

The Fate of Organochlorine Pesticides in Water from Chaohu Lake, China

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Abstract. Forty samples of surface water and bottom water were collected from Chaohu Lake, China. The organochlorine pesticides (OCPs) in the samples were extracted by a solid-phase extraction (SPE) system and detected by Soxhlet extraction followed by gas chromatography (GC) using a mass-selective detector. The fate of OCPs in the water from Chaohu Lake was studied. The concentrations of total OCPs in the surface water and the bottom water varied from 0.17 to 2.89 ng L⁻¹ and from 0.01 to 3.49 ng L⁻¹, respectively, which were lower than total OCP concentrations detected in other lakes around the world. Sixteen types of OCPs were detected, with a concentration sequence of hexachlorocyclohexanes (HCHs) > dichlorodiphenyltrichloroethanes (DDTs) > hexachlorobenzene (HCB) > chlordanes > endosulfans. The levels of all HCH and DDT isomers in the bottom water were higher than those in the surface water. Results suggested that the presence of DDTs could be attributed to residue from technical DDTs and the recent use of dicofol, as well as antifouling paints for ships, while HCHs were most likely primarily derived from aged technical-grade HCH and lindane.

Keywords: organochlorine pesticides, chaohu Lake, HCH, DDT, source identification

1. Introduction

Organochlorine pesticides (OCPs) are types of persistent organic pollutants that were widely used in China from 1952 to 1983. [1] OCPs (such as DDTs, HCHs, chlordane, and endosulfan) were used extensively in the past to control agricultural pests. [2] There has been considerable interest from scientists in OCPs due to the high toxicity, persistence, semivolatility, and bioconcentration of these compounds. [3] They have been detected in various environmental media, such as water, sediment, soil, and air in different areas in the world, including desert plateaus, and the polar zones. They pose threats to the ecosystem and human health through their existence in the food chain; therefore, OCP pollution has become one of the most important environmental issues across the world. [4] It is imperative to study their fate and behavior in the natural environment, especially in countries where there has been large-scale use of OCPs in the past. [5] Approximately 0.27 and 4 million tons of technical-grade dichlorodiphenyltrichloroethanes (DDTs) and hexachlorocyclohexanes (HCHs) were used from 1952 to 1983 in China, accounting for 20% and 33% of the total production around the world, respectively. Although these toxic OCPs have been banned in China since 1983, they continue to be detected in different environmental media in China due to their persistence in the environment. [6]

Chaohu Lake is the fifth largest freshwater lake in China and is located in the center of Anhui Province in southeast China (Fig. 1). It is also an important source of drinking water, industrial water, and agricultural water for approximately 9.6 million residents in the coastal region; its water quality directly affects human health and economic development. There are cotton, tea, vegetable, and fruit plantations near Chaohu Basin. The climatic conditions are also suitable for growth and reproduction of termites. Some literatures have also

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reported that OCP residues and their associated risks in the water and sediment are relatively high in Chaohu Lake. [7] However, there is a need to consider the residues and sources of OCPs at different water depths in Chaohu Lake. The objective of this paper is to investigate and analyze the pollutant status and compositions of OCPs in the waters of Chaohu Lake and to explore the pollution source.

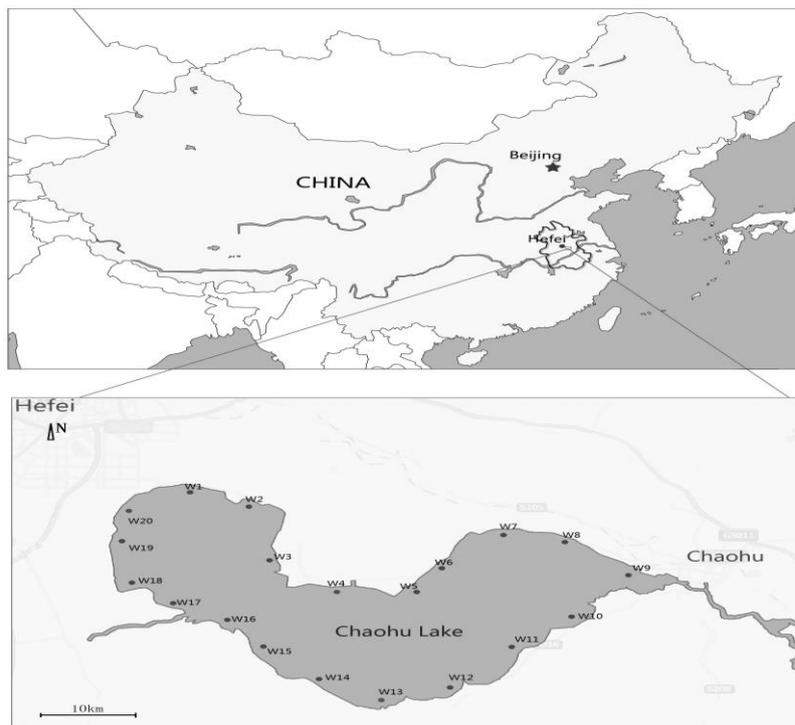


Fig. 1: Locations of sampling points

2. Materials and Methods

2.1. Sampling

A total of 40 water samples were collected from 20 sampling locations in Lake Chaohu in July 2014. Surface water samples and bottom water samples were collected from every sampling site. The sampling locations were located using a global positioning system (GPS) unit. The surface water samples were collected from a depth of 0.5 m below the water surface, and the bottom water samples were collected from a depth of 1 m above the bottom of the lake. All samples were collected with an organic glass water sampler. Fig. 1 shows the study area and the sampling locations. A volume of 3 L of water was collected for each sample, and then the samples were transported to the laboratory and stored at $-20\text{ }^{\circ}\text{C}$ (OCPs are easy to evaporate at high temperature) in a refrigerator before they were removed for chemical analysis. All of the equipment was rinsed with methanol, dichloromethane, and hexane to remove organic contaminants.

2.2. Sample Preparation and Extraction

A volume of 1 L of water from each sample was filtered through a glass filter (heated at $450\text{ }^{\circ}\text{C}$ for 4 h) and stored in an amber glass bottle at $4\text{ }^{\circ}\text{C}$ after filtration and analyzed within 12 h. 4,4'-Dichlorobiphenyl was added to the water as a recovery indicator. The organic compounds were extracted with C18 solid-phase extraction (SPE) cartridges. Before the extraction, the C18 cartridges (Supelco, USA) were washed with 5 ml of dichloromethane, 5 mL of methanol, and 5 mL of ultrapure water. The liquid level in the cartridge was tangent to the filler to keep the C18 wet. Approximately 5 g anhydrous sodium sulfate was used to condition every cartridge. The water sample was extracted with a 10-ml dichloromethane (DCM) solution for each elution, and every sample was eluted three times. The extracts were concentrated to 1 mL with a rotary evaporator. A volume of 10 mL of hexane was then added as a change solvent, and the extracts were again concentrated to 1 mL, transferred to vials, and kept sealed at $-20\text{ }^{\circ}\text{C}$ prior to the instrumental analysis.

2.3. Instrumental Analysis

The sample analysis was conducted using an Agilent 6890 Series GC System connected to an Agilent 5973 Network Mass Selective Detector. A DB-5MS fused silica capillary column (30 m×0.25 mm×0.25 mm) was used for separation. Helium gas (99.999%) was used as the carrier gas. The samples (1 µL) were injected automatically by splitless mode. The MS was operated in EI⁺ mode with selected ions. The electron energy was 70 eV. The column flow was maintained at 1 mL min⁻¹. The temperature change was programmed as follows: began at 80 °C for 1 min; ramped at a rate of 12 °C min⁻¹ to 200 °C for 10 min, then to 220 °C at 1 °C min⁻¹ and maintained for 5 min; and finally to 290 °C at 15 °C min⁻¹ and maintained for 5 min.

3. Results and Discussion

3.1. Residues of OCPs in Water

Table I: Concentrations (ng/L) of individual OCPs in surface and bottom water of Chaohu Lake

| Sources of surface water | ∑HCHs | ∑DDTs | |
|---|----------------------|-----------------------|------------|
| Ebro River, Spain | 3.1 | 3.4 | |
| Mumbai Sea, India | 0.16-15.92 (5.42) | 3.01-33.21 (12.45) | |
| St.Lawrence River, Canada | 0.06 | 0.9-22 | |
| Kucuk Menderes River, Turkey | 187-337 | 72-120 | |
| Mar Menor Lake | 30-300 | ND | |
| EI-Haram, Egypt | 20.7-86.2 | 2.300-61 | |
| rural–urban river basin, Tanzania | 4.7 | 1.27 | |
| the International Anzali Wetland, North of Iran | 57.73 | 108.83 | |
| Minjiang River Estuary, Southeast China | 205.5 | 142.0 | |
| Jinjiang River | 14.04 | 3.56 | |
| Honghu Lake | 2.97(2.36) | 0.24(0.41) | |
| Haihe River | 300-1070 | 9-152 | |
| Suzhou River | 17-90 | 17-99 | |
| the Pearl Artery Estuary | 5.8-99.7 | 0.52-9.53 | |
| Daya Bay, China | 35.3-1228.6 | 8.6-29.8 | |
| Poyang Lake (China) | 4.38-59.65 | 2.31–33.4 | |
| Chaohu Lake | 1.39 | 1.01 | this study |

Twenty-two types of OCPs analyzed in the surface water and bottom water samples are presented in Table I. Sixteen (α -HCH, β -HCH, γ -HCH, δ -HCH, o'p-DDE, o'p-DDD, o'p-DDT, p'p-DDE, p'p-DDD, p'p-DDT, heptachlor, heptachlor epoxide, cis-chlordane, trans-chlordane, Endosulfan I, and HCB,) of 22 OCPs were detected. The concentrations of total OCPs ranged from 0.17 to 2.89 ng L⁻¹, with a mean value of 2.23 ng L⁻¹ in the surface water. The concentration of total OCPs ranged from 0.01 to 3.49 ng L⁻¹, with a mean value of 2.68 ng L⁻¹ in the bottom water. The concentrations of individual OCPs in the surface water were generally lower than those in the bottom water. The detection frequencies and the means for different categories of OCPs in the surface water and the bottom water are ranked as follows: HCHs > DDTs > HCB > chlordanes > endosulfans. The high detection frequencies and levels of HCHs (including α -HCH, β -HCH, γ -HCH, δ -HCH) and DDTs (including o'p-DDE, o'p-DDD, o'p-DDT, p'p-DDE, p'p-DDD and p'p-DDT) suggest that there was a higher level of agricultural and industrial activity in past decades in the Chaohu Lake Basin. Chaohu Basin is a primary area for cotton, tea, vegetable, and fruit plantations. Historically, OCPs (such as DDTs and HCHs) were used extensively to address the need for agricultural pest control.[8] The mean concentration of HCB detected in the Chaohu Lake water samples was 0.29 ng L⁻¹, with a detection rate of 73%. All analyzed chlordane compounds (including heptachlor, heptachlor epoxide, cis-chlordane and trans-chlordane) were detected in one or more samples, and the detected levels of the chlordane parent compound, heptachlor, were lower than those of the other isomers. These results indicated that the different residual levels of OCPs might be influenced by their use in the past or their different degradation conditions in the environment. [9] Chlordane was typically used for protecting construction.

Technically, it is a mixture of many components, with trans-chlordane (13%), cis-chlordane (11%), and heptachlor (5%) being the principal components. Heptachlor was used in China from the 1960s to the 1970s. [10]

3.2. Comparisons between Areas

Compared with other lakes and rivers in the world (listed in Table II except for the HCHs content in the St. Lawrence River, Canada, the pollutant level of Chaohu area was lower than most other rivers such as Ebro River, Jinjiang River, and Poyang Lake. Except for the DDTs content in Mar Menor Lake and Honghu Lake, the pollutant level of Chaohu area was also lower than most other rivers such as Ebro River, Minjiang River Estuary, and Poyang Lake. In summary, the results indicate that the pollutant levels of OCPs in Chaohu Lake are relatively low when compared with other lakes and rivers in the world.

3.3. Compositions and Sources of OCPs

The compositions of HCHs and DDTs were variable between the surface water and the bottom water. As shown in Fig. 2, the levels of HCH and DDT isomers detected in the bottom water were higher than those in the surface water, which is due to the low water solubility of OCPs and a strong affinity for particulate matter to sink in the bottom water. [11] Among the single OCP isomers detected, β -HCH was the primary isomer detected in both the surface water and the bottom water, as shown in Fig. 2 (a). It has been reported that technical-grade HCHs were used in agriculture activities in China from 1952 to 1983. The typical composition of technical-grade HCHs is as follows: α -HCH > β -HCH > γ -HCH > δ -HCH. [12] When comparing this HCH composition to the results of our study, the HCH data we obtained indicated higher percentages of β -HCH and lower percentages of α -HCH. The reason may be that α -HCH and γ -HCH can transform into β -HCH in the environment over time. [13] In addition, α -HCH is more readily volatilized than other HCH isomers. In general, the degradation rates of HCH isomers in the environment are ranked as follows: β -HCH < δ -HCH < γ -HCH < α -HCH. [14] Therefore, residual β -HCH would be expected to be relatively higher over time. The predominance of β -HCH might suggest the input of HCHs from local agricultural activities runoff because β -HCH is most stable and resistant to microbial degradation when compared to other isomers. Furthermore, the high proportion of γ -HCH identified in the collected water samples suggests that lindane is still used in the vicinity of the Chaohu Lake region.

The composition of DDT in the collected water samples is plotted in Fig. 2 (b), and the detected DDT isomers are ranked in the following order: p'p-DDT > p'p-DDE > p'p-DDD > o'p-DDE > o'p-DDT > o'p-DDD. It has been reported that technical-grade DDT typically consists of p'p-DDT (85%). In addition, DDT can degrade to DDD and DDE under anaerobic and aerobic conditions, respectively, over time. [15] In comparison with the other DDT isomers detected during our study, p'p-DDT was the primary isomer detected in both the surface water and the bottom water, as indicated above. The high level of residual p'p-DDT is indicative of “new” sources of DDT in this region in recent years. Some studies have indicated that a “new” source of DDT pollution in China is dicofol. [16] Dicofol is a major type of insecticide currently used in China. The atmospheric transport of dust could bring high concentrations of DDT to Chaohu Lake from regions where dicofol is in use, such as Jiangsu, Jiangxi, Guangxi, and Fujian. [17] Similar research was conducted in the Taihu Lake area, in which atmospheric transport was identified as a source of DDT. [18] Another current DDT source in China is the use of antifouling paint for fishing ships. Former studies showed that the use of antifouling paint is the source for approximately 150–300 million metric tons of DDT found in the environment in China every year, and most of these antifouling paints are used for fishing ships. [16] The presence of many fishing ships in Chaohu Lake have resulted in the enrichment of DDT in the lake.

There was a high frequency of detection of HCB in the water evaluated in this study. In addition, the levels of detected HCB in the bottom water were higher than those in the surface water. It has been reported that there has not been excessive use of HCB in China. HCB is typically a byproduct of industrial production. [19] Therefore, we have concluded that the HCB detected in our study most likely originated from industrial production in the Chaohu Lake region.

Chlordanes were also detected in the water samples collected during our study. The components of technical-grade chlordane typically include trans-chlordane (13%), cis-chlordane (11%), heptachlor (5%),

and the other components. Generally, the ratio of cis-chlordane to trans-chlordane is approximately 0.77 in technical-grade chlordane. In addition, trans-chlordane can transform into cis-chlordane in the environment. A ratio of cis-chlordane to trans-chlordane greater than 1.0 is indicative of “old” chlordane. In this study, the average ratio is 0.69 in surface water and 1.05 in bottom water, which suggests that “old” chlordane sank to the bottom water and “new” chlordane enters the lake in the surface water.

Table II: Comparisons of OCPs in surface water of the other areas (ng/L)

| Compounds | Surface water | | | Bottom water | | |
|---|---------------|----------------------|-----------------------|--------------|------|--------------------|
| | Range | Mean | Detection rate (%) | Range | Mean | Detection rate (%) |
| α -HCH | BDL -0.89 | 0.34 | 63.40 | BDL -1.39 | 0.79 | 73.40 |
| β - HCH | BDL -0.81 | 0.62 | 17.90 | BDL -0.52 | 0.69 | 67.90 |
| γ - HCH | BDL -0.77 | 0.14 | 85.80 | BDL -0.88 | 0.34 | 65.80 |
| δ - HCH | BDL -0.75 | 0.45 | 90.20 | BDL -0.59 | 0.49 | 80.20 |
| o'p-DDE | BDL -0.34 | 0.18 | 47.92 | BDL -0.63 | 0.39 | 57.61 |
| o'p-DDD | BDL -0.40 | 0.01 | 33.33 | BDL -0.57 | 0.41 | 43.24 |
| o'p-DDT | BDL -0.21 | 0.07 | 12.91 | BDL -0.65 | 0.37 | 52.91 |
| p'p-DDE | BDL -0.32 | 0.24 | 45.83 | BDL -0.81 | 0.61 | 55.80 |
| p'p-DDD | BDL -0.49 | 0.07 | 23.30 | BDL -0.78 | 0.61 | 85.37 |
| p'p-DDT | BDL -0.71 | 0.66 | 57.50 | BDL -0.90 | 0.79 | 87.50 |
| Heptachlor | BDL -0.23 | 0.02 | 18.75 | BDL -0.31 | 0.22 | 18.12 |
| Aldrin | BDL | BDL | BDL | BDL | BDL | BDL |
| Heptachlor epoxide | BDL -0.53 | 0.25 | 23.71 | BDL -0.13 | 0.05 | 43.11 |
| Trans-chlordane | BDL -0.27 | 0.23 | 17.80 | BDL -0.43 | 0.22 | 27.50 |
| Cis-chlordane | BDL -0.34 | 0.16 | 8.75 | BDL -0.45 | 0.23 | 18.53 |
| Endosulfan II | BDL -0.18 | 0.11 | 14.10 | BDL -0.39 | 0.03 | 34.50 |
| Endosulfan I | BDL | BDL | BDL | BDL | BDL | BDL |
| HCB | BDL -0.51 | 0.29 | 23.00 | BDL -0.67 | 0.51 | 73.00 |
| Dieldrin | BDL | BDL | BDL | BDL | BDL | BDL |
| Methoxychlor | BDL | BDL | BDL | BDL | BDL | BDL |
| Mirex | BDL | BDL | BDL | BDL | BDL | BDL |
| Endrin | BDL | BDL | BDL | BDL | BDL | BDL |
| DDTs | 0.21-1.71 | 1.01 | 86.80 | 0.57-1.90 | 1.53 | 87.74 |
| HCHs | 0.41-1.89 | 1.39 | 74.33 | 0.52-2.39 | 1.78 | 71.83 |
| OCPs | 0.17-2.89 | 2.23 | 84.64 | 0.01-3.49 | 2.68 | 81.45 |
| Sources of surface water | | Σ HCHs | Σ DDTs | | | |
| Ebro River, Spain | | 3.1 | 3.4 | | | |
| Mumbai Sea, India | | 0.16-15.92 (5.42) | 3.01-33.21 (12.45) | | | |
| St.Lawrence River, Canada | | 0.06 | 0.9-22 | | | |
| Kucuk Menderes River, Turkey | | 187-337 | 72-120 | | | |
| Mar Menor Lake | | 30-300 | ND | | | |
| EI-Haram, Egypt | | 20.7-86.2 | 2.300-61 | | | |
| rural-urban river basin, Tanzania | | 4.7 | 1.27 | | | |
| the International Anzali Wetland, North of Iran | | 57.73 | 108.83 | | | |
| Minjiang River Estuary, Southeast China | | 205.5 | 142.0 | | | |
| Jinjiang River | | 14.04 | 3.56 | | | |
| Honghu Lake | | 2.97(2.36) | 0.24(0.41) | | | |
| Haihe River | | 300-1070 | 9-152 | | | |
| Suzhou River | | 17-90 | 17-99 | | | |
| the Pearl Artery Estuary | | 5.8-99.7 | 0.52-9.53 | | | |
| Daya Bay, China | | 35.3-1228.6 | 8.6-29.8 | | | |
| Poyang Lake (China) | | 4.38-59.65 | 2.31-33.4 | | | |
| Chaohu Lake | | 1.39 | 1.01 | | | this study |

Endosulfan was a type of pesticide used in China in the 1990s to kill pests in the crops. Endosulfan includes Endosulfan I (70%) and Endosulfan II (30%), and the typical ratio of Endosulfan I to Endosulfan II is 2.33. Endosulfan I degrades more readily than Endosulfan II, and a ratio of less than 2.33 in

analyzed samples is indicative of no input of new sources. Endosulfan II was detected in the water samples collected in our study but Endosulfan I was not detected, which suggests that there have been no recent inputs of endosulfan in the vicinity of Chaohu Lake.

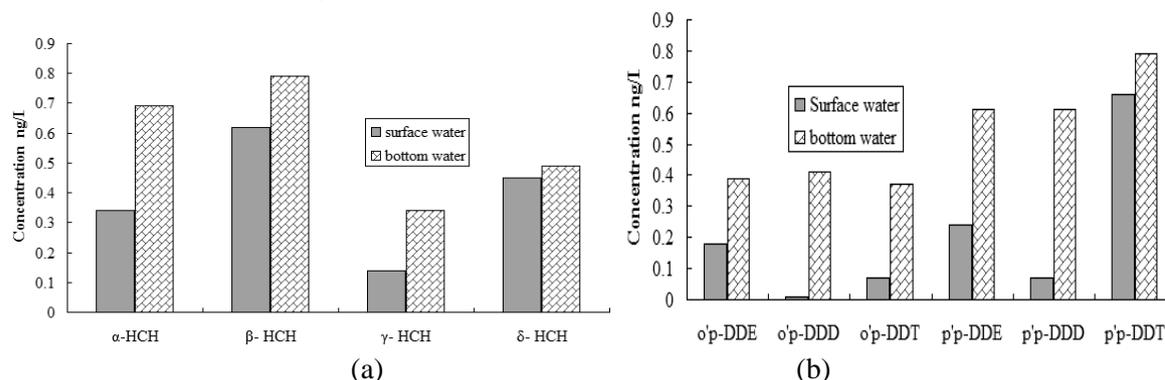


Fig. 2: The compositions of HCHs and DDTs between surface and bottom water

4. Conclusions

The present study has provided new data on the residue levels of OCPs in the water of Chaohu Lake, and the results of the study indicate low levels of OCP residues in the water. Sixteen types of OCPs were detected in collected water samples, with a concentration sequence as follows: HCHs > DDTs > HCB > chlordanes > endosulfans. Compared with the concentrations of OCPs in most rivers and lakes in China and other countries, the concentrations of OCPs detected in this study were relatively low. The levels of all detected HCH and DDT isomers in the bottom water were higher than those in the surface water. β -HCH and p,p-DDT were the primary OCP isomers detected in both the surface water and the bottom water. Based on our evaluation of the sources of the OCPs, the main sources of DDTs were residue from technical DDTs and the recent use of dicofol, as well as antifouling paints for ships, while HCHs were most likely primarily derived from aged technical-grade HCH and lindane. Therefore, in order to reduce the pollution of OCPs in Chaohu Lake, we should reduce the number of ships on Chaohu Lake; reduce the wastewater discharge from the farmland and industrial in the surrounding.

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