

Residues and Distributions of Organochlorine Pesticides in China's Weihe River

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Abstract

To examine the residues and distributions of hexachlorocyclohexanes (HCHs), dichlorodiphenyltrichloroethane (DDT), and other organochlorine pesticides (OCPs) in the Weihe River basin of northwest China, a gas chromatography-mass spectrometer (GC-MS) was employed to analyze the samples collected from surface water, suspended solids, and sediments. Results showed that total concentrations of HCHs, DDT, and other OCPs in surface water were in the range of 2.41-178.18, 0.94-116.83, and 3.64-37.17 ng/L, respectively; in suspended solids they were 51.76-241.23, 2.82-12.23, and 11.35-37.67 ng/L, respectively; and in sediments they were 74.13-517.49, 1.20-370.98, and 7.94-110.13 ng/g dry weight, respectively. The α -HCH/ γ -HCH ratio indicated that historical usage of technical mixtures of HCHs was the main source of HCHs. The DDT ratio indicated that DDT at most sites came from older uses of technical DDT. Compared with some guideline values of OCPs in surface water, the concentrations of HCHs and DDT were at safe levels. Meanwhile, the Weihe River sediments have high ecological risk pesticides.

Keywords: organochlorine pesticides, water, suspended solids, sediments, Weihe River

Introduction

Organochlorine pesticides (OCPs) in the environment are not only recognized as priority pollutants, but also typical of persistent pollutants (POPs). OCPs such as hexachlorocyclohexanes (HCHs) and dichlorodiphenyltrichloroethane (DDT) have caused global attention due to their environmental persistence, high toxicity, bioaccumulation, and biomagnification in the environment [1, 2]. China has been one of the countries with high production and consumption of HCHs and DDT [3]. Large amounts of OCPs were used in past

decades to obtain high yield to sustain overpopulation in China. HCHs and DDT were the most widely used pesticides in China during the 1950s to 1980s due to their low cost and high insecticidal efficacy. The productions of HCHs and DDT in China were 4.9 and 0.4 million tons, respectively, accounting for 33 percent and 20 percent of total worldwide production [1]. Even after the ban of technical HCH and DDT in 1983, approximately 11,400 tons of lindane (almost γ -HCH) reportedly were still being produced [4]. And DDT has been continuously produced for about 20 years because of exports to other countries for control of malaria and production of dicofol [5]. OCPs are hard to be degraded, and they can still be detected in various environmental media such as soil, water, suspended solids, sediments, atmosphere, and organisms.

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In the water environment, trace amounts of OCPs even cause adverse biological effects on ecosystems, and the contamination can even be hazardous to humans through aquatic food [6]. Therefore, investigating the residues and distributions of OCPs in water is important because of its environmental significance.

Dated sediments are natural archives that can be used to provide comprehensive estimations of historical OCP records and reconstruct their chronology in aquatic environments [7]. Information regarding the historical accumulation processes of OCPs in lake sediments is of importance for understanding how OCPs in river systems threaten the environment and human health. Several studies have investigated the historical records of OCPs in sediment cores from marine and estuary environments [8, 9]; however, limited data are available regarding the sedimentary records of OCPs in inland aquatic ecosystems – particularly in water, suspended solids, and sediments from rivers in arid and semiarid regions. Studies about OCP contamination in rivers in China [10-13] show that almost all of these rivers are located in humid and semi-humid regions rather than in arid and semiarid regions. However, as a typical river, the Weihe belongs to an arid and semiarid region in northwest China and is the biggest tributary of the Yellow River. In addition, it is also a source of drinking water and industrial water with a lot of human activities along the river. Reservoir construction downstream causes deposition of sediments, and the river's velocity becomes slow, which accelerates sediment deposition.

The aims of this research are to survey the residual levels and pollution status, discussing the distributions of OCPs in water, suspended solids, and river sediments. In addition, we compare the temporal trend of the pollution of OCPs with previous measurements to assess ecological risks in the basin. Furthermore, we discuss the contamination profiles and possible sources of HCHs and DDT for the purpose of future strategic environmental management.

Material and Methods

Instruments and Reagents

A gas chromatography-mass spectrometer (USA Agilent GC-MS, 7890A-5975C) and OCPs mixed solution with 18 components (including α -, β -, γ -, δ -HCH, p,p'-DDT, o,p'-DDT, p,p'-DDE, o,p'-DDE, p,p'-DDD, o,p'-DDD, tecnazene, hexachlorobenzene, heptachlor, aldrin, heptachlor epoxide (isomer A), heptachlor epoxide (isomer B), tetrachlorvinphos, and endrin, with each component concentration 10 $\mu\text{g/mL}$) were purchased from Sigma Company (USA). All solvents used for sample pretreatment and analysis (hexane, dichloromethane, methanol, and acetone) were purchased from Oceanpak (Sweden). A Florisil SPE cartridge was purchased from CNM Technology (Germany). Anhydrous sodium sulfate is the analytical reagent. Neutral silica gel (60-100 mesh) was kept in a sealed desiccator before use.

Sample Collection

Fig. 1 shows the sampling sites in the study. We selected 37 sampling sites along the Weihe and its tributaries in order to cover the whole aquatic system in this area. The sediment samples (0-5 cm deep) were collected with a stainless steel static gravity corer. The top 1 cm surface layer was carefully removed with a stainless steel spoon and stored in aluminium containers. At the same time, water samples were collected at the sediment sampling sites using a cleaned brown glass bottle. The suspended solid samples were obtained by filtering the water samples using a peristaltic pump and a glass fiber membrane with a diameter of 47 mm. All the equipment used for sample collection, transportation, and preparation were free from organochlorine contamination. The basic test indexes include COD (7.14-1056.35 mg/L), TDS (154-1370 mg/L), and pH (7.46-9.96).

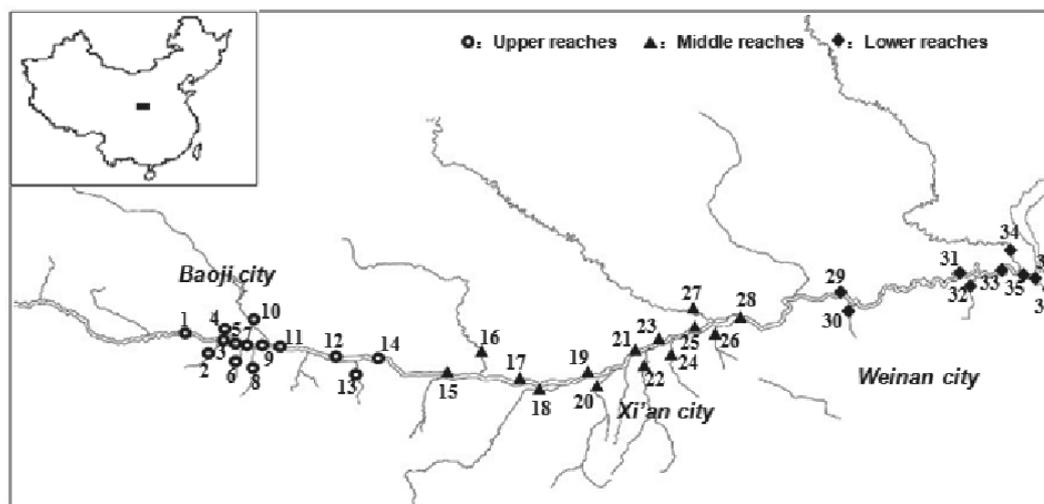


Fig. 1 Map of Weihe River and sampling sites.

Sample Pretreatment

Water samples (1.0 L) were passed through the glass fiber membrane (47 mm) at a flow rate of 5 mL/min using a vacuum. All water samples were extracted and purified using a solid phase extraction (SPE) system. The Florisil SPE cartridges were first conditioned with 5 mL of hexane, then 5 mL of methanol followed by 5 mL of ultrapure water. Following extraction, the cartridges were eluted with 10 mL of ethylacetate. Then the extracts were dehydrated with anhydrous Na_2SO_4 and concentrated to 1 mL by rotary evaporator prior to GC-MS analysis.

All sediment samples were freeze-dried, homogenized, and passed through a 150- μm sieve. Hexane-acetone (1:2, v/v) was added into each sample (2 g) and then the samples were extracted by ultrasonic extraction for 10 min before eluting by 10 mL hexane-dichloromethane (1:1, v/v). The extracts were concentrated to 1 mL by rotary evaporator prior to GC-MS analysis.

The suspended solids samples were air-dried for 48 h, and then eluted with acetone-hexane (1:1, v/v). The extracts were subjected to a silica gel column to remove impurities and isolates. The purification methods were the same as the methods for sediment samples indicated above.

Sample Analysis

Chromatographic Condition

Calibration column was HP-5 (30 m \times 320 μm \times 0.25 μm). High-purity nitrogen served as carrier gas with flow rate of 1 mL/min in column. The sample injection volume was 1 μL , and flow rate was 50 mL/min. Temperatures at the sample inlet and around the detector were 250°C and 300°C, respectively.

A temperature rise program was used for the calibration column. As a result, the temperature in the column was maintained at 80°C for 1 min. After that, the temperature was increased again to 200°C with an increasing rate of 18°C/min. Then the temperature was raised to 240°C with an increasing rate of 1.5°C/min, followed by the process of temperature increased to 300°C with an increasing rate of 20°C/min for 3 min.

Quality Control Procedures

All analytical operations were conducted under strict quality control guidelines. Procedural blank and spiked samples consisting of all reagents was run to check interference and cross-contamination. Surface water, suspended solids, and sediment samples were determined with good precision and recoveries.

The limits of detection for OCPs were determined as analyte concentrations in a sample that gives rise to a peak with a signal-to-noise ratio (S/N) of 3, while the limits of quantification were determined with an S/N of 10:1. All results were lower than the limits of quantification, while those were lower than the limits of detection. The method

detection limits (MDLs) for OCPs in sediments were in the range 0.01-0.03 ng/g (dw). The spiked recoveries of OCPs in sediments ranged from 70.26% to 118.12%. The MDLs for OCPs in surface water and suspended solids were in the range 0.02-0.06 ng/L. The spiked recoveries of OCPs in surface water and suspended solids ranged from 76.11% to 108.32%.

Results and Discussion

Concentrations of OCPs in Surface Water

Eighteen kinds of OCPs (α -, β -, γ -, δ -HCH, p,p'-DDT, o,p'-DDT, p,p'-DDE, o,p'-DDE, p,p'-DDD, o,p'-DDD, tecnazene, hexachlorobenzene, heptachlor, aldrin, heptachlor epoxide (isomer A), heptachlor epoxide (isomer B), tetrachlorvinphos, and endrin) were detected in the samples from all 37 sampling sites. The residual concentrations in surface water are summarized in Table 1. The total concentrations of OCPs in water were 9.35-194.93 ng/L, with a mean value of 43.45 ng/L.

As OCPs, HCHs and DDT were widely used in China. Residues of total HCHs (including α -, β -, γ -, and δ -HCH) and DDT (including p,p'-DDT, o,p'-DDT, p,p'-DDE, o,p'-DDE, p,p'-DDD, and o,p'-DDD) in water varied from 2.41 to 178.18 ng/L (mean value of 19.85 ng/L), and 0.94 to 116.83 ng/L (mean value of 7.37 ng/L), respectively. The results showed that the content of HCHs in surface water was higher than that of DDT. This may be because the HCHs can go into the water more easily than DDTs. Among HCHs, δ -HCH isomer was far more than α -, β -, and γ -HCH. The order of the mean concentrations of the HCH isomers from high to low was: δ -HCH (14.32 ng/L) > γ -HCH (3.85 ng/L) > β -HCH (4.50 ng/L) > α -HCH (1.10 ng/L).

DDD (p,p'-DDD, o,p'-DDD) occupied 73% of total DDTs in water. The highest concentration of DDTs in water was found at the upper reaches of the Weihe (Fig. 2), which is surrounded by many villages.

Compared with other regions around the world, the levels of HCH in Weihe surface water from were lower than those in the Qiantang River, China [14] and Küçük Menderes River, Turkey [15], but were higher than those in the Chenab River in Pakistan [16], the Yangtze in China [17], and the Nestos in Greece [18] (Table 2 [14-18]). The DDT content in water from the Weihe was similar to that in the Küçük Menderes [15]. HCHs and DDT were sourced from past heavy application, as well as recent new input such as lindane and dicofol. Some kinds of OCPs (including aldrin, endrin, heptachlor, and heptachlor epoxide), which had never been used in large amounts in northwest China, could also be detected in most Weihe samples (Fig. 2).

According to the Chinese official surface water guidelines (GB3838-2002) [19], the concentrations of HCHs and DDT in a drinking water source should be below 5,000 and 1,000 ng/L, respectively. The present study showed that the concentrations of HCHs

Table 1. OCPs in water and suspended solids and sediments from the Weihe River.

OCP compounds	Water (ng/L)		Suspended solids (ng/L)		Sediment (ng/g dw)	
	Range	Mean	Range	Mean	Range	Mean
tecnazene	0.07-3.59	0.47	0.18-8.02	1.42	0.06-3.53	0.81
α -HCH	0.04-2.87	1.10	1.16-43.09	11.85	0.96-131.64	26.25
hexachlorobenzene	0.11-7.67	1.16	1.47-8.42	4.00	0.65-63.71	16.62
β -HCH	0.28-21.10	4.50	5.50-55.89	23.54	6.27-382.44	47.17
γ -HCH	0.13-24.55	3.85	6.13-120.96	36.51	14.44-235.57	70.70
δ -HCH	0.14-157.89	14.32	15.43-140.08	63.87	8.92-346.91	118.07
heptachlor	0.05-3.22	0.89	1.31-12.00	6.52	2.46-26.55	10.22
aldrin	0.11-2.00	0.54	0.31-1.70	0.83	0.09-1.93	0.49
heptachlorepoxyde(isomerA)	0.03-2.51	0.38	0.08-0.96	0.33	0.03-0.43	0.14
heptachlorepoxyde(isomerB)	0.10-10.95	2.51	0.68-6.45	2.41	0.11-4.20	1.03
o,p'-DDE	0.03-1.05	0.37	0.06-0.96	0.61	0.08-6.38	0.56
tetrachlorvinphos	0.12-11.44	1.63	0.30-6.32	2.90	0.35-8.93	2.75
p,p'-DDE	0.03-2.17	0.43	0.21-2.59	0.87	0.03-4.22	0.87
o,p'-DDD	0.01-1.00	0.15	0.01-1.02	0.30	0.01-1.92	0.22
endrin	0.37-26.28	4.74	1.06-10.63	5.26	0.25-18.62	4.53
p,p'-DDD	0.04-4.32	0.98	0.28-3.73	1.71	0.11-364.37	25.25
o,p'-DDT	0.04-115.53	4.68	0.29-2.69	1.27	0.07-18.92	1.16
p,p'-DDT	0.10-4.50	0.77	0.35-3.23	1.51	0.15-3.83	0.91
Σ HCH	2.41-178.18	19.85	51.76-241.23	135.77	74.13-517.49	262.19
Σ DDT	0.94-116.83	7.37	2.82-12.23	6.27	1.20-370.98	28.97
Σ other OCP	3.64-37.17	16.23	11.35-37.67	23.66	7.94-110.13	36.59
Σ OCPs	9.35-194.93	43.45	77.85-281.38	165.70	88.57-900.10	328.11
α -HCH/ γ -HCH	0.02-6.01	0.92	0.03-2.77	0.52	0.02-29.23	1.32
(DDE+DDD)/ Σ DDT	0.01-0.79	0.54	0.35-0.85	0.56	0.29-0.99	0.66

(178.18 ng/L) and DDT (116.83 ng/L) were much lower than the guideline values.

Concentrations of OCPs in Suspended Solids

The Weihe is not only a large river, but also has complexity in its bottom environment so that the content

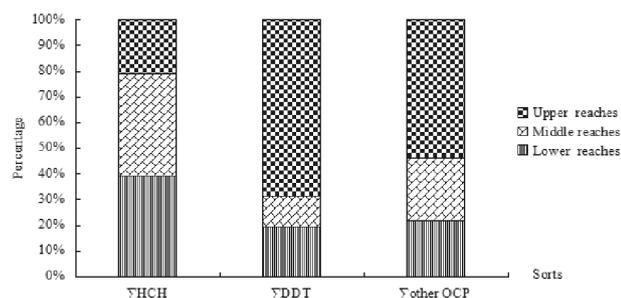


Fig. 2 Percentage composition of OCPs in surface water

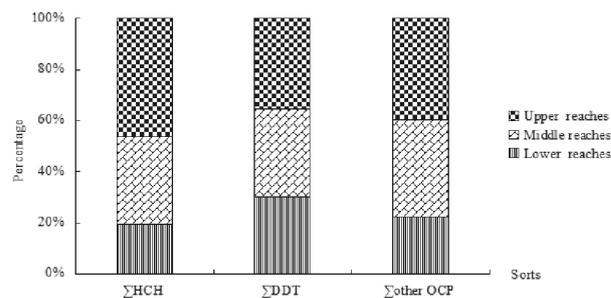


Fig. 3 Percentage composition of OCPs in suspended solids.

Table 2. Concentrations of HCHs and DDT in water and sediments of lakes and rivers around the world.

	Location	Σ HCH	Σ DDT	Reference
Water (ng/L)	Weihe River, China	2.41-178.18	0.94-116.83	This study
	Chenab River, Pakistan	0.10-6.50	2.92-15.22	2014 [16]
	Baiyangdian Lake, China	3.13-10.60	4.05-20.59	2011 [11]
	Yangtze River, China	0.55-28.07	–	2008 [17]
	Qiantang River, China	0.74-202.8	0.40-97.54	2006 [14]
	Küçük Menderes River, Turkey	101-398	nd-120	2003 [15]
	Nestos River, Greek	nd-68	nd-64	2003 [18]
Sediment (ng/g dw)	Weihe River, China	74.13-517.49	1.20-370.98	This study
	Ravi River, Pakistan	nd-16	1.5-58.5	2014 [20]
	Lake Qarun, Egypt	0.13-62.6	nd-5.88	2013 [9]
	Baiyangdian Lake, China	1.75-5.70	0.91-6.48	2011 [11]
	Hanoi area, Vietnam	<0.2-36	4.4-1100	2010 [22]
	Gomti River, India	nd-155.2	1.63-368.7	2009 [21]

^and: not detected

of suspended solids will also have great differences. In suspended solid samples, OCP concentrations ranged from 77.85 to 281.38 ng/L with a mean value of 165.70 ng/L (Table 1). These concentrations were obviously higher than in water (9.35-194.93 ng/L), perhaps because Weihe flow speed is too fast around these sites during sampling, resulting in sediment resuspension, and contaminants may be released from sediments into the suspended solids. This seems to be a local source of OCPs for these sites, which need to be further studied. The concentrations of HCHs and DDT ranged from 51.76 to 241.23 ng/L (mean value of 135.77 ng/L), and 2.82 to 12.23 ng/L (mean value of 6.27 ng/L), respectively (Table 1). The concentrations of HCHs and DDT are similar at the upper reaches, middle reaches, and lower reaches of the Weihe (Fig. 3).

Concentrations of OCPs in Sediments

HCHs and DDT were detected in all sediment samples, with total concentrations ranging from 74.13

to 517.49 ng/g, and 1.20 to 370.98 ng/g, respectively (Table 1). For HCHs, the mean concentrations of the HCH isomers in sediments decreased in the following order: δ -HCH (118.07 ng/g) > γ -HCH (70.70 ng/g) > β -HCH (47.17 ng/g) > α -HCH (26.25 ng/g). The DDD (p,p'-DDD, o,p'-DDD) occupied 88% of total DDT in sediments. The DDT level was higher than that of HCHs at the upper reaches of the Weihe (Fig. 4), in accordance with previous studies on OCP residues in sediments in China. This may be because DDT has low-water solubility, vapor pressure, biodegradability, high lipophilicity, and particle affinity compared with HCHs [9, 12].

In comparison, the levels of HCHs in Weihe sediments were much higher than those observed in Ravi River sediments in Pakistan [20], and the Gomti River in India [21]. The DDT levels in Weihe sediments were much lower than those in the Hanoi area of Vietnam [22] (Table 2 [20-22]).

Ratios of α -HCH/ γ -HCH in Environments

Composition differences of HCH isomers or DDT congeners in the environment could reveal different pollution sources. Typical technical HCH contains α -HCH (60-70%), β -HCH (5-12%), γ -HCH (10-15%), and δ -HCH (610%). γ -HCH is above 99% in lindane. It should be noted that α -HCH and γ -HCH could be converted to β -HCH in the environment. Therefore, β -HCH was the most stable isomer of HCHs because of its lower vapor pressure. The abundance of β -HCH as the only isomer indicated that most HCHs came from older residues in the study area [23]. The possible sources of HCHs in the environment are historical residues or recent inputs of

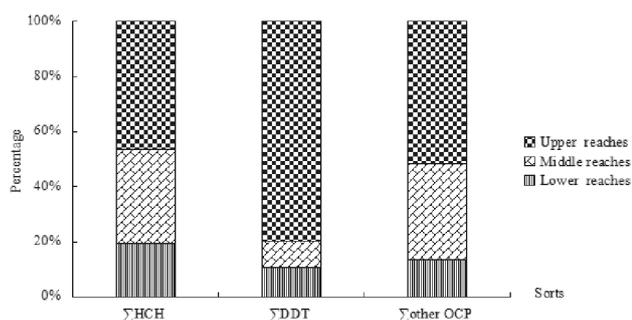


Fig. 4 Percentage composition of OCPs in sediments.

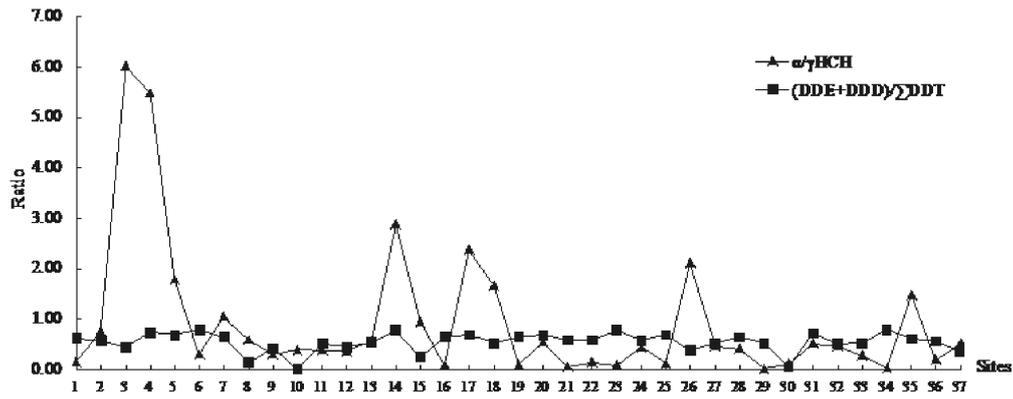


Fig. 5 Relationship between α/β HCH and $(DDE+DDD)/\sum DDT$ in surface water.

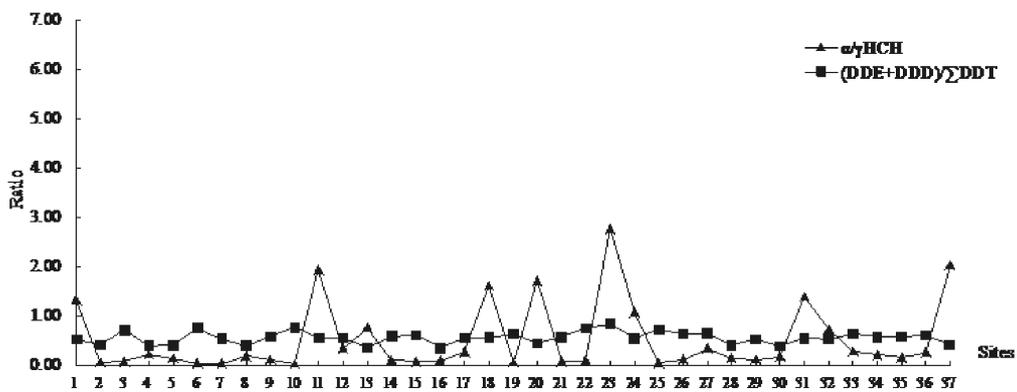


Fig. 6. Relationship between α/β HCH and $(DDE+DDD)/\sum DDT$ in suspended solids.

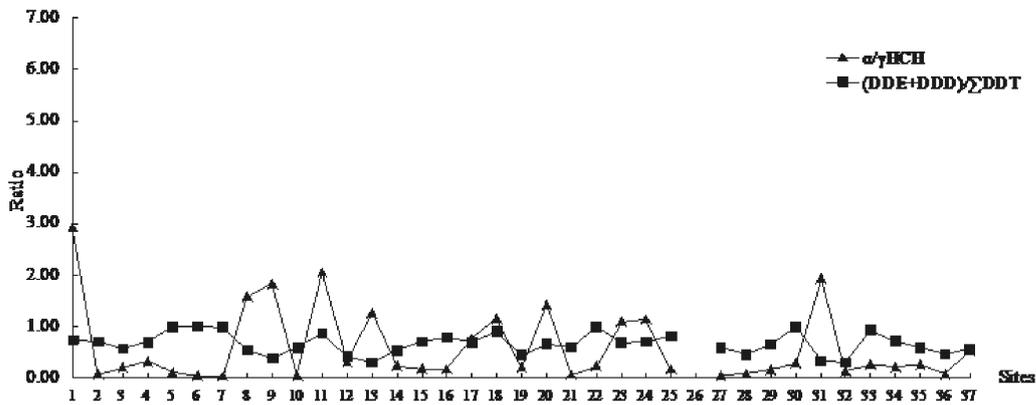


Fig. 7. Relationship between α/β HCH and $(DDE+DDD)/\sum DDT$ in sediments.

Table 3. Assessments of ecological risks of selected OCPs in Weihe River surface sediments.

Chemical	Range(ng/g)	ERL ^a	ERM ^b	<ERL(%) ^c	ERL-ERM(%) ^c	>ERM(%) ^c
o,p'-DDT and p,p'-DDT	0.34-22.60	1	7	44	53	3
o,p'-DDE and p,p'-DDE	0.17-8.81	2.2	27	78	22	0
o,p'-DDD and p,p'-DDD	0.13-364.66	2	20	64	23	13
DDTs	1.20-370.98	1.58	46.1	8	81	11

^aEffects range-low value ^bEffects range-median value ^cPercentage of samples above the corresponding levels

industrial HCHs or lindane, which can be identified by their isomer ratios such as α -HCH/ γ -HCH. In this study, δ -HCH was the predominant isomer and was detected in all water, suspended solids, and sediment samples, while α , β , γ -HCH were lower, with α / γ -HCH ratios ranging from 0.02 to 6.01 (mean value of 0.92) for water, and 0.03 to 2.77 (mean value of 0.52) for suspended solids, and 0.02 to 29.23 (mean value of 1.32) for sediments. The α -HCH/ γ -HCH ratio in areas where lindane has been used typically range between 0.2 and 1, due to the photochemical transformation of γ -HCH to α -HCH, compared to a range of 4-15 for technical mixtures of HCH [24]. The high α -HCH/ γ -HCH ratio in sites 3 and 4 range between 5 and 6 in Fig. 5, which indicated that historical usage of technical mixtures of HCHs was the main source of HCHs in the water.

Ratios of (DDE+DDD)/ Σ DDT in Environments

The relative concentration of DDT and its metabolites can provide useful information on pollution sources. Technical DDT generally contains p,p'-DDT (75%), o,p'-DDT (15%), and p,p'-DDE (5%), and less o,p'-DDE, p,p'-DDD, and o,p'-DDD. Generally speaking, a ratio of (DDE+DDD)/ Σ DDT of more than 0.5 indicates long-term biotransformation of DDT to DDE and DDD, while a ratio of less than 0.5 may indicate recent input of DDT [25]. Dicofol was probably the new input source, an acaricide manufactured from technical DDT used mainly in orchards to treat mites in Shaanxi Province on a small scale. The ratios of (DDE+DDD)/ Σ DDT found in the Weihe ranged from 0.01 to 0.79 (mean value of 0.54) for water, 0.35 to 0.85 (mean value of 0.56) for suspended solids, and 0.29 to 0.99 (mean value of 0.66) for sediments (Table 1), with most values being more than 0.5 (Figs 5-7). This indicated that the DDT compounds in the Weihe may be mainly derived from DDT-treated aged and weathered agricultural sources (note that site 26 did not collect samples in sediments).

Ecological Risk Assessment

It is necessary to maintain aquatic ecosystems of long-term stability and therefore do risk assessments in sediments. Because there is no way to evaluate the ecological risk by HCH, we evaluated DDT. The effects range-low value (ERL) and effects range-median value (ERM) guidelines [26] were applied to evaluate the possible ecological risk assessment of OCP contamination in the Weihe (Table 3). When pollutant content in sediments is less than ERL, ecological risk is less than 10%; when the pollutant content in sediments is greater than ERM, ecological risk is greater than 75% [27]. The value of DDE (o,p'-DDE and p,p'-DDE) in sediment samples is less than ERM, and the value of DDE in 78% of the samples was less than ERL. On the whole, Weihe sediments have a high ecological risk from pesticides.

Conclusions

In the present study we provided the first systemic data on OCP levels in surface water, suspended solids and sediments from the Weihe River in northwest China. Results showed that OCPs in surface water, suspended solids, and sediments were in the range of 9.35-194.93 ng/L, 77.85-281.38 ng/L, and 88.57-900.10 ng/g, respectively. HCHs and DDT were the dominating components of OCPs in water, suspended solids, and sediment samples. The concentrations of HCHs and DDT in Weihe water were all below the threshold prescribed by official Chinese guidelines (GB3838-2002), thus it is within the acceptable level. The α -HCH/ γ -HCH ratio indicated that historical usage of technical mixtures of HCHs was the main source of HCHs in the Weihe. Meanwhile, new input of γ -HCH existed in this region, indicating the recent application of prohibited pesticides such as linane, which emphasizes the importance of pesticide control in this region. The DDT ratio indicated that the DDT found at most sites came from past uses of technical DDT in the river. Compared with some guideline values for DDT in sediments, the DDT concentrations have high ecological risk. Thus, more research is needed in the future to address this contamination.

Acknowledgements

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