The levels and distribution of organochlorine pesticides (OCPs) in sediments from the Haihe River, China

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Abstract

The levels and distribution patterns of the selected organochlorine pesticides (OCPs = *p,p'*-DDT, *o,p'*-DDT, *p,p'*-DDE, *p,p'*-DDD, α -, β -, γ - and δ -HCH) in surficial sediments from the Haihe River and Dagu Drainage River of Tianjin were investigated by means of gas chromatography coupled with micro-electronic capture detector (GC- μ ECD). Concentrations of OCPs in the sediments from the Haihe River ranged from 1.88 to 18.76 ng g⁻¹ (mean 7.33 ng g⁻¹) for $\sum$ HCH, 0.32–80.18 ng g⁻¹ (mean 15.94 ng g⁻¹) for $\sum$ DDT. Compared with the Haihe River, the Dagu Drainage River was much more contaminated by HCHs and DDTs, wherein $\sum$ HCH ranged from 33.24 to 141.03 ng g⁻¹ (mean 87.74 ng g⁻¹) and $\sum$ DDT ranged from 3.60 to 83.49 ng g⁻¹ with a mean value of 35.52 ng g⁻¹. The concentration distribution of $\sum$ DDT and $\sum$ HCH was different indicated their different contamination sources. Composition analyses indicated that a recent usage or discharge of HCH and DDT into the Dagu Drainage River.

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1. Introduction

Organochlorine pesticides (OCPs) have been of great concern because of the large production and usage, their deleterious effect on non-target organism, ubiquity, bioaccumulation and persistence in the environment (Willet et al., 1998). Technical HCH and DDT were the most extensively used pesticides in China, resulting in widespread contamination in various environmental compartments (Wu et al., 1999; Zhou et al., 2001). These

chemicals can enter aquatic environment through several ways such as effluents release, agricultural runoff, atmospheric deposition and other means. Due to the low water solubility, OCPs have a strong affinity for suspended particulates and subsequently settle down to sediments. One of the major sinks is thought to be the river and marine sediments (Doong et al., 2002). Therefore, the investigation of OCPs in sediments can provide a valuable record of contamination in aquatic environment.

Tianjin is one of the most severely contaminated areas by HCHs and DDTs in China because of its long-term usage and wastewater discharge from local industries. Although the production of HCH and DDT was officially banned in China since 1983, the

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actual application lasted for four decades from 1953 to 1993 in Tianjin. In addition, the production of technical HCH and lindane (pure γ -HCH) was not stopped until 2000, while the DDT production may continue. Recent studies indicated that the levels of OCPs in agricultural soil of Tianjin region remained high (Gong et al., 2004).

The Haihe River, the largest water system in north China, flows through Tianjin city and empties into the Bohai Sea. With the rapid economic growth, industrialization, and urbanization, accompanied by inadequate infrastructure investment and management capacity, water shortage and water pollution are becoming extremely concernful. Water shortage has become a bottleneck to the further development of the economy and crop production. Serious pollution and environmental degradation have aggravated the water scarcity by reducing the availability of usable fresh water. The Dagou Drainage River is a very important channel to discharge various wastewater from domestic and industries in Tianjin and finally empties into the Bohai Sea. However, to our knowledge, few data are available for the contamination of OCPs in these rivers. This paper presents the levels and distributions of OCPs in the surficial sediments from the Haihe River and the Dagou Drainage River and their contamination profiles and possible sources of OCPs were also discussed.

2. Materials and methods

2.1. Sample collection

A total of 40 sediment samples in 13 sites along the Haihe River and Dagou Drainage River were collected

during four sampling campaigns on April 8–9 (H1), July 12–13 (H2), September 6–7 (H3) and November 15–16 (H4), 2003, respectively. Fig. 1 shows the study area and the sampling locations. The sediments of S1 and S2 were collected from Beiyun River and Ziya River, which converged into the Haihe River before flowing through Tianjin city. S3, lies in urban districts, is an important crossing point of several branches of the Haihe River. S4 is the locus where Haihe River flowing out from Tianjin city. S6 and S9 are located at the two brakes of the Haihe River. S10 is the estuary of the Haihe River. S7 is the location just before the Machangjian River impouring into the Haihe River, while sediments S8 were collected from the Machangjian River. Dagou Drainage River is an important drainage river in Tianjin city and samples of S11, S12 and S13 were collected.

The surficial sediments (top 0–3 cm) were collected with a stainless steel grab and then placed into polytetrafluoroethylene (PTFE) bags. All the samples were immediately transferred to the laboratory and kept at -20°C in the refrigerator. The samples were freeze-dried, homogenized and passed through a $63\text{-}\mu\text{m}$ sieve. All the equipments used for sample collection, transportation, and preparation, were free from organochlorines contamination.

2.2. Instrumental

Quantitative analyses of OCPs in sediments were accomplished by an Agilent 6890 A gas chromatography (GC) equipped with a ^{63}Ni electron capture detector (μECD). A HP-1 fused silica capillary column (50 m length $\times$ 0.32 mm i.d. $\times$ 0.25 μm film thickness) was used

Fig. 1. Map of the study area and sampling sites.

for separating the OCPs. One microliter of each sample was injected in a splitless model. Oven temperature increased from 80 °C (held for 1 min) to 180 °C (held for 2 min) at the rate of 50 °C/min, followed to 230 °C (held for 2 min) at the rate of 5 °C, and then programmed to 270 °C at 10 °C/min, held for 15 min. The temperatures of injector and detector were 220 °C and 300 °C, respectively. High pure nitrogen was used as both the carrier gas and make-up gas with a flow rate of 1.0 and 59 ml/min, respectively. Peak identification was conducted by the accurate retention time of each standard ($\pm 1\%$) and also confirmed by GC–MS.

2.3. Standards and reagents

The standard solution of composite OCPs including α -, β -, γ -, δ -HCH and p,p' -DDT, o,p' -DDT, p,p' -DDE, p,p' -DDD was purchased from National Research Center for Certified Reference Materials of China. In the meanwhile, the corresponding individual standard of OCPs was also prepared for qualitative purpose. The internal standard 2,2', 3,3' 4,4' 5,5' 6,6'-decachlorobiphenyl was purchased from Accustandard, Inc. USA. Florisil (100–200 mesh, Beijing Yizhong Chemical Reagents Factory, China) was activated in the oven at 160 °C for 8 h. The organic reagents including hexane, dichloromethane were redistilled before use.

2.4. Analytical procedure

Total organic carbon contents were determined using Apollo 9000 TOC analyzer (Tekmar-Dohrmann Co., USA). The analytical procedure of OCPs residues in sediments was a modification of the method described by Yang et al. (2004). Briefly, 5 g of freeze-dried sample was weighed and a suitable amount of internal standard was added then extracted for 40 min with sonication method using 60 ml of hexane–dichloromethane (1:1 v/v), stood overnight (about 10 h) and was again sonicated for 20 min. The extract was decanted, another 10 ml of hexane was used to extract for 20 min. The two extracts were combined and the activated Cu was added to remove element sulfate. The extracts were concentrated to about 1–2 ml by a rotary evaporator, and further purified with a glass column (12 mm i.d.) loaded with 10 g of activated Florisil. The elution was subsequently carried out using 10 ml of hexane, followed by 30 ml of hexane containing 25% dichloromethane (v/v). The effluents were concentrated to a final volume of 0.5 ml for GC analysis.

The residue levels of OCPs were quantitatively determined by the internal standard method using peak area. The correlation coefficients (r) of calibration curves of HCHs and DDTs were all higher than 0.998. The detection limits (LODs) of the method were described as three times signal versus noise value (S/N). For every set of

eight samples, a procedural blank and a spiked sample with standards were run to check for the interference and cross-contamination. The LODs ranged from 0.04 ng g⁻¹dw for δ -HCH and p,p' -DDE to 0.19 ng g⁻¹dw for α -HCH. The spiked recoveries of OCPs using 10 ng of composite standards were in the range of 87–103% and the relative standard deviation (RSD) values ranged from 2% to 14%.

3. Results and discussion

3.1. Contamination status of OCPs

The contents of total organic carbon (TOC) and the concentrations of HCHs (α -, β -, γ -, and δ -HCH) and DDTs (p,p' -DDT, o,p' -DDT, p,p' -DDE, p,p' -DDD) of the samples were all shown in Table 1. The detection rates of both of $\sum$ HCH and $\sum$ DDT in the sediments are up to 100%, which indicates wide occurrence of these compounds in the Haihe River and Dagu Drainage River. Significant differences of $\sum$ HCH and $\sum$ DDT in four batches of samples (H1, H2, H3 and H4) were tested using One-way ANOVA method. The results showed that there were no significant differences among these four batches of samples ($P = 0.05$). The status of OCPs contamination in sediments in this study was compared with those in other riverine and coastal regions (Table 2). Concentrations of OCPs in the sediments from the Haihe River ranged from 1.88 to 18.76 ng g⁻¹ (mean 7.33 ng g⁻¹) for $\sum$ HCH, 0.32–80.18 ng g⁻¹ (mean 15.94 ng g⁻¹) for $\sum$ DDT. Amongst the Chinese large rivers, the levels of $\sum$ HCH and $\sum$ DDT in sediments from Haihe River ranked the highest, with which only the residue levels in sediments from Mingjiang can be comparable (Hu et al., 1996; Wu et al., 1999; Zhang et al., 1996; Hong et al., 1999; Doong et al., 2002). When compared with other regions over the world, mean concentration of $\sum$ HCH in the sediments of Haihe River is less than those of the Parramata River, Australia and the Mataniko River, Solomon but higher than others. For the concentrations of $\sum$ DDT, some regions such as Indian Mandovi River Estuary, Australian Parramata River, Solomon Mataniko River showed the relatively higher concentrations (Iwata et al., 1994; Lee et al., 2001). However, compared with the Haihe River, the Dagu Drainage River was much more contaminated by HCHs and DDTs, wherein $\sum$ HCH ranged from 33.24 to 141.03 ng g⁻¹ (mean 87.74 ng g⁻¹), while $\sum$ DDT ranged from 3.60 to 83.49 ng g⁻¹ with a mean value of 35.52 ng g⁻¹.

3.2. Spatial distribution of OCPs

The kinetic behavior of hydrophobic organic pollutants is much influenced by organic carbon contents in

Table 1

Concentrations of OCPs (ng g⁻¹ dw) and the TOC contents in the sediments from the Haihe River and Dagu Drainage River

Sampling	TOC (mg g ⁻¹)	α -HCH	β -HCH	γ -HCH	δ -HCH	Σ HCH	<i>p,p'</i> -DDE	<i>p,p'</i> -DDD	<i>o,p'</i> -DDT	<i>p,p'</i> -DDT	Σ DDT
<i>April 8–9 (H1)</i>											
S1	9.95	2.88	1.77	0.30	1.00	5.95	5.56	1.66	0.31	1.41	8.94
S2	12.08	1.02	0.88	<0.05	0.31	2.21	0.19	0.15	<0.09	0.14	0.48
S4	34.40	1.32	3.21	1.05	0.48	6.06	1.04	3.07	0.73	2.48	7.31
S5	9.18	2.38	1.53	0.18	0.50	4.59	0.81	2.68	0.28	0.97	4.75
S6	18.33	3.23	4.31	0.91	1.04	5.49	0.12	0.20	<0.09	<0.12	0.32
S7	11.02	1.77	5.93	0.21	0.54	8.45	8.03	2.62	0.69	1.92	13.26
S8	20.20	1.82	6.20	1.04	1.98	11.04	0.61	0.38	0.69	<0.12	1.68
S10	13.82	3.53	3.32	1.69	1.11	9.65	1.78	3.91	0.38	1.28	7.35
<i>July 12–13 (H2)</i>											
S1	8.20	2.29	1.40	0.72	0.97	5.38	59.24	11.04	0.72	4.43	75.43
S2	9.08	1.59	2.45	1.48	0.89	6.41	0.68	0.26	<0.09	0.34	1.38
S4	12.40	1.41	1.56	1.68	0.68	5.33	2.53	7.35	2.29	5.47	17.64
S6	15.65	0.67	1.50	0.09	0.39	2.65	2.92	1.86	<0.09	<0.12	4.78
S8	10.54	1.06	7.00	1.79	1.74	11.59	1.35	1.60	<0.09	0.70	3.65
S9	6.41	5.55	3.00	1.52	1.29	11.36	<0.04	1.57	<0.09	<0.12	1.57
S10	4.15	2.77	1.10	0.41	0.48	4.76	0.44	4.54	<0.09	0.82	5.80
S11	58.63	6.85	18.46	5.36	4.00	34.67	18.95	39.64	3.36	1.53	63.48
S12	33.00	55.82	19.46	10.99	38.27	124.54	<0.04	0.67	0.74	5.03	6.43
S13	5.47	81.20	6.23	26.85	9.84	124.12	0.37	0.48	0.86	3.08	4.79
<i>September 6–7 (H3)</i>											
S1	5.44	1.32	0.21	<0.05	<0.04	1.53	20.53	4.15	0.20	3.23	28.10
S2	6.52	0.67	1.00	0.07	0.22	1.96	1.35	0.54	<0.09	0.80	2.69
S3	22.40	5.66	7.55	3.46	2.36	19.03	37.56	31.41	1.27	9.94	80.18
S4	7.30	3.34	1.13	0.96	1.10	6.53	5.14	2.57	<0.09	0.89	8.61
S6	33.16	1.01	2.38	0.66	1.45	5.50	2.97	2.69	<0.09	<0.12	5.66
S8	8.54	1.07	7.07	1.52	2.06	11.72	2.05	1.00	<0.09	1.30	4.36
S9	4.18	0.87	0.33	0.86	1.41	3.47	0.49	3.72	0.48	2.21	6.91
S10	4.66	0.90	0.40	0.74	0.63	2.67	1.14	6.45	0.87	3.44	11.89
S11	80.20	8.68	20.43	2.64	2.50	34.25	17.56	58.36	3.69	3.88	83.49
S12	45.66	51.47	20.72	13.47	16.72	102.38	9.26	7.18	0.92	3.25	20.61
S13	4.58	78.28	24.87	23.05	14.83	141.03	1.17	0.57	0.42	1.44	3.60
<i>November 15–16 (H4)</i>											
S1	37.02	2.30	1.44	0.23	0.33	5.30	25.19	4.03	0.59	2.72	32.54
S2	26.00	0.57	0.48	0.37	0.46	1.88	1.48	0.36	0.84	1.42	4.10
S3	53.50	4.88	7.95	3.87	2.06	18.76	31.70	18.01	0.97	11.03	61.71
S4	13.03	1.68	1.21	0.75	0.99	4.63	4.45	1.65	0.30	1.33	7.73
S6	44.69	1.79	2.60	0.75	0.51	5.65	4.25	2.85	0.31	0.54	7.85
S8	20.46	2.10	2.48	1.10	1.00	6.68	4.05	2.13	0.27	0.90	7.34
S9	8.42	1.44	0.84	1.35	1.08	4.71	0.58	3.14	0.31	1.26	5.29
S10	10.35	1.81	0.89	2.04	1.19	5.93	1.26	3.45	0.64	1.10	6.45
S11	19.07	7.71	20.65	2.64	2.24	33.24	12.36	48.35	2.36	5.62	68.69
S12	68.78	36.16	18.55	13.24	19.32	87.27	8.30	25.33	2.31	10.59	46.54
S13	10.01	61.67	13.79	15.70	17.03	108.19	<0.04	1.48	3.83	16.72	22.03

sediments and soils (Karickhoff, 1981). Our study shows that the concentrations of Σ DDT, but not Σ HCH, in sediments from Haihe River are correlated well with TOC contents ($r = 0.553$, $p < 0.05$), which is possibly due to the less lipophilic and more volatile nature of HCHs relative to the DDTs. This relationship was also confirmed by Lee et al. (2001). The distributions of Σ HCH and Σ DDT in sediments along the Haihe River

and Dagu Drainage River were shown in Figs. 2 and 3, respectively.

Fig. 2 shows that in general the concentrations of Σ HCH in sediments for samples S1 and S2 collected from Beiyun River and Ziya River and for samples S3–S10 from the Haihe River displayed uniform low distribution. Because, the sediment sample in the Nanyun River was not available, the contamination status of

Table 2

Concentrations of $\sum$ HCH and $\sum$ DDT (ng g^{-1} dw) in surface sediments from large Chinese rivers and other world rivers, estuary and bays

Locations	Sampling year	$\sum$ HCH	$\sum$ DDT	References
Haihe River	2003	1.88–18.76 (7.33)	0.32–80.18 (15.94)	This study
Daliaohe	1989	0.50–0.80 (0.65)	0–0.40 (0.20)	Wu et al. (1999)
Luanhe	1991	0.20	ND	Wu et al. (1999)
Changjiang	1988	0.40–0.70 (0.60)	0.10–0.20 (0.20)	Wu et al. (1999)
Huanghe	1986	1.00–5.00 (3.00)	ND	Wu et al. (1999)
Huangpujiang	1988	2.90	1.30	Wu et al. (1999)
Qiantangjiang	1984	0.70	0.10	Wu et al. (1999)
Jiulongjiang	1993	0.29–0.69 (0.49)	4.10–6.10 (5.20)	Zhang et al. (1996)
Minjiang	1986	4.20–9.40 (6.80)	6.90–13.10 (10.00)	Hu et al. (1996)
Pearl River	1996–1997	0.28–1.23 (0.68)	1.36–8.99 (2.84)	Hong et al. (1999)
Wu-shi River, Taiwan	1997–1998	0.99–14.5 (3.78)	0.53–11.4 (2.51)	Doong et al. (2002)
Osaka Bay, Japan	1990	1.3	2.5	Iwata et al. (1994)
Mandovi River Estuary, India	1989	3.8	73	Iwata et al. (1994)
Parramata River, Australia	1990	7.7	26	Iwata et al. (1994)
Swan River, Australia	1990	1.2	2.1	Iwata et al. (1994)
Mataniko River, Solomon Islands	1990	140	750	Iwata et al. (1994)
Kyeonggi Bay, Korea	1995	<0.19–1.2	<0.046–32	Lee et al. (2001)
Casco Bay, USA	1991	<0.25–0.48	<0.25–20	Lee et al. (2001)

ND, not detected; A–B (C) means that the range of concentration is A–B ng g^{-1} , while the mean value is C ng g^{-1} .

Fig. 2. The distributions of $\sum$ HCH in sediments from the Haihe River and Dagu Drainage River.

the Nanyun River by HCH cannot be determined. However, after the three streams converged into the Haihe River in the urban area of Tianjin, a higher TOC content and HCH was found, indicating possibly relatively high historical input of $\sum$ HCH from the Nanyun River. In addition, sediments from the Machangjian River showed the relatively higher content of $\sum$ HCH. In case of Dagu Drainage River, very high concentrations of $\sum$ HCH were measured, especially in sediments of S12 and S13, indicating important sources of HCH in this re-

gion. In fact, there were many chemical manufactures, including HCH and DDT producers, operated near the sites of S12 and S13.

A different distribution pattern has been found for DDT. Obviously, Beiyun River was seriously contaminated by DDT compounds, while Ziya River was much less polluted. Similarly, although the pollution of DDT in Nanyun River was unclear, the Beiyun River is probably one of the major contributors of DDT in the urban sites of Haihe River due to the high concentration of

Fig. 3. The distributions of $\sum$ DDT in sediments from the Haihe River and Dagu Drainage River.

$\sum$ DDT in the samples from the converged site (S3). In the other samples from the Haihe River showed the medium contamination level of $\sum$ DDT. The Dagu Drainage River also showed the high level of $\sum$ DDT.

3.3. Compositions analyses

Composition differences of HCH isomers or DDT congeners in the environment could indicate different contamination sources (Iwata et al., 1995; Doong et al., 2002). Technical HCH has been used as a broad spectrum pesticide for agricultural purpose, which has been officially banned since 1983 in China. The typical technical HCH generally contains 55–80% of α -HCH, 5–14% of β -HCH, 8–15% of γ -HCH and 2–16% of δ -HCH, respectively (Lee et al., 2001). The physico-chemical properties of these HCH isomers are different. β -HCH has the lowest water solubility and vapor pressure, which is the most stable and relatively resistant to microbial degradation (Ramesh et al., 1991). Also it should be noted that α -HCH can be converted to β -

HCH in the environment (Wu et al., 1997; Walker, 1999). Therefore, the predominant of α -isomer in some environmental samples reflects the recent use of technical HCH (Kannan et al., 1995). Many studies have reported that β -HCH was dominant in sediments from the river or estuary environment after long-term migration and transformation (Wu et al., 1999; Lee et al., 2001; Doong et al., 2002). However, regarding the composition of HCH isomers measured in this study, a high percentage of α isomers was recorded (Fig. 4). The average compositions of HCH isomers measured in the samples are α : 59.0%, β : 32.7%, γ : 5.7% and δ : 2.5%.

Technical DDT generally contains 75% p,p' -DDT, 15% o,p' -DDT, 5% p,p' -DDE, and <5% others. DDT can be biodegraded to DDE under aerobic condition and to DDD under anaerobic condition (Kalantzi et al., 2001). Comparing the concentrations of p,p' -DDT and its metabolites, it can be inferred whether DDT's input are recent or not (Phuong et al., 1998). The ratio of $(DDE + DDD)/\sum DDT > 0.5$ can be thought to be subjected to a long-term weathering (Hites

Fig. 4. Composition of HCHs in sediments from the Haihe River and Dagu Drainage River.

Fig. 5. Composition of DDTs in sediments from the Haihe River and Dagu Drainage River.

and Day, 1992; Hong et al., 1999). In most present samples, DDE and DDD occupied the predominant percentage (Fig. 5). Here the mean ratio of (DDE + DDD)/ $\sum$ DDT in the sediments from the Haihe River was 0.76, which indicated that the degradation occurred significantly. For the Dagu Drainage River, in sediment of S11 and S12, the breakdown products of DDE and DDD occupied predominant percent of DDTs. However, the *p,p'*-DDT in the sediment of S13 occupied about 70%. The high levels of $\sum$ DDT and high percentage of DDT indicated the recent input of DDT into the downstream of the Dagu Drainage River.

4. Conclusion

This work investigated the contamination status of HCH and DDT in surface sediments of the Haihe River and Dagu Drainage River. Wide occurrence and high residue levels of these two OCPs have been found in the study areas, especially for the Dagu Drainage River. The distribution of $\sum$ DDT and $\sum$ HCH was different, indicating their different contamination sources. The Ziya River, one of the main branches of the Haihe River, was seriously contaminated by DDT. Composition analyses and high residue levels of HCH and DDT indicated that a recent input of these two OCPs into the Dagu Drainage River.

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