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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

INTRODUCTION

² Organochlorine pesticides (OCPs), polychlorinated
³ biphenyls (PCBs) and polybrominated diphenyl
⁴ ethers (PBDEs) are persistent organic pollutants
⁵ (POPs) of major concern in marine environments
⁶ due to their toxic impacts on wildlife and humans.
⁷ These contaminants have high bioaccumulation
⁸ potential in marine environments and living or⁹ ganisms. Elevated residue concentrations of POPs
¹⁰ and toxic heavy metals in sediments and tissues can
¹¹ exert adverse effects on aquatic organisms, such as
¹² impaired reproduction, immune suppression, and
¹³ endocrine disruption ^{1,2}.

14 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 metals⁵.

Earlier studies have reported heavy metal pollution in 22 surface sediments in several river basins and estuar- 23 ies in Vietnam^{6,7}. Tong et al. (2023) reported that ²⁴ the concentrations of heavy metals in sediments in the 25 Saigon River basin, City Ho Chi Minh City, during the 26 rainy season (Zn: 691-2002; Cr: 250-552; Cu: 78.0-27 435; Pb: 10.0–342 μ g/g) were significantly greater ²⁸ than those during the dry season (Zn: 247-725; Cr: 29 not detected-145; Cu: not detected-88 µg/g; Pb: not 30 detected)⁶. The concentrations of these metals were 31 significantly lower in sediment samples taken from 32 the Ganh Rai Bay area, Vung Tau city (Zn: 20.2-77.1; 33 Cr: 21.0-64.4; Cu: 3.9-29.7; Pb: 8.5-27.7; Cd: 0.04-0.21 μ g/g)⁷. A decreasing trend in pollution levels 35 from river areas to estuaries and bays was reported for organic substances such as polychlorinated biphenyls 37 (PCBs) and organochlorine pesticides (OCPs)⁸. The 38 simultaneous examination of OCPs, PCBs, and PB-DEs was also conducted on surface and core sediment 40 samples collected in the coastal areas of central Vietnam^{9,10}. Nevertheless, comprehensive investigations 42 on the spatial and vertical distributions of multiple 43

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- ⁴⁴ classes of environmental contaminants, such as POPs⁴⁵ and heavy metals, in sediment samples from a wide
- ⁴⁶ range of coastal areas of northern and central Viet-⁴⁷ nam are still limited.
- In this study, the residue concentrations of several 48 heavy metals (Cd, Cr, Cu, Pb, and Zn) and three 49 groups of POPs (PCBs, OCPs, and PBDEs) were de-50 termined in surface and sediment core samples col-51 lected from different coastal areas in northern and 52 central Vietnam to understand their accumulation characteristics and trends of accumulation in both 54 spatial and temporal terms. OCPs such as hex-55 achlorocyclohexanes (HCHs) and dichlorodiphenyltrichloroethane and its metabolites (DDTs) have been 57 extensively used in many developing countries, in-58 cluding Vietnam, for agricultural and public health 59 purposes, and the contamination status of these chemicals in different environmental samples in Vietnam has been previously reported, as reviewed by 62 Minh et al. (2008)¹¹. Moreover, PCBs are an important group of industrial chemicals that are widely used 64 65 as dielectric and coolant fluids in electrical equipment and as additives in many industrial and consumer products. PBDEs, important brominated flame retar-67 dants, are used as additive flame retardants in vari-68 ous polymers and textiles and exist in electrical and electronic appliances, building materials, and auto-70 mobiles. These pollutants have already been added to 71 the list of chemicals that need to be banned or regu-⁷³ lated under the Stockholm Convention^{11,12}.

74 **METHODOLOGY**

75 Collection and preparation of sediment76 samples

Surface sediment samples (0 - 20 cm) were collected 77 in several central coastal areas, including Cua Hoi (n 78 = 7), Nhat Le (n = 8), Cua Viet (n = 6), Cua Dai (n = 6)79 8), Lang Co (n = 5), and the Han River (n = 6) (for POP analysis), using a sampled Peterson grab (Fig-81 ure 1). Core sediment samples were taken from the 82 Nhat Le Estuary (n = 9), Quang Binh Province (n =83 8), Cua Dai Estuary, Quang Nam Province (n = 7)84 (for POP analysis) and Day Estuary, Ha Nam Province 85 (n = 8) (for heavy metal analysis) using gravity tube 86 sampling. These are important estuaries and beaches in central Vietnam with extensive tourism and busi-88 ness activities. The surface sediment samples were 89 transferred to glass containers, stored in a cooler, and 90 transported to the laboratory. The samples were dried 91 to a constant weight at room temperature in the dark 92 93 in the laboratory, crushed in a ceramic mortar, passed $_{94}$ 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 ²¹⁰Pb isotope, as described in detail in Cuesta et al. (2022)¹³. Briefly, the ²¹⁰Pb isotope was determined by an alpha-particle spectrometric method though the daughter ion ²⁰⁹Po. The 100 sample was extracted with tributyl phosphate. Po isotopes were deposited in a HCl acid solution onto sil- 102 ver disks, and ascorbic acid was added with gentle ag- 103 itation. After deposition, the disks were washed with 104 distilled water and counted by alpha spectrometry. 105 The constant initial concentration (CIC) model was 106 used to assess the accumulation rate and dating of the 107 cores. 108

Heavy metal analysis

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The concentrations of heavy metals (Cd, Cr, Cu, Pb, 110 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 μ m 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 sed- 125 iment sample was Soxhlet extracted with an ace- 126 tone/hexane mixture (1:1, v/v) for 16 h. The ex- 127 tract was then concentrated, and the colored organic 128 compounds and sulfur were removed by concentrated 129 sulfuric acid, metallic copper, gel permeation chro- 130 matography, and a column containing Florisil ad- 131 sorbent. The PCBs (CB-28, -52, -101, -138, -153 132 and -180) and OCPs, including the hexachlorocyclo- 133 hexane group (α -HCH, β -HCH, γ -HCH, δ -HCH) ¹³⁴ and the dichlorodiphenyltrichloroethane group (4,4'- 135 DDT, 4,4'-DDD, 4,4'-DDE), were analyzed on a gas 136 chromatography system coupled to an electron cap- 137 ture detector (GC-2010 ECD-2010, Shimadzu, Japan) 138 with a DB-5 capillary column (30 m imes 0.25 mm imes 139 0.25 µm, Agilent Technologies). 140

For PBDEs, approximately 20 g of sediment sample 141 was Soxhlet extracted with an acetone/hexane mix- 142 ture (1:1, v/v) for 16 h. The extract was then con- 143 centrated and cleaned with concentrated sulfuric acid, 144



Figure 1: Map showing sampling locations for sediment samples.

¹⁴⁵ metallic copper, and a multilayer column containing ¹⁴⁶ silica gel, silica gel impregnated with 44% H₂SO₄, sil-¹⁴⁷ ica gel impregnated with 2% KOH, and silica gel im-¹⁴⁸ pregnated with 10% AgNO₃. PBDEs (BDE-28, BDE-¹⁴⁹ 47, BDE-99, BDE-100, BDE-153, BDE-154, BDE-183, ¹⁵⁰ and BDE-209) were analyzed on a gas chromatogra-¹⁵¹ phy coupled mass spectrometry detector (GC 7890B ¹⁵² MS 5977A, Agilent Technologies) with an Rtx-1614 ¹⁵³ capillary column (15 m × 0.25 mm × 0.1 μ m, Restek).

155 RESULTS AND DISCUSSION

156 Heavy metals in sediment core samples

The heavy metal concentrations in the sediment core 157 samples from the Day River are shown in Figure 2. 158 The accumulation patterns (mean and range concen-159 tration, mg/g dry wt.) were in the order of Zn (89,2; 160 35,7-215) > Cr (65,8; 43,3-80,2) > Pb (38,9; 18,5-161 52,6) > Cu (34,2; 23,0-46,9) > Cd (0,40; 0,19-0,61). 162 The concentrations of Zn, Cr, Pb, Cu, and Cd ex-163 hibited peaks in the layers corresponding to the mid-164 1970s, 1990s, and 2010s. Generally, residue levels 165 tend to increase toward the upper sediment layers, 166 which may be related to rapid industrial and eco-168 nomic growth in recent years. An interesting result 169 was observed for Cd, which gradually increased from

the mid-1980s to the early 2010s. This result is sim- 170 ilar to that found in the Thi Vai estuary, southern 171 Vietnam, where the residue concentrations of Cd in- 172 creased during the 2002-2012 period 14. 173 The concentrations of heavy metals in sediment core 174 samples from the Day River were generally lower than 175 those from the Saigon River, Ho Chi Minh City⁶, and 176 higher than those from Vung Tau City⁷. The rela- 177 tively high heavy metal concentrations in the Saigon 178 River area reflect the discharge of inadequately treated 179 wastewater from industrial zones and residential ar- 180 eas into the environment. The concentrations of Zn, 181 Cr, Cu and Pb in the wet season were greater than 182 those in the dry season, probably due to the impacts 183 of more extensive human activities in the rainy sea- 184 son. Our previous study also revealed greater levels of 185 OCP and PCB residues in the rainy season in the Cua 186 Dai Estuary, which could be due to the rapid increase 187 in tourism activities in Cua Dai – Hoi An Port dur- 188 ing the summer⁹. In the present study, heavy metal 189 concentrations in sediment from the Day River were 190 moderate or low. In particular, the concentrations 191 of Zn, Cu, Pb, Cr, and Cd were still lower than the 192 guideline values of the Vietnamese Government in the 193 National Technical Regulation on Sediment Ouality 194 (QCVN 43:2017/BTNMT). The low accumulation of 195 heavy metals in coastal sediments is probably due to 196

- 197 the deposition or dilution of these substances during198 transport from inland sources.
- 198 transport from mand sources.

The concentrations of heavy metals in the sediment 199 core samples from the Day River generally tended 200 to increase gradually to the upper layers, reflecting 201 that the pollution tended to increase over time. A 202 gradual increase in heavy metal concentrations over 203 three decades (from approximately 1985 to 2015) was 204 more clearly observed for Zn and Cd, followed by Cr 205 and Cu. Pb is the metal with the slightest fluctua-206 tion. A similar trend was also observed in sediment cores from Shuanglong, China¹⁵, and the Pasur River, 208 Bangladesh¹⁶. The increase in heavy metal concen-209 trations in the sediment cores over time can be explained by the recent rapid increase in production and 211 human activities in the study area^{15,16}. 212

213 POP accumulation in surface and sediment214 core samples

Concentrations of HCHs, DDTs, PCBs and PBDEs 215 were detected in surface sediments collected from var-216 ious sites along coastal areas of middle Vietnam, indi-217 cating the widespread occurrence of these contami-218 nants in estuary environments (Figure 3). The accu-219 mulation patterns (mean and range concentrations) 220 221 were in the order of PBDEs (75; 12-310), PCBs (69; 10-330), DDTs (3,2; 0,44-27), and HCHs (3,1; 0,49-222 23) ng/g dry wt. The highest concentrations of PBDEs 223 and PCBs were detected in the sediments collected 224 from Nhat Le and Cua Dai, respectively. DDTs and HCHs were elevated in the Nhat Le and Han Rivers. 226 227 Gamma HCHs and 4.4'-DDT were the predominant compounds for HCHs and DDTs, respectively. 228

²²⁹ The sediment core samples from Nhat Le at a depth of ²³⁰ 50 cm correspond to the period from 1960–2015. The

²³¹ residue concentrations followed the order of PBDEs

232 (85; 18-280) > PCBs (21; 6,7-39) > DDTs (2,5; 1,4-

 $_{233}$ 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; $_{235}$ 13-54) > DDTs (1,9; 0,70-3,7) \geq HCHs (1,8; 0,90-3,2)

236 ng/g.

²³⁷ The residue concentrations of PCBs and PBDEs were ²³⁸ generally greater than those of HCHs and DDTs ²³⁹ in surface and sediment core samples from central ²⁴⁰ coastal areas. This result suggests that banned pes-²⁴¹ ticides are no longer used in Vietnam and reflects ²⁴² the effectiveness of environmental regulations. The ²⁴³ highest concentrations of OCPs appeared in the lay-²⁴⁴ ers corresponding to the years 1960 to 1990¹⁰. Re-²⁴⁵ garding PCBs, sediment cores at Nhat Le and Cua Dai ²⁴⁶ have high levels of PCBs corresponding to the lay-²⁴⁷ ers from 1960 to 1970, which was the period when PCBs were produced and extensively used world- 248 wide¹⁰. After commercial PCB mixtures began to be 249 limited in terms of production and use in the 1970s, 250 PCB contamination in the environment generally de- 251 creased, which was consistent with the results of sediment sample analyses in Vietnam and Japan¹⁷. How- 253 ever, PCB concentrations have tended to increase in 254 recent years at some survey sites, reflecting emissions 255 from unintentional sources. The concentrations of 256 PBDEs increased from approximately 1970-1980 to 257 the present. Although commercial mixtures of PBDEs 258 have been listed as chemicals for phase-out under the 259 Stockholm Convention, these compounds may con- 260 tinue to be released into the environment during in- 261 dustrial and consumer product use, storage, and dis- 262 posal¹⁸. 263

In general, the residue concentrations of DDT, HCH 264 and PCBs in surface sediment from Nhat Le and Cua 265 Dai were similar to those reported in our previous survey¹⁹ and higher than those in some tidal flat areas 267 along the northern coasts from Mong Cai to the Ba Lat 268 estuary²⁰ (Nhon et al. 2014). The levels were in a sim- 269 ilar range or lower than those in sediment collected 270 from the Red River areas from Hanoi to Nam Dinh 271 Province, northern Vietnam²¹ (Tham et al. 2022). 272 Data on PBDEs in estuary sediment from Vietnam are 273 rather limited. PBDEs in the Nhat Le and Cua Dai Es- 274 tuary in this study were greater than those reported in 275 urban sediment from the Hanoi and Hochiminh city 276 metropolitan areas^{22–24}. The relatively high PCB and 277 PBDE levels in sediment from the Cua Dai estuary 278 suggest the effects of riverine discharge and various 279 anthropogenic sources, including extensive tourism 280 activities, in Da Nang city. 281

CONCLUSION

This study provides an overall picture of heavy metal283pollution and the presence of several typical POP284groups (HCHs, DDTs, PCBs and PBDEs) in surface285and sediment core samples from northern and cen-286tral Vietnamese estuary areas. The residue concentra-287tions of OCPs tend to decrease, while those of PCBs,288PBDEs and some heavy metals tend to increase in the290upper sediment layers, suggesting that emissions from290industrialization and urbanization have entered estuary291ary environments in recent years. These results sug-292gest the need for continued monitoring and assess-293ment studies on heavy metals and POPs in Vietnam294sediments, aiming at controlling and safely managing295these substances in coastal environments.296

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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).

297 LIST OF ABBREVIATION

- 298 GC/MS: gas chromatography-mass spectrometry
- 299 ICP-MS: Inductively coupled plasma-mass spec-
- 300 trometry
- 301 OCPs: organochlorine pesticides
- 302 PBDEs: Polybrominated diphenyl ethers
- 303 PCB: Polychlorinated biphenyl
- 304 POPs: Persistent organic pollutants

305 CONFLICT OF INTEREST

³⁰⁶ There are no conflicts of interest for any of the authors.

308 AUTHORS CONTRIBUTION

³⁰⁹ Bui Thi Phuong: experiment, data analysis,³¹⁰ manuscript preparation

- 311 Trinh Thi Tham: experiment, data analysis,
- 312 manuscript preparation
- 313 Trinh Thi Thuy: experiment, data analysis
- 314 Hoang Quoc Anh: experiment, data analysis,
- ³¹⁵ manuscript preparation
- 316 Tu Binh Minh: supervision, conceptualization, 317 manuscript revision.
- 318 Nguyen Manh Ha: sample collection, data analysis
- 319 Dang Minh Huong Giang: sample collection, data 320 analysis
- 321 Nguyen Thi Hong Yen: experiment, data analysis
- 322 All the authors commented on the manuscript and 323 read and approved the final version.

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