the recent rapid increase in production and human activities in the study area [15,16].

POP accumulation in surface and sediment core samples

Concentrations of HCHs, DDTs, PCBs and PBDEs were detected in surface sediments collected from various sites along coastal areas of middle Vietnam, indicating the widespread occurrence of these contaminants in estuary environments (Figure 3). The accumulation patterns (mean and range concentrations) were in the order of PBDEs (75; 12–310), PCBs (69; 10–330), DDTs (3,2; 0,44–27), and HCHs (3,1; 0,49–23) ng/g dry wt. The highest concentrations of PBDEs and PCBs were detected in the sediments collected from Nhat Le and Cua Dai, respectively. DDTs and HCHs were elevated in the Nhat Le and Han Rivers. Gamma HCHs and 4,4'-DDT were the predominant compounds for HCHs and DDTs, respectively.

The sediment core samples from Nhat Le at a depth of 50 cm correspond to the period from 1960–2015. The residue concentrations followed the order of PBDEs (85; 18–280) > PCBs (21; 6,7–39) > DDTs (2,5; 1,4–5,2) $\geq$ HCHs (2,4; 1,9–3,8) ng/g. In the Cua Dai estuary, the pattern was PCBs (41; 22-91) > PBDEs (35; 13-54) > DDTs (1,9; 0,70-3,7) $\geq$ HCHs (1,8; 0,90-3,2) ng/g.

The residue concentrations of PCBs and PBDEs were generally greater than those of HCHs and DDTs in surface and sediment core samples from central coastal areas. This result suggests that banned pesticides are no longer used in Vietnam and reflects the effectiveness of environmental regulations. The highest concentrations of OCPs appeared in the layers corresponding to the years 1960 to 1990 [10]. Regarding PCBs, sediment cores at Nhat Le and Cua Dai have high levels of PCBs corresponding to the layers from 1960 to 1970, which was the period when PCBs were produced and extensively used worldwide [10]. After commercial PCB mixtures began to be limited in terms of production and use in the 1970s, PCB contamination in the environment generally decreased, which was consistent with the results of sediment sample analyses in Vietnam and Japan [17]. However, PCB concentrations have tended to increase in recent years at some survey sites, reflecting emissions from unintentional sources. The concentrations of PBDEs increased from approximately 1970–1980 to the present. Although commercial mixtures of PBDEs have been listed as chemicals for phase-out under the Stockholm Convention, these compounds may continue to be released into the environment during industrial and consumer product use, storage, and disposal [18].

In general, the residue concentrations of DDT, HCH and PCBs in surface sediment from Nhat Le and Cua Dai were similar to those reported in our previous survey [19] and higher than those in some tidal flat areas along the northern coasts from Mong Cai to the Ba Lat estuary [20] (Nhon et al. 2014). The levels were in a similar range or lower than those in sediment collected from the Red River areas from Hanoi to Nam Dinh Province, northern Vietnam [21] (Tham et al. 2022). Data on PBDEs in estuary sediment from Vietnam are rather limited. PBDEs in the Nhat Le and Cua Dai Estuary in this study were greater than those reported in urban sediment from the Hanoi and Hochiminh city metropolitan areas [22–24]. The relatively high PCB and PBDE levels in sediment from the Cua Dai estuary suggest the effects of riverine discharge and various anthropogenic sources, including extensive tourism activities, in Da Nang city.

CONCLUSION

This study provides an overall picture of heavy metal pollution and the presence of several typical POP groups (HCHs, DDTs, PCBs and PBDEs) in surface and sediment core samples from northern and central Vietnamese estuary areas. The residue concentrations of OCPs tend to decrease, while those of PCBs, PBDEs and some heavy metals tend to increase in the upper sediment layers, suggesting that emissions from industrialization and urbanization have entered estuary environments in recent years. These results suggest the need for continued monitoring and assessment studies on heavy metals and POPs in Vietnam sediments, aiming at controlling and safely managing these substances in coastal environments.

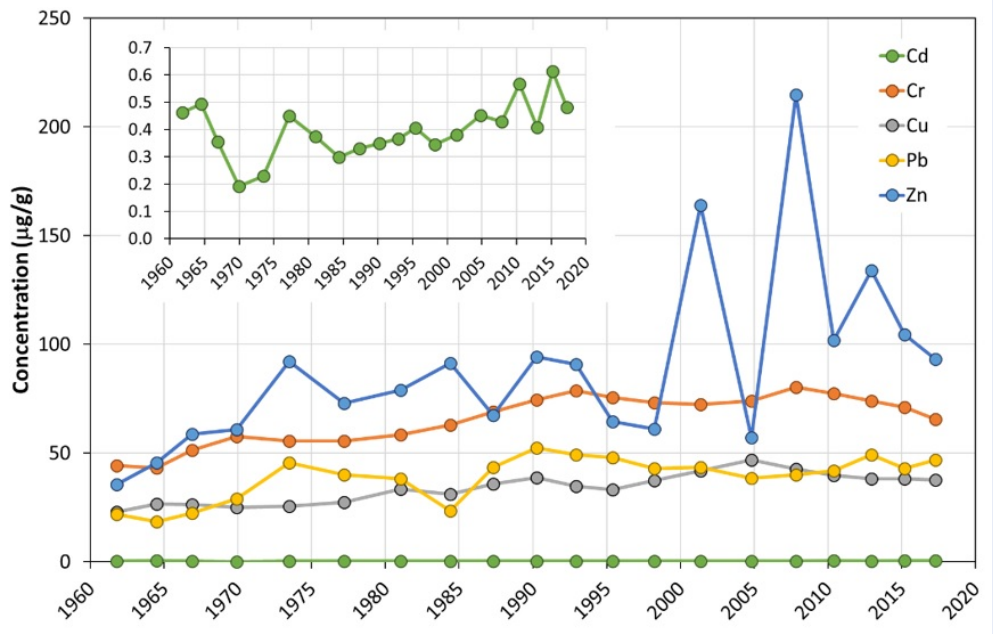

Figure 2: **Concentrations and depth profiles of heavy metals in sediment core samples from Day River, North Vietnam, showing increasing trend of Cd and some other elements such as Cr, Zn**. The age of the sediment layers was determined by geochemical methods using the 210Pb isotope.

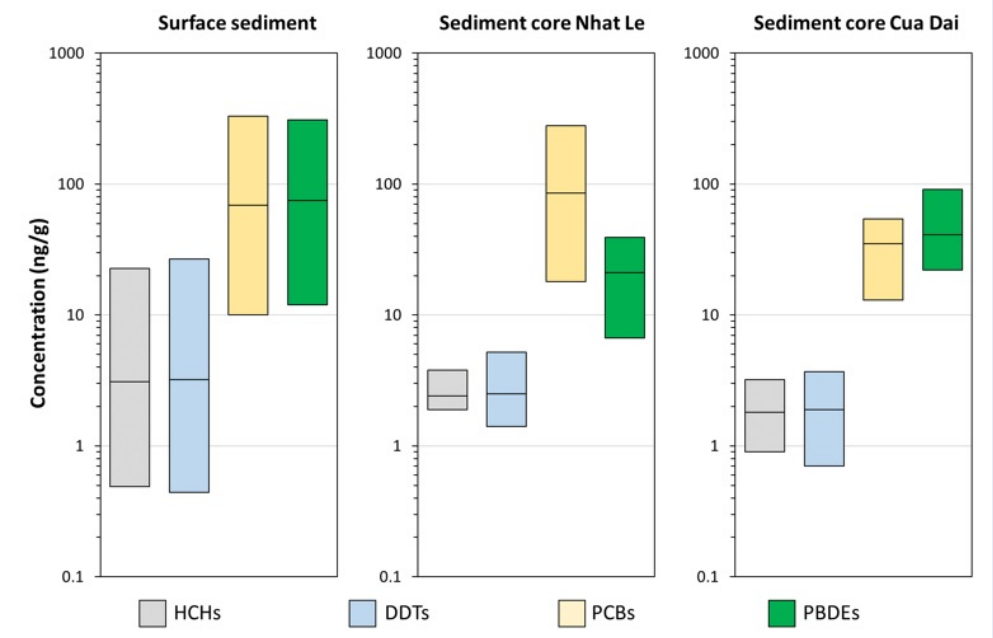

Figure 3: **Mean and range concentrations of HCHs, DDTs, PCBs, and PBDEs in surface and sediment core samples from coastal areas of middle Vietnam, indicating remarkably higher residues of PCBs and PBDEs.** Concentrations of contaminants were determined by a gas chromatography coupled with mass spectrometry detector (GC-MS).

LIST OF ABBREVIATION

GC/MS: gas chromatography–mass spectrometry
ICP-MS: Inductively coupled plasma–mass spectrometry
OCPs: organochlorine pesticides
PBDEs: Polybrominated diphenyl ethers
PCB: Polychlorinated biphenyl
POPs: Persistent organic pollutants

CONFLICT OF INTEREST

There are no conflicts of interest for any of the authors.

AUTHORS CONTRIBUTION

Bui Thi Phuong: experiment, data analysis, manuscript preparation
Trinh Thi Tham: experiment, data analysis, manuscript preparation
Trinh Thi Thuy: experiment, data analysis
Hoang Quoc Anh: experiment, data analysis, manuscript preparation
Tu Binh Minh: supervision, conceptualization, manuscript revision.
Nguyen Manh Ha: sample collection, data analysis
Dang Minh Huong Giang: sample collection, data analysis
Nguyen Thi Hong Yen: experiment, data analysis
All the authors commented on the manuscript and read and approved the final version.

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