Research Article

Concentration Levels and Ecological Risks of Persistent Organic Pollutants in the Surface Sediments of Tianjin Coastal Area, China

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Sediments were sampled from different surface water bodies in Tianjin coastal area, China, and persistent organic pollutants (POPs) including polycyclic aromatic hydrocarbons (PAHs), organochlorine pesticides (OCPs), polychlorinated biphenyls (PCBs), and polybrominated diphenyl ethers (PBDEs) were measured using GC/MS or GC/ECD. The purposes were to investigate the concentration levels of the POPs and to assess their ecological risks. The results showed that all the 16 priority PAHs were detected from the 10 sediments sampled with the total concentrations of the 16 PAHs ranging from 274.06 μg/kg to 2656.65 μg/kg, while the concentrations of the halogenated POPs were generally low except in the Dagu waste discharging river where the total concentration of 24 OCPs, 35 PCBs, and 14 PBDEs were 3103.36 μg/kg, 87.31 μg/kg, and 13.88 μg/kg, respectively. In the studied sediments, PAHs exhibited risks to benthonic organisms; particularly the concentrations of naphthalene and/or acenaphthene exceeded their probable effect concentrations in several locations. In comparison, only in the Dagu waste discharging river, OCPs exhibited risks with the concentrations of heptachlor epoxide and lindane exceeding their probable effect concentrations. PCBs and PBDEs posed rare risks in the studied area.

1. Introduction

Persistent organic pollutants (POPs) are organic compounds resisting degradation through chemical, biological, and photolytic processes in the environment. They can bioaccumulate through food webs from the environment and pose a risk of causing harmful effects to the ecosystem and human health according to animal experiments and epidemiological studies [1]. Common POPs such as polycyclic aromatic hydrocarbons (PAHs), organochlorine pesticides (OCPs), polychlorinated biphenyls (PCBs), and polybrominated diphenyl ethers (PBDEs) are widely detected in the environment and the organisms [2–10]. Although there are a few natural sources of POPs, most are created by humans in industrial processes (details about the source analysis of PAHs are presented in the supplemental materials available at http://dx.doi.org/10.1155/2013/417435).

Tianjin is located in the west side of Bohai Bay and the center of the circular Bohai Belt. Depending on its rich natural resources and solid industrial base, Tianjin has become one of the most active and potential regions in economic development in China. Particularly, the development and opening up of the Tianjin coastal area has been fit into the National Eleven Five program and development strategy. However, with the development of economy and urbanization, Tianjin, especially the coastal area, faces increasing environmental problems that cause harm to the health of residents and the development of the city. Researches show that the surface soils in Tianjin coastal area have been widely polluted by PAHs, with the mean value of the total concentration of the 16 priority PAHs (ΣPAH$_{16}$) being 1148.10 ng/g [11]. In the sediments of the Dagu waste discharging river, the ΣPAH$_{16}$ ranged from 370 ng/g to 5607 ng/g, and the total concentrations of 9 OCPs (ΣOCP$_9$) and 13 PBDEs (ΣPBDE$_{13}$) ranged from 325.3 ng/g to 1389.1 ng/g and 0.1 ng/g to 15.4 ng/g, respectively [12]. However, currently little is known about the concentration levels of POPs in other surface water bodies in the Tianjin coastal area.

Sediment-associated POPs are known to exhibit narcotic effects in benthic organisms, and they also have been
implicated in the development of tumors in bottom-feeding fish and in the induction of malformation, loss of fertility, or immuno deficiency in many organisms [13]. POPs can enter the aquatic food webs and pose a risk to human health via consumption of seafood [14]. It has been reported that consumption of shellfish polluted by PAHs may cause lung cancer in humans [15]. Therefore, analyzing the ecological risk of POPs is crucial for protecting human health and marine environment security.

In this study, surface sediments were sampled from different surface water bodies in Tianjin coastal area. The concentrations of POPs including PAHs, OCPs, PCBs, and PBDEs were measured and sources of the POPs were analyzed. Based on the toxicity data of benthic organisms, the ecological risks of POPs were assessed. The purposes were to investigate the concentration levels of PAHs, OCPs, PCBs, and PBDEs in the surface sediments of Tianjin coastal area and to determine their ecological risks.

2. Materials and Methods

2.1. Sampling of the Sediments. In January 2009, surface sediments were sampled from 10 locations in Tianjin coastal area, as shown in Figure 1. The 10 locations were labeled b1 to b10. Among them, b1, b4, b7, and b9 were normal water bodies, b2, b3, b5, b6, and b10 were waste discharging rivers, and b8 was waste discharging lake. The waste discharging rivers and lake were artificial excavated canals and reservoir that were mainly used for discharging waste water from industry or municipality, while normal water bodies referred to natural water bodies. The locations of two major rivers, Hai River and Dagu waste discharging river, were labeled in Figure 1. A grab dredging device was used to sample the surface sediments. The samples were collected in polyethylene bags with air being squeezed out and transported to the laboratory with ice. After arriving in the laboratory, the samples were stored in a $-20\,^\circ\text{C}$ freezer.

2.2. Analysis of Physiochemical Properties of the Sediments. Before analysis, the sediment samples were frozen dried and ground to pass through a 70 mesh metal sieve. Physicochemical properties including pH, available nitrogen (available-N), available phosphate (available-P), and total organic carbon (TOC) of the sediments were analyzed. For the pH, 5 g of each of the dried and sieved samples was thoroughly mixed with 25 mL distilled water and shaken for 3 h in a shaker; the turbid liquid was centrifuged at 5000 rpm for 10 min ($g$ value 3773), then the supernatant was filtered through 0.45 $\mu\text{m}$ filter membrane (cellulose acetate) and measured using a pH meter (METTLER DELTA320, Swiss). The available-N was measured using the alkali hydrolysis diffusion method, and the available-P was measured using the sodium bicarbonate method [16]. The TOC was measured using the TOC analyzer (TOC-5000A, Shimadzu).

2.3. Analysis of Persistent Organic Pollutants in the Sediments. Microwave-assisted extraction was applied to extract the POPs from the dried and sieved sediments. For each sample, 5 g of the sediment and 50 ng of each of the surrogate
standards (naphthalene-d_{8},acenaphthene-d_{10},anthracene-
\textit{d}_{10},chrysene-d_{12},andperylened_{12}forPAHs,2,4,5,6-
tetrachlorom-xylene for OCPs, C_{13}^\text{-}PCB-209 for PCBs, and
C_{13}^\text{-}PCB-141 for PBDEs) were added into a 55 mL extraction
vessel of the Microwave Accelerated Reaction System (CEM
MARS Xpress, USA) and extracted with 20 mL mixture of
n-hexane and acetone (1:1, v/v) at 110 °C and 1200 W for
20 min. The extract was evaporated to near dryness under
reduced pressure at 35 °C with a rotary evaporator. The
cleanup of the extract was conducted using a multilayer
chromatography column (30 cm × 10 mm i.d.). For measuring
PAHs and OCPs, a two-layer column (from bottom to
top, sequentially packed with aluminum oxide and silica
gel) was used; the extract was successively eluted with
20 mL n-hexane and 70 mL dichloromethane. For measuring
PCBs, and PBDEs, a five-layer column (from bottom to
top, sequentially packed with aluminum oxide, neutral silica
gel, alkaline silica gel, neutral silica gel, and acid silica gel)
was used; the extract was eluted with 70 mL mixture of
n-hexane and dichloromethane (1:1, v/v). The eluent was
concentrated with the rotary evaporator and transferred to
a 1.2 mL GC sample bottle. The final volume was adjusted to
approximately 1 mL under a nitrogen stream. Then, 100 ng
of each of the internal standards (2-fluoro-1,10-biphenyl
and \textit{p}-terphenyl-d_{14} for PAHs, 4,4′-dichlorobiphenyl for
OCPs, pentachloronitrobenzene for PCBs, and C_{13}^\text{-}PCB-
208 for PBDEs) was added to the GC bottle. Thereafter,
the bottle was tightly sealed up with Teflon-lined butyl
rubber septa and aluminum cap. The PAHs, OCPs, PCBs
and PBDEs were, respectively, measured with GC/MS (Agil-
ent GC6890/5973MSD, USA), GC/ECD (Agilent GC7890A,
\textit{Ni}-ECD, USA), GC/MS/MS (Varian 320-MS, USA), and
GC/NCI/MS (Agilent 7890A/5975C, USA). More details
about the measurements can be seen in [17–19].

For quality analysis and quality control, reagent and
procedure blanks were included in the measurements.
Concentrations of the studied POPs in the blanks were below
the detection limits, and recoveries of the objective compounds
in the blanks spiked with the standards ranged from 60%
to 128%. For all the sediment samples, the recoveries of
the surrogates were 59% ~ 94% for PAHs, 75% ~ 102%
for OCPs, 76% ~ 103% for PCBs, and 57% ~ 119% for
PBDEs. Two replicates were analyzed for some samples and
the relative deviation between the replicates was below 30%
for the studied POPs.

The reagents acetone, n-hexane, and dichloromethane
(analytical grade, Beijing Reagent, China) were purified by
distillation before use. Silica gel (100~200 mesh, Qingdao
Marine Chemical, China) was baked at 450 °C for 4 h and
activated at 130 °C for 16 h prior to use. The surrogate standards,
internal standards, and working standards (guarantee grade)
were purchased from J&K Chemical, USA.

2.4. Ecological Risk Assessment of the POPs in the Sediments.
Based on the toxicity data of the benthic organisms, the
threshold and probable effect concentrations (TEC and PEC)
derived from consensus-based sediment quality guidelines
for the analytes were used to assess the ecological risks of
PAHs, OCPs, and PCBs in the studied sediments [20, 21].
TEC values represent concentrations below which adverse
effects to these organisms are not likely, whereas PEC values
represent concentrations above which adverse effects are
likely. At levels between the TEC and PEC benchmarks,
incremental increases in toxicity of sediments have been
noted [20]. Two hazard quotients, one representing the
threshold effect concentration (TEC-HQ) and the other
representing the probable effect concentration (PEC-HQ),
were calculated as the ratio of the level of each POP in the
sediments to either the TEC or PEC for that POP. When TEC-
HQ values were less than 1, rare adverse ecological effects
were expected. When TEC-HQ values were greater than 1 but
PEC-HQ values were less than 1, adverse ecological effects
were possible but less frequent than those observed at the
PEC level. Finally, when PEC-HQ values were greater than 1,
frequent adverse ecological effects were expected.

Based on the toxicity data of benthic organisms [22],
the multiple-species no-observed-effect concentrations (MS
NOEC) were derived to assess the ecological risks of PBDEs
in the studied sediments [23]. The hazardous quotient (HQ)
calculated as the ratio of the measured level to the MS
NOEC was used to assess the ecological risk of each PBDE
in the sediment. When HQ values were less than 1, rare
adverse ecological effects were expected. When HQ values
were greater than 1, frequent adverse ecological effects were
expected.

3. Results and Discussion

3.1. Physicochemical Properties of the Sediments. Physico-
chemical properties of the 10 sediment samples were mea-
sured and shown in Table 1. Overall, the concentrations
of available-N in the sediments collected from waste dis-
charging rivers (b2, b3, b5, b6, b8, and b10) were greater
than those collected from normal water bodies (b1, b4,
b7, and b9), while no much difference between the nor-
mal water bodies and the waste discharging rivers was
observed for the other parameters. The highest TOC value
was detected in b1 (mouth of Hai River), followed by b3
(mouth of Dagu waste discharging river). The physicochem-
ical properties may influence the behavior of POPs in the
sediments.

3.2. Concentration Levels of POPs in the Sediments

3.2.1. Concentrations of PAHs in the Sediments. The 16
priority PAHs were all detected in the 10 sediment samples,
as shown in Table 2. Overall, the concentration levels of PAHs
in the river mouth (b1 and b3) were higher than those in the
other locations, the levels of PAHs in the waste discharging
rivers were higher than those in the normal water bodies, and
the levels of low molecular weight (LMW, 2 ~ 3 rings) PAHs
were higher than those of high molecular weight (HMW,
4 ~ 6 rings) PAHs. The ΣPAH_{200} ranged from 274.06 µg/kg
to 2656.64 µg/kg, with the mean value being 1198.51 µg/kg.
The levels of PAHs in Tianjin coastal area were lower than
### Table 1: Physicochemical properties of the sampled sediments.

| Label       | Location                   | pH | Available-N (mg/L) | Available-P (mg/L) | TOC (%) |
|-------------|----------------------------|----|-------------------|--------------------|---------|
| b1          | Mouth of Hai River         | 6.1 | 88.55             | 24.12              | 4.83    |
| b2          | Dagu waste discharge river | 8.3 | 326.48            | 16.49              | 3.18    |
| b3          | Mouth of Dagu waste discharge river | 7.1 | 104.34           | 19.11              | 4.69    |
| b4          | Offshore river             | 7.5 | 58.91             | <0.10              | 2.65    |
| b5          | Du waste discharge river   | 8.1 | 104.72            | 30.97              | 1.47    |
| b6          | Bei waste discharge river  | 7.6 | 75.85             | 27.65              | 3.25    |
| b7          | Offshore sea               | 6.9 | 26.57             | 25.96              | 1.78    |
| b8          | Yinghe reservoir           | 7.0 | 41.97             | 16.49              | 1.35    |
| b9          | Mouth of Yunding new river | 8.1 | 58.14             | 37.01              | 1.53    |
| b10         | Beitang waste discharging river | 6.9 | 75.68             | 59.14              | 1.94    |

### Table 2: Concentrations of PAHs in the sampled sediments (μg/kg).

| PAH                  | b1   | b2   | b3   | b4   | b5   | b6   | b7   | b8   | b9   | b10  |
|----------------------|------|------|------|------|------|------|------|------|------|------|
| Naphthalene          | 771.09 | 657.56 | 582.06 | 126.68 | 90.56 | 145.79 | 75.69 | 85.16 | 76.97 | 226.21 |
| Acenaphthylene       | 41.77  | 15.16 | 24.72 | 10.66 | 4.19  | 7.83  | 4.08 | 42.83 | 3.91  | 13.99 |
| Acenaphthene         | 344.24 | 77.51 | 51.62 | 51.00 | 22.86 | 33.48 | 33.98 | 21.09 | 27.04 | 358.71 |
| Fluorene             | 245.17 | 79.40 | 129.82 | 208.65 | 27.88 | 81.69 | 40.79 | 37.79 | 28.04 | 255.55 |
| Phenanthrene         | 289.16 | 103.49 | 312.03 | 54.23 | 37.76 | 215.01 | 56.15 | 83.57 | 31.26 | 134.74 |
| Anthracene           | 52.72  | 15.59 | 60.03 | 10.54 | 4.86  | 41.23 | 9.69 | 29.46 | 6.08  | 27.11 |
| Fluoranthene         | 6.72   | 68.19 | 256.16 | 93.34 | 32.67 | 275.56 | 55.69 | 32.44 | 2.41  | 27.11 |
| Pyrene               | 239.06 | 55.64 | 272.13 | 77.43 | 24.52 | 220.19 | 46.16 | 194.15 | 27.22 | 78.34 |
| Benzo(a)anthracene   | 71.86  | 7.18  | 42.41 | 15.08 | 3.66  | 75.73 | 3.35 | 34.37 | 3.04  | 30.47 |
| Chrysene             | 77.46  | 17.62 | 117.84 | 13.90 | 7.22  | 171.90 | 12.73 | 84.18 | 7.56  | 36.50 |
| Benzo(b)fluoranthene | 194.85 | 16.85 | 83.22 | 25.53 | 15.83 | 106.85 | 21.32 | 84.79 | 13.95 | 86.15 |
| Benzo(k)fluoranthene | 42.01  | 0.02  | 52.08 | 16.11 | 9.91  | 66.88 | 13.34 | 53.07 | 8.73  | 20.19 |
| Benzo(a)pyrene       | 90.40  | 6.72  | 105.78 | 6.29  | 3.82  | 136.30 | 7.44 | 30.10 | 4.27  | 36.76 |
| Indeno(1,2,3-cd)pyrene| 78.22  | 2.23  | 59.36 | 6.37  | 2.65  | 130.04 | 6.30 | 17.85 | 2.89  | 38.89 |
| Dibenzo(a,h)anthracene| 22.09  | 0.51  | 14.32 | 1.33  | 0.54  | 5.64  | 1.21 | 2.98  | 0.65  | 9.57  |
| Benzo(ghi)perylene   | 89.83  | 2.71  | 52.93 | 4.84  | 2.72  | 94.66 | 4.97 | 11.19 | 0.01  | 1.28  |

\[ \sum_{\text{PAH}}^{16} \]

\[ 2656.64 \]

\[ 1126.39 \]

\[ 2216.51 \]

\[ 721.98 \]

\[ 291.63 \]

\[ 1808.79 \]

\[ 392.88 \]

\[ 1139.40 \]

\[ 274.06 \]

\[ 1356.86 \]

It is generally believed that PAHs of different sources have different structures and compositions, and therefore characteristic ratios of some PAHs could be used to characterize the sources [29–31]. In this study, four ratios, that is, and fluoranthene to pyrene (FLA/PYR), pyrene to benzo(a)pyrene (PYR/BaP), indeno(1,2,3-cd)pyrene to the sum of indeno(1,2,3-cd)pyrene and benzo(ghi)perylene (IcdP/(IcdP+BghiP)), and fluoranthene to the sum of fluoranthene and pyrene (FLA/(FLA+PYR)), were calculated to analyze the sources of PAHs in Tianjin coastal area. The results showed that combustion of fossil fuels (such as coal and gasoline) was the major source of PAHs in the sediments, and in a few places there were inputs of petroleum products. These observations were in agreement with the results obtained in other studies [32, 33].

It was found that TOC was positively correlated with \[ \sum_{\text{PAH}}^{16} \] (\( R = 0.900, P = 0.000 \)) in this study, as shown in Figure 2. This further demonstrated that the sources of PAHs were nonpoint. PAHs from the sources might enter the water bodies through atmospheric sedimentation, surface runoff, and so forth and accumulated in the sediments owing to the adsorption by the organic matters in the sediments. In addition, the sediments (particularly the polluted ones) were generally in anaerobic conditions under which the biodegradation of PAHs were slow. Therefore, there was good correlation between TOC and \[ \sum_{\text{PAH}}^{16} \].

#### 3.2.2. Concentration Levels of OCPs, PCBs, and PBDEs in the Sediments.

The concentrations of OCPs, PCBs, and PBDEs were generally low in the studied sediments except in the Dagu waste discharging river. Figures 3, 4, and 5 show the total concentrations of 24 OCPs (\[ \Sigma_{\text{OCP}}^{24} \]), 35 PCBs (\[ \Sigma_{\text{PCB}}^{35} \]), and 14 PBDEs (\[ \Sigma_{\text{PBDE}}^{14} \]) in the sediments of various locations. The OCPs data for b1 and b3 were missing due to sample damages. In the Dagu waste discharging river (b2), high concentrations for the halogenated POPs were observed, where hexachlorobenzene (HCB), \( \alpha \)-benzene hexachloride (\( \alpha \)-BHC) and \( \beta \)-benzene hexachloride (\( \beta \)-BHC) were the major OCPs with the concentrations being 1994.99 μg/kg, respectively.
337.27 µg/kg and 557.26 µg/kg, respectively. PCB-209, PCB-87, and PCB-70 were the major PCBs with the concentrations being 26.77 µg/kg, 32.30 µg/kg and 16.39 µg/kg, respectively, and BDE-209 was the major PBDE with the concentration being 12.97 µg/kg. The Dagu waste discharging river is a major waste discharging river in Tianjin, with daily discharge amount being over 800 thousand tons [34]. There are several chemical plants such as Tianjin chemical plant, Tianjin Dagu chemical plant located along the banks of the Dagu waste discharging river. These plants historically produced substantial OCPs like HCB, BHCs, and heptachlor epoxide. Wastes from the chemical plants may also contain PCBs and PBDEs that are used in the industry. In the mouth of the Dagu waste discharging river, the concentrations of PCBs and PBDEs were decreased, which were probably due to dilution by the sea water. Tables S1 to S3 in the supplemental materials show the individual concentrations of the OCPs, PCBs, and PBDEs in the studied sediments.

There was no significant correlation between TOC values and the concentrations of halogenated POPs in the sediment (R < 0.400, P > 0.300), indicating the sources of the halogenated POPs were of point. Compared to other coastal areas in China, the levels of OCPs in Tianjin were relatively high, exceeding the OCPs in the Pearl River Delta and its adjacent coastal areas of the South Sea, Yangtze River, the offshore of East Sea, and so forth [35–38]. The levels of PCBs and PBDEs were lower than most of the coastal areas in China [39–41].

3.3. Ecological Risks of the POPs in the Sediments. The ecological risks of PAHs, OCPs, PCBs, and PBDEs in the studied sediments were assessed on basis of the toxicity data of benthic organisms. Relatively high risks were observed for PAHs, particularly LMW-PAHs. At each sampling location, at least one LMW-PAH had concentration over its TEC, and in several locations (b1, b2, b3, and b10), the concentrations of naphthalene and/oracenaphthene exceeded their PEC values, indicating adverse ecological effects to benthic organisms. Tables 3 and 4, respectively, show the TEC-HQ and PEC-HQ of PAHs in the studied sediments.

At six locations (b2, b5, b7, b8, b9, and b10), the concentrations of heptachlor epoxide exceeded the TEC value. In the Dagu waste discharging river (b2), the concentrations
of heptachlor epoxide and γ-BHC exceeded their PEC values (the PEC-HQ of γ-BHC was up to 7.49). In the other locations, there were rare risks for OCPs. For PCBs, slight risk was observed in the Dagu waste discharging river (b2) where the ΣPCB35 exceeded the TEC but was far less than the PEC; no risk was observed in the other locations. As for PBDEs, all the HQ values were far less than 1 indicating no risk.

4. Conclusions

In the 10 sediments sampled from different surface water bodies in Tianjin coastal area, the 16 priority PAHs were all detected and the mean value of ΣPAH16 was 1198.51 μg/kg. The major source of PAHs was the combustion of fossil fuels, with inputs of petroleum products in a few places. The concentration levels of OCPs, PCBs, and PBDEs were generally low except in the Dagu waste discharging river where there were many chemical plants along the banks. The TOC in the sediments had good correlation with ΣPAH16 but not with the halogenated POPs.

There were relatively high risks for PAHs in the studied sediments. At each sampling location, at least one LMW-PAH had concentration over its TEC, and in several locations, the concentrations of naphthalene and/or acenaphthene exceeded their PEC values. In the Dagu waste discharging river, relatively high risk for OCPs and slight risk for PCBs
were observed. In the other locations, the risks for the halogenated POPs were rare.

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Conflict of Interests

The authors declare they have no conflict of interests.

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