DETERMINATION AND ASSESSMENT OF HCHs and DDTs RESIDUES IN SEDIMENTS FROM LAKE DONGTING, CHINA

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(Received 27 September 2004; accepted 12 May 2005)

Abstract. The contamination of organochlorine pesticides hexachlorocyclohexane (HCH) and Dichlorodiphenyltrichloroethane (DDT) and their eco-environmental assessment in surface sediments from Lake Dongting, the second-largest freshwater lake in China, were studied. Concentrations of $\sum$ HCH ($=\alpha$ -HCH + β -HCH + γ -HCH + δ -HCH) were 0.21–9.59 ng/g dry weight and those of $\sum$ DDT = p,p'-DDD + p,p'-DDE + o,p'-DDT + p,p'-DDT ranged from under detectable limit to 10.15 ng/g dry weight. The ratios of α -HCH to γ -HCH were above 7 at most sampling sites while no or a small amount of β -HCH were found at all sites, suggesting the degradation of HCH used in the history and possibly current use of HCH in the region. The low ratios (below 2.0 in most cases) of (p,p'-DDE + p,p'-DDD) to p,p'-DDT and high levels of individual isomers of DDT at some sites also suggested that there have still been fresh inputs of DDT into Lake Dongting. Through the comparison between concentrations of HCH and DDT residues in sediments of Lake Dongting and those from other places in China and also from the results of our eco-environmental assessment, it can be concluded that Lake Dongting is the water body with high contamination of both HCH and DDT in its sediments in comparison with other water bodies in China.

Keywords: organochlorine pesticides, HCH, DDT, sediments, assessment, ERL, ERM

1. Introduction

Persistent organic pollutants (POPs) are pollutants with characteristics of persistence, bioaccumulation and toxicity, and the exposure to POPs can result in adverse acute and chronic effects to fishes, animals, and even to human beings, which has caused public concerns for many decades. In China, most of the pesticides produced and used from the 1950s to 1980s are organochlorine pesticides (OCPs, mainly including hexachlorocyclohexane (HCH) and Dichlorodiphenyltrichloroethane (DDT)), which are a major portion of POPs. According to Li *et al.* (1998, 2001), the total production of technical HCH produced in China from 1952 to 1983 was 4.46×10^6 tons, which is very close to 4.9×10^6 tons of accumulated consumption of this insecticide given by Lin *et al.* (2000) during the same

period of time. However, total 2.7×10^5 tons of DDT production given by Li *et al.* (1999) between 1952 and 1983 is much less than 4.0×10^5 tons of accumulated consumption of DDT during the same period of time presented by Lin *et al.* (2000). Since the use of DDT and HCH being banned in China in 1983 more than 20 years ago, residues of these two OCPs in soils have gradually reduced. However, as the main pesticides used in China in the past, relatively high levels of these two OCPs residues have still been found in some areas of China (Lin *et al.*, 2000).

As important sinks and sources for OCPs, sediments constitute a reservoir of bio-available OCPs and also play a significant role in the remobilization of OCPs in aquatic systems under favorable conditions, in interactions between water and sediment. The direct transfer of OCPs from sediments to organisms is now considered to be a major route of exposure for many species (Zoumis *et al.*, 2001). Studies on OCPs in sediments in rivers, lakes and seas have been a major environmental focus especially in the last decade in China (Feng *et al.*, 2003; Fu *et al.*, 2003; Zhang *et al.*, 2003).

Lake Dongting, the second-largest freshwater lake in China covering a large surface area of around 3000 km², is located in Hunan Province in the south of China. Lake Dongting plays an extreme important role in supporting many life forms, water supply for agriculture, aquatic product culture, fisheries, and it is also a good source of water for drinking water production by municipal water works and industry manufactures in the surrounding areas. In Lake Dongting region and the neighboring areas, agriculture and aquaculture are the most developed economic activities in Hunan Province. For example, annual production of crayfish from Lake Dongting consumed by people all over the China is 1.7×10^4 tons, accounts to above 80% of the total consumption of crayfish each year in China. Hunan is also one of the provinces with highest historical usage HCH and DDT in China. The amounts of technical HCH used in Hunan in 1980 was the highest among different provinces and autonomous regions in China, and the usage of pesticides (mainly HCH and DDT) in this province in 1970 was the second highest in China (Li *et al.*, 2001). The total use of technical HCH in Hunan was almost 10% of the total consumption in China between 1952 and 1984, among which 36% was used in 18 counties around Lake Dongting (Tu, 2001).

The extensive historical application of these two OCPs, and possibly current use of these two OCPs, could have seriously contaminated Lake Dongting. The study of contamination status of HCH and DDT in Lake Dongting region, however, has been neglected for many years, for example, data on current contamination status of HCH and DDT in Lake Dongting sediments are not available.

The objectives of the present work are to study the contamination status of HCH and DDT in Lake Dongting sediments and to assess its eco-environmental risk, as a contribution to the knowledge and rational management of the lake and its surrounding regions in the future.

2. Materials and Methods

2.1. REAGENTS

A composite stock standard solution of organochlorine pesticides including α -, β -, γ -, δ -HCH and p,p'-DDE, p,p'-DDD, o,p'-DDT, p,p'-DDT was purchased from National Research Center for Certified Reference Materials of China. The concentration of each pesticide in the stock standard solution was 100 mg/L and further diluted to obtain the desired concentration in experiments. Florisil (60–100 mesh, from Supelco (Bellfonte, USA)) was activated in drying oven at 140 °C for 24 h. All other chemicals used (n-hexane, acetone, Na₂SO₄, etc.) were of analytical grade and redistilled in all-glass system to remove impurities prior to use.

2.2. SAMPLING LOCATIONS DESCRIPTION

Lake Dongting connects in the north the Yangtze River, the longest river of China, and four main rivers (Lishui River, Yuang River, Zi River and Xiang River), which flow through the major economic areas in Hunan province, enter Lake Dongting from the west, south and east (see Figure 1). Eight sampling locations (S1–S8) selected in Lake Dongting and their geographic positions are shown in Figure 1. While S1 and S4–S7 are close to the inlets of the four main rivers converging into the lake, S2, S3 and S8 lie at the body of the lake.

2.3. SAMPLING AND SAMPLE PRETREATMENT

The top 5 cm of the surface sediment samples were collected from the eight sampling sites in Lake Dongting in March, 2004 using a modified Van Veen Grab Sampler made in China. The wet sediments were sealed and refrigerated (–4 °C) immediately to avoid any adverse changes. After being transported to the laboratory, the samples were freeze-dried in vacuum at –46 °C for 36 h, grinded evenly and sieved with a screen of 100 meshes. About 5 g of the dried sediment were weighed precisely for extraction procedure.

2.4. EXTRACTION AND INSTRUMENTAL ANALYSIS

The weighed sediment sample with 40 ml n-hexane/acetone (1:1) was extracted for 30 min each time with ultrasonic extraction twice. The extraction solutions were merged and filtered to remove solids. The filtered solution was transferred to a 250 ml separatory funnel to remove the acetone with 50 ml Na₂SO₄-H₂O (2%) and thereafter 4–5 ml H₂SO₄ (98%) was added to wash off the color twice. Additional 25 ml Na₂SO₄-H₂O (2%) was used to remove the acid from the extraction for three times. Then the organic phase was dried with anhydrous sodium sulfate (purified at 400 °C) and cleaned up with Florisil column chromatography. Finally, the sample

Figure 1. Geographical locations of sampling sites (scale 1:350 000).

solution was concentrated to 1 ml with a K-D concentrating tube for the GC-ECD analysis. The GC-ECD analysis including identification and quantification of HCH and DDT was carried out with Agilent 6890 gas chromatograph equipped with ^{63}Ni electron capture detector ($\mu\text{-ECD}$) and a fused silica capillary column (DB-5, 30 m $\times$ 0.25 mm id, and 0.25 μm film thickness) with nitrogen as carrier gas and make-up gas. The GC oven temperature was programmed as follows: initial temperature of 60 $^{\circ}\text{C}$ was held for 2 min, increased at a rate of 3 $^{\circ}\text{C}/\text{min}$ to 280 $^{\circ}\text{C}$, then was held for 5 min. The injector and detector temperature were 260 $^{\circ}\text{C}$ and 290 $^{\circ}\text{C}$, respectively. 1 μl of sample was injected with the splitless mode. The external calibration method was used to quantitatively determine the concentrations of HCH and DDT. Series of calibration solutions with different concentrations prepared from dilution of the standard solution were used to obtain calibration curves

of these two OCPs, and the correlation coefficients of calibration curves were all above 0.99. The limits of detection (LDs) were described as 3 times of signal-to-noise ratio (S/N). The detectable limits of α -, β -, γ -, δ -HCH were 0.05, 0.15, 0.06, 0.08 ng/g, and those of p,p'-DDE, p,p'-DDD, o,p'-DDT, p,p'-DDT were 0.28, 0.86, 0.07 and 0.06 ng/g, respectively.

2.5. QUALITY ASSURANCE

A blank experiment was run under the same experimental conditions to avoid any contamination. Spiking standard experiment was made to obtain the method recovery. All experiments were carried out in duplicate. The even method recoveries of α -, β -, γ -, δ -HCH were in the range of 66–94%, and those of p,p'-DDE, p,p'-DDD, o,p'-DDT, p,p'-DDT ranged from 78 to 103% in this study.

3. Results and Discussion

HCH is available in two technical formulations: technical HCH and lindane. While lindane produced in China is almost pure γ -HCH, technical HCH contains 65–70% α -HCH, 5–6% β -HCH, 13% γ -HCH, and 6% δ -HCH (Cai *et al.*, 1992; Li *et al.*, 1998), among which only γ -HCH exhibits any significant insecticidal activity. In soil, the average half-lives of α -HCH and δ -HCH are 20–50 days and 20 weeks, respectively (FAO, 2000). The ratio of α -HCH to γ -HCH, 4–7 in technical HCH and zero for lindane, can be used to indicate the degradation, formulation types and the status of application of HCH (Strandberg *et al.*, 2000; Kim *et al.*, 2002).

Technical grade DDT is actually a mixture of two isomers of DDT, including the p,p'-DDT isomer (80%) with the o,p'-DDT (20%) (Ramesh *et al.*, 1989). While DDT is highly resistant to degradation, some microbes can degrade DDT into a variety of metabolites. Among the more important of these is DDE and DDD, and DDE is more un-degradable. The ratio of (p,p'-DDE + p,p'-DDD) to p,p'-DDT can provide an indication of how long the technical DDT has been released into the environment, since the ratio increases over time as the DDT degrades (Zhou *et al.*, 2001).

In China, the use of HCH and DDT in agriculture has been restricted since 1983, but the use of technical HCH is probably continued in some areas for public health, such as epidemic prevention, and lindane has been used in agriculture since 1990 (Li *et al.*, 2001). Through evaporation (including wind-driven transport of suspended particulate) and surface runoff from the land, HCH and DDT can enter the watercourses, and ultimately into rivers, lakes and seas. River is believed to carry a considerable load of chlorinated pesticides.

The concentration distributions of HCH and DDT isomers and their metabolites in Lake Dongting sediments are shown in Table I, and concentrations for $\sum$ HCH ($= \alpha$ -HCH + β -HCH + γ -HCH + δ -HCH) and $\sum$ DDT ($=$ p,p'-DDD + p,p'-DDE + o,p'-DDT + p,p'-DDT) at different sampling sites are also shown in Figure 1.

TABLE I
Concentrations (ng/g dry weight) of HCH and DDT in Lake Dongting sediments

	α -HCH	β -HCH	γ -HCH	δ -HCH	Σ HCH	p,p'-DDE	p,p'-DDD	o,p'-DDT	p,p'-DDT	Σ DDT
S1	ND	ND	0.21	ND	0.21	2.86	4.05	ND	3.24	10.15
S2	8.21	0.88	0.50	ND	9.59	2.98	3.30	ND	3.26	9.54
S3	2.76	ND	ND	ND	2.76	ND	ND	ND	ND	ND
S4	5.81	0.88	0.44	ND	7.13	1.31	ND	ND	ND	1.31
S5	5.31	ND	0.51	ND	5.82	2.46	ND	2.44	1.99	6.89
S6	2.08	ND	0.27	ND	2.35	1.22	ND	ND	ND	1.22
S7	7.77	ND	0.65	ND	8.42	2.89	2.96	ND	2.95	8.80
S8	4.81	ND	1.53	ND	6.34	2.28	ND	2.29	ND	4.57

ND: Under detectable limit.

Concentrations for total HCH isomers ranged from 0.21 to 9.59 ng/g dry weight, while those for all DDT metabolites were in the range between ND (under detectable limit) and 10.15 ng/g dry wt. Basically, most of the concentrations of these two OCPs were higher in the sediments at the eastern side of the lake than those at the western side. Table I also shows that, while α -HCH and γ -HCH were found at most sites (7 out of 8 sites), δ -HCH was under detectable limit at all sites, and β -HCH was detected only at 2 sites (25% of total sites). Among DDT metabolites, p,p'-DDE was the most frequently detected metabolite, being found at 7 sites (87.5% of the total), in comparison with p,p'-DDD at 3 sites (37.5%), o,p'-DDT at 2 sites (25%), and p,p'-DDT at 4 sites (50%).

The ratios of α -HCH/ γ -HCH and (p,p'-DDE + p,p'-DDD)/p,p'-DDT are listed in Table II. It shows that, while the ratios of α -HCH/ γ -HCH were zero at S1 and 3.1 at S8, indicating no or very little current input of technical HCH to these sites, the ratios of α -HCH/ γ -HCH were very large at site S3 and 7.7–16.4 at other sites, indicating that current input of technical HCH occurred to these areas of the lake. On the other hands, the ratio of (p,p'-DDE + p,p'-DDD)/p,p'-DDT was zero at S3,

TABLE II
Ratios of α -HCH to γ -HCH and (p,p'-DDE + p,p'-DDD) to p,p'-DDT in sediments at each site from Lake Dongting (ng/g dry weight)

	S1	S2	S3	S4	S5	S6	S7	S8
α -HCH/ γ -HCH	0	16.4	VL ^a	13.2	10.4	7.7	11.9	3.1
(p,p'-DDE + p,p'-DDD)/p,p'-DDT	2.13	1.93	0	VL ^b	1.24	VL ^b	1.98	VL ^b

VL: Very large.

^a γ -HCH is under detectable limit, see Table I.

^bp,p'-DDT is under detectable limit, see Table I.

indicating current input of DDT to this site, the very large values of ratios of $(p,p'\text{-DDE} + p,p'\text{-DDD})/p,p'\text{-DDT}$ at site S4, S6, and S8 indicate that no current input of DDT entering these areas of the lake, and those DDTs ($p,p'\text{-DDT}$ and $o,p'\text{-DDT}$) used before has been almost metabolized.

Information of current use of technical HCH in Hunan Province is not available. The source of DDT in Lake Dongting sediments, however, could be traced to the use of pesticide dicofol. Since the use of DDT was banned in 1983, dicofol has been widely applied in agriculture in China. Considering that DDT are the major impurity of dicofol, the above fresh input of DDT in sediments at some sites maybe relates to the present agricultural use of dicofol in Lake Dongting Basin. Interestingly, high concentrations of $p,p'\text{-DDT}$ and $o,p'\text{-DDT}$ were also observed in air above Lake Taihu from July 23 to August 11, 2002, and were also blamed to the use of dicofol in the surrounding region (Qiu *et al.*, 2004).

To understand the contamination status of HCH and DDT in Lake Dongting sediments, the comparison of average HCH and DDT levels in surface sediments from different places in China was made, and the results are shown in Table III. Table III shows that, among all locations, average concentration of $\sum\text{HCH}$ in Lake Dongting sediments is the third highest, following by Mingjiang River Estuary and Lake Taihu, and average concentration of $\sum\text{DDT}$ in Lake Dongting sediments is also the third highest, following by Yangtze River Delta and Mingjiang River Estuary. This indicates that contamination status of both HCH and DDT in Lake Dongting sediments is relatively severe, which is not surprise since the usage of both technical HCH and DDT in Hunan Province in the past were high (see Introduction).

It is difficult to confirm the contamination standards and to assess the risk of pollutants including organochlorine pesticides in sediments due to complex nature of the issue. Despite many studies have been carried out, there is no uniform standard available so far (McCauley *et al.*, 2000). Many publications were reviewed and screened for the assessment on the biological adverse effects of trace heavy metals and OCPs in sediments by Long *et al.* and two guideline values (effects range-low (ERL) and effects range-median (ERM)) were suggested (Long *et al.*, 1995, 1998). The lower 10th percentile of the effects data for each chemical was identified and referred to as the ERL. The median, or 50th percentile of the effects was identified and referred to as the ERM. The guideline values and eco-environment risk evaluation of these two OCPs from different sites in this study are listed in Table IV. It can be seen that the biological adverse effects of DDT and HCH from all sites are not beyond the ERM, indicating the eco-environmental risk of these two OCPs in Lake Dongting is not high. Through comparison of the risk levels among the sites (S1–S8), the relatively higher risk levels at S1, S2, S5, S7 and S8 can be found, suggesting that the contamination of DDTs near the east bank of Lake Dongting is more serious than that of other position, it maybe relates closely to the surface runoff from the agricultural land through the four connecting rivers (Lishui River, Yuang River, Zi River and Xiang River).

TABLE III
Comparison of the average HCH and DDT levels (ng/g dry weight) in surface sediments from different places in China

DT	TH	MJ	DL	ZJ	YR	YRD	HH	DY	HK
α -HCH	4.59	1.74	0.25	0.16	0.4	0.67	3	0.38	1.26
β -HCH	0.22	3.85	2.43	0.12	0.2	1.75	ND	0.09	0.51
γ -HCH	0.51	1.67	0.48	0.18	ND	0.20	ND	0.75	1.54
δ -HCH	NA	1.36	0.07	0.22	ND	NA	ND	NA	NA
p,p'-DDE	2.00	3.29	0.48	0.46	0.2	2.64	ND	0.07	0.38
p,p'-DDD	1.29	0.95	0.92	1.13	ND	1.68	ND	0.12	1.51
o,p'-DDT	0.59	NA	NA	ND	0.31	ND	NA	NA	
p,p'-DDT	1.43	2.46	0.81	1.25	ND	2.39	ND	2.52	2.86
Σ HCH	5.32	8.62	3.16	0.68	0.6	2.62	3.0	1.22	3.31
Σ DDT	5.31	6.70	2.21	2.84	0.2	7.02	ND	2.71	4.75
Sampling date	Mar., 2004	Mar., 2000	Nov., 1999	Jul., 1996	Jul., 1996	Aug., Sep., 1986 and 1987	May, 1999	Jun., Aug., 1985 and 1986	April, 1999
Ref.	Present Study, 2004	Yuan <i>et al.</i> , 2003	Zhang <i>et al.</i> , 2003	Liu <i>et al.</i> , 2001	Hong <i>et al.</i> , 1999	Wu <i>et al.</i> , 1999	Xu <i>et al.</i> , 2000	Wu <i>et al.</i> , 1999	Zhou <i>et al.</i> , 2001
									Richardson and Zhang, 1999

Note: DT, Lake Dongting; TH, Lake Taihu; MJ, Mingjiang River Estuary; DL, Dalian Bay; ZJ, Zhujiang River Estuary; YR, Yangtze River; YRD, Yangtze River Delta; HH, Huanghe River; DY, Daya Bay; HK, Hongkong; ND is under detectable limit; NA is not available.

TABLE IV
ERL and ERM values of organochlorine pesticides (US EPA) and eco-environment risk evaluation of HCH and DDT in sediments from Lake Dongting, ng/g dry weight

	ERL	ERM	Cons.	<ERL	ERL ~ ERM	>ERM
o,p'- and p,p'-DDT	1	7	ND ~ 4.43	S3, S4, S6	S1, S2, S5, S7, S8	No
p,p'-DDD	2	20	ND ~ 4.05	S3, S4, S5, S6, S8	S1, S2, S7	No
p,p'-DDE	2.2	27	ND ~ 2.98	S3, S4, S6	S1, S2, S5, S7, S8	No
Total-DDTs	1.58	46.1	ND ~ 10.15	S3, S4, S6	S1, S2, S5, S7, S8	No
Total-HCHs	NA	NA	0.21 ~ 9.59	NA	NA	NA

Note: ND is under detectable limit, and NA is not available.

4. Conclusion

Lake Dongting is the water body with high contamination of both HCH and DDT in its sediments in comparison with other water bodies in China on the basis of the above comparison and eco-environmental assessment. Within the lake sediment contamination was more severely near the east bank of the lake than the west bank. It is suggested that the contamination of HCH and DDT in Lake Dongting sediments is related to the agricultural use of these two OCPs in the past and dicofol at present. Although the use of these two OCPs has been strictly banned in China for over 20 years, there are still recent inputs of HCH (both technical HCH and lindane) and DDT to Lake Dongting. To control the use of HCH in both agriculture and epidemic prevention and dicofol in agriculture should be the principal measure for the local governments to protect this important freshwater body.

Acknowledgement

This study was supported by National Basic Research Program of China (2003CB415006).

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