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Original Research

Air-Water Gas Exchange of Organochlorine Pesticides (OCPs) in Honghu Lake, China

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Abstract

Air, water, dry particulate, and wet deposition samples were collected to estimate the air-water mass transfer coefficient at Honghu Lake, China, 2005-06. Collected samples were analyzed by using gas chromatography with electron capture detection (Agilent 6890 GC-ECD). Concentration vs. flux of α-HCH (1,348 ng $m^{-2}\cdot m^{-1}$) was found to be higher volatilization in atmosphere in the month of March; however δ-HCH (286 ng $m^{-2}\cdot m^{-1}$) was deposited in water body. Further, *p,p'*-DDE (380 ng $m^{-2}\cdot m^{-1}$) metabolite was higher in Honghu lake atmosphere. The ratios of *p,p'*-DDE/*p,p'*-DDT and *o,p'*-DDT/*p,p'*-DDT indicated recent use of DDT and dicofol in Honghu Lake. The α-endosulfan (640 ng $m^{-2}\cdot m^{-1}$) was also found to be more deposited in the water body compared to β-endosulfan.

Keywords: organochlorine pesticides, air-water exchange, Honghu Lake

Introduction

Since the industrial revolution, the Earth has been influenced by several human activities, particularly in the 1950s. Production and use of synthetic chemicals for the development of human society is one such activity that has seriously damaged the environment and is difficult to repair [1, 2]. Among all synthetic chemicals, persistent organic pollutants (POPs) are of great concern to the international community [3]. Its ecosystem damage is a major global environmental issue. POPs have the following notable features in the environment [4]:

(1) persistence, slow degradation, stay for a long time in water, soil, sediment and other environmental media, retained for several years, decades or even centuries
(2) lipophilic, water-repellent, easy to concentrate in the fat of living, progressively larger along the food chain, resulting in low concentrations in the atmosphere, water, soil and eventually enter the POPs through the food chain at the highest trophic level of the human body.

POPs cause serious harm to human health [5, 6]. For example, they have a "carcinogenic," "teratogenic," and "mutagenic" effect and will interfere with the human endocrine system, giving rise to the "feminization" phenomenon, a class of very important environmental hormone pollutants [7, 8].

OCPs are considered to be semi-volatile, and are transported in the atmosphere both in the gas and particle phases [9, 10]. Various factors that influence the distribution of OCPs between gas and particle phases include vapor pressure of the compound, ambient temperature, particle concentration in air, and the nature of particles [9]. The distribution of the OCP compound between the phases plays an important role in the deposition process [11].

China began to produce organochlorine pesticides (OCPs) in the 1950s, and this production continued until it

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was banned in the 1980s [12]. However, a number of OCPs are still being produced and used for non-agricultural purposes (DDT for malaria control and for dicofol production) [12]. Some OCPs were reintroduced later, such as chlordane in 1988 [13], while others were produced for the first time, such as lindane in the early 1990s [14]. The concentration of OCPs in China is still high even after it was banned [15, 16]. It would be challenging to know where these OCPs come from. However, only limited information on the sources and environmental behaviors of OCPs is available. OCP gradients across the air-water interface may have reversed and large water bodies may currently becoming sources of organic contaminants to the atmosphere. In this paper, we report the concentration of HCHs; DDTs and Endosulfans determined simultaneously in air and surface water from Honghu Lake. Further, this paper aims to investigate the magnitude and direction of air-water exchange fluxes of OCPs at Honghu Lake, China.

Material and Methods

Study Area

Honghu Lake (Fig. 1) is located southwest of Honghu City at latitude 29º49'N and longitude 113º17'E. Honghu city is known to be the city of a hundred lakes. Honghu Lake constitutes 348.2 km^2 surface area and is in the shape of a polygon, stretching across South Hubei Honghu, with a mean depth of 1.34 m and a maximum depth of 2.3 m [17]. Hubei province is one of the most well-known freshwater districts in central China.

Air and Water Sampling

Twenty six water samples were collected from thirteen sampling points (Fig. 1) during February 2005 to January 2006, covering entire seasons. Water samples in the winter were collected only in the daytime; otherwise they were collected in the evening during summer seasons. All the water samples were collected in pre-acid-washed and cleaned polyethylene bottles of 5 liters capacity, using a water sampler for taking the water samples from various depths (<3 m). Water samples were filtered with steel less steel mesh (50 µm aperture) to remove algae and some suspended particulate matter. Rainwater also was collected during spring, summer, autumn, and winter seasons.

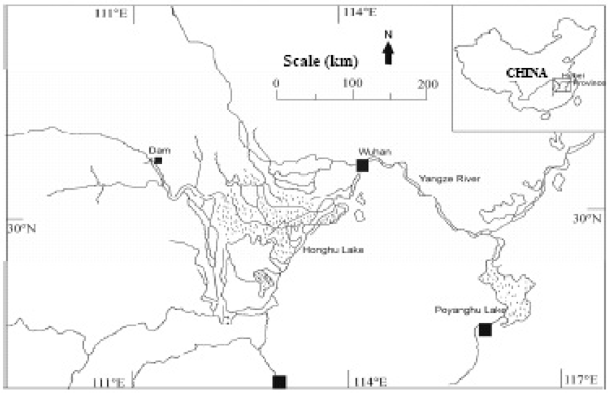


Fig. 1. Map showing Honghu Lake and sampling sites.

Sixty air samples were collected from five sampling stations around Honghu Lake during the four seasons. Ambient air samples were deployed for 24 h at a height of 10 m using high volume sampler at 1.1 m^3·min^{-1} through glass-fiber (Whatman EPM2000) filters (20.36 × 25.4 cm) followed by two 7.8 cm diameter, 7.0 cm thick polyurethane foam (PUF) plugs. The filters were prewashed in hexane and dried at 500ºC for 8 h and then in desiccators before and after sampling. The filter-paper was weighed before and after the sample collection to determine the dust load. The PUF plugs were pre-cleaned by squeezing with detergent and then with analytical-reagent grade hexane. The further clean-up of the PUF plugs using hexane was done in a Soxhlet extractor for 5 d. During the Soxhlet cleaning, the hexane was changed three times. After the Soxhlet cleaning, the PUF plugs were checked for contamination and interfering substances using gas chromatography with electron capture detection (Agilent 6890 GC-ECD). If the PUF plugs were found to contain organochlorine residues or interfering substances, they were further cleaned to remove these residues. The cleaned PUF plugs were immediately dried in desiccators and then sealed in polyethylene bags until sampling. After sampling, the PUF plugs were sealed in polyethylene covers and stored in a deepfreeze at -20ºC prior to Soxhlet extraction.

Analytical Technique

For the air samples, PUF plugs and filter-paper were Soxhlet extracted with 50 ml of hexane for 16 h. Concentrated sulfuric acid was then added to the extract in a ratio of 1:10 by volume to oxidize organic compounds, mostly fats. Excess acid was removed by washing with deionized water. The sample was dried by passing it through a glass column filled with anhydrous sodium sulfate. The hexane fraction was evaporated to reduce the volume and was further cleaned using a Florisil column. For the lakewater, 500 ml of each sample were taken and filtered through a Millipore system using a vacuum pump. The filtered sample was then extracted with 50 ml of hexane to obtain the dissolved organochlorine residues in the lakewater.

OCPs were analyzed by GC-ECD. The instrumental condition of GC-ECD is an Agilent 6890 gas chromatograph equipped with a 63Ni electron capture detector (GC-ECD) and an HP-5 (30 m × 0.32 mm i.d. × 0.25 µm film thickness) fused silica capillary column. Nitrogen was used as a carrier gas at 2.5 ml/min under the constant flow mode. Injector and detector temperatures were maintained at 290ºC and 300ºC, respectively. The temperature program is as follows: the oven temperature began at 100ºC (equilibrium time 1 min), rose to 200ºC at 4ºC/min, then to 230ºC at 2ºC/min, and at last reached 280ºC at a rate of 8ºC/min, holding for 15 min. A 2 µl sample was injected into the GC-

ECD for analysis. A six-point response factor calibration curve was established to quantify the target analyses (according to a series of standard samples with 10, 20, 50, 100, 150, and 200 ng ml^{-1} concentrations). Data were acquired and processed with the HP-3365 Chemstation software.

Quality Control and Quality Assurance (QC/QA)

Method blanks (solvent), spiked blanks (internal standard compounds were spiked into solvent) and field blanks were analyzed. No target matters were detected in field blanks and method blanks.

Surrogate standards were added to each sample to monitor procedural performance and matrix effects. The average recoveries of the surrogates of TCMX and PCB209 were more than 80%, respectively.

Air-Water Gas Exchange

The two-film resistance model developed by Whitman [18] and further modified by Liss and Slater [19], Mackay and Yeun [20], and others to estimate air-water exchange.

$$F = K_{ol}(C_w - C_a RT/H) \quad (1)$$

...where: R is the universal gas constant, T (K) is the absolute temperature in Henry's law constant (Pa m^3·mol^{-1}), C_w and C_a are the dissolved and gaseous concentrations of target compounds, which are considered to be the measured air and water concentrations. K_{ol} is determined by mass transfer coefficients on the air (K_a) and water (K_w) side as follows:

$$1/K_{ol}=1/K_w + RT/HK_a \quad (2)$$

For water side mass transfer, first need to estimate K_w (cm·h^{-1}) for CO_2 from [18] as follows:

$$K_w\,(CO_2) = 0.45\, u_{10}^{1.64} \quad (3)$$

...where u_{10} is wind speed at 10 m height (ms^{-1})

From Schwarzenbach et al. [21], air side mass transfer coefficients K_a are estimated with that of H_2O as:

$$K_{a(H_2O)}=0.2\, u_{10}+0.3 \quad (4)$$

and

$$K_{a(x)}= K_{a(H_2O)}[D_{a(x)}/D_{a(H_2O)}]^{EXP}$$
$$= (0.2\, u_{10}+0.3)\, [D_{a(x)}/D_{a(H_2O)}]^{EXP} \quad (5)$$

...where:

$$\mathrm{EXP} = \begin{cases} -0.5 & \text{if } u_{10} > 3.6\ \mathrm{ms}^{-1} \\ -0.67 & \text{if } u_{10} < 3.6\ \mathrm{ms}^{-1} \end{cases} \quad (6)$$

This exchange may be further subdivided in to deposition and volatilization to quantify both components of this two-way exchange [22].

Results and Discussions

OCP Flux Concentrations in Lake Water

The concentrations of organochlorine pesticides (OCPs) in Honghu Lake water is measured in two different seasons and has been shown in Table 1. The average concentrations of HCHs in summer and winter season was 2.97 and 2.36 ngL^{-1}, respectively. The commercial HCHs are produced mainly in two forms; technical HCHs and lindane of purified γ-isomer. Lee et al. [23] noted that the technical grade HCH consists of four isomers; α-HCH (55-80%), β-HCH (5-14%), γ-HCH (8-15%), and δ-HCH (2-16%). The technical HCH had been widely used as pesticide all over the world and contains α-HCH as its main component [24].

Even after the ban of technical HCH in China, the concentration of α-HCH is present in Honghu Lake (1.23ngL^{-1} in summer and 0.24 ngL^{-1} in winter). This may come from the use of technical HCH, whereas γ-HCH come from both technical HCH and lindane (almost pure γ-HCH). The ratio of α-HCH/γ-HCH was 0.9 in the summer season, which indicates the use of lindane in nearby lake areas. This is similar to the southern Baltic Sea; where (ratio of α-HCH/γ-HCH) water was lower than 1.0 because of the use of lindane in the coastland [25]. However, during the winter season, β-HCH showed the higher concentration, though it has the lowest water solubility. The atmospheric deposition and discharge from agricultural activities may be responsible for higher concentrations of β-HCH in water bodies.

During the summer season, the average value of DDTs in Honghu Lake was 0.24 ngL^{-1}. Among various metabolites of DDTs, *p,p′*-DDE and *o,p′*-DDT concentration was higher in Honghu Lake (0.10 ngL^{-1}, and 0.07 ngL^{-1}, respectively) which account for 42% and 29%, followed by *p,p′*-DDT (21%), and *p,p′*-DDD (8%) of the total DDTs (Table 1). The average concentration of DDTs is higher in winter (0.41ngL^{-1}) than the concentration of DDTs in summer (0.24 ngL^{-1}). Among four metabolites of DDTs, *p,p′*-DDT concentration was higher (0.15 ngL^{-1}), accounting for 37% of the total DDTs, followed by *p,p*'-DDE (29%), *o,p′*-DDT (24%) and *p,p′*-DDD (10%). Whether it is winter or summer, concentrations of *p,p′*-DDE were higher in lake water, which may be due to aerobic degradation and transformation of *p,p′*-DDT. This could be also due to the decomposition of the DDT used in the past [26]. The ratio of *p,p′*-DDE/*p,p′*-DDT could be used to reveal the history period of DDTs released to the environment. Namely, low ratio of *p,p′*-DDE/*p,p′*-DDT indicates the recent usage of DDTs and the ratio of *o,p′*-DDT/*p,p′*-DDT could show whether there was usage of dicofol. Therefore, in lake water *p,p′*-DDE/*p,p′*-DDT ratio ranges from (0.8-2) implied that recent used of DDT. Further, *p,p*'-DDE and *p,p*'-DDT were only detected in the dissolved phase. Therefore, Henry's Law constant does not show fluctuation in ratios.

The technical endosulfan mixture contains approximately 70% α-Endosulfan and 30% β-Endosulfan. Both isomers of Endosulfan undergo degradation to form

Table 1. Organochlorine pesticides exchange flux data in air and water.

Compounds	Lake water (ng/L)				Concentration in air (pg/m^3)
	Summer season		Winter season		
	Range	Mean	Range	Mean	
α-HCH	0.35-3.43	1.23	0.06-0.81	0.24	52
β-HCH	0.12-0.60	0.33	0.49-1.72	1.12	9
γ-HCH	0.33-2.74	1.31	0.23-1.61	0.66	51
δ-HCH	nd -0.89	0.10	nd -1.29	0.33	51
ΣHCHs	0.94-7.04	2.97	0.79-4.00	2.36	163
p,p′-DDT	nd -0.15	0.05	0.07-0.24	0.15	105
p,p′-DDE	0.04-0.18	0.10	0.06-0.28	0.12	44
p,p′-DDD	nd -0.06	0.02	nd -0.15	0.04	17
o,p′-DDT	nd -0.26	0.07	nd -0.26	0.10	159
ΣDDTs	0.06-0.48	0.24	0.15-0.83	0.41	325
α-Endosulfan	nd -0.44	0.08	nd -0.06	0.02	103
β-Endosulfan	nd -0.29	0.08	0.02-0.12	0.07	20
ΣEndosulfan	nd -0.73	0.16	0.02-0.14	0.10	123

nd: Not detected
ΣHCHs: sum of α-, β-, γ- and δ-HCHs
ΣDDTS: sum of *pp′*-DDE, *pp′*-DDD, *pp′*-DDT and *op′*-DDT
ΣEndosulfans: sum of α-Endosulfan and β-Endosulfan

Table 2. Organochlorine pesticide subsidence quantity in Honghu Lake (ng·m^{-2}).

Seasons	Spring		Summer		Autumn		Winter		Whole year	
Rainfall amount	357.4 mm		371.6 mm		240.1 mm		144.9 mm		1,114 mm	
Compound	Dry deposition	Wet deposition	Dry deposition	Wet deposition	Dry deposition	Wet deposition	Dry deposition	Wet deposition	Total dry deposition	Total wet deposition
α-HCH	9.6	83.4	11.1	66	18.1	117.9	9.3	48.9	48.1	316.2
β-HCH	19.8	133.2	17.1	100.9	28.8	179.7	26.4	73.2	92.1	487
γ-HCH	48.4	225.2	31.3	144.9	42.3	158	15	94.3	137	622.4
δ-HCH	70.9	179.2	20.1	76.3	42.6	136.3	19	74.2	152.6	466
∑HCHs	144.4	621	79.6	389.5	131.9	591.9	69.8	290.6	425.7	1893
p,p’-DDT	64.6	232.7	45.6	76.3	44.4	76.9	44.9	64.1	199.5	450
p,p’-DDE	15.5	17.4	14.3	11.6	49.8	24.2	33.8	35.3	113.4	88.5
p,p’-DDD	15.1	36.1	9.2	11.6	20.3	13.4	17.5	2.0	62.1	63.1
o,p’-DDT	55.1	176.7	34.3	722	35.6	123.7	26.5	13.1	151.5	1035.5
∑DDTs	146.6	462.9	103.5	820.3	150	238.3	122.7	114.5	522.8	1636
α-endosulfan	10.1	12.4	9.3	25.9	14.2	20.9	7.3	24.2	40.9	83.4
β-endosulfan	47.9	82.1	15	49.2	54.1	113.7	28.2	41.9	145.2	286.9
∑endosulfan	54.7	94.6	24.2	76.3	68.3	134.6	35.5	65.6	182.7	371.1

Endosulfan sulphate [27]. The concentration of α-endosulfan and β-endosulfan was higher in summer than winter, indicating deposition of endosulfan in water body. The α-Endosulfan isomer, which is more volatile than the β-endosulfan, can easily dissipate into the atmosphere [28]. This may suggest that atmospheric transport and deposition (by runoff) will be the sources of endosulfan in Honghu Lake. Furthermore, wastewater discharge from agricultural and industrial sources is the main pollution source in Honghu Lake [17].

OCP Flux in Atmosphere

The compositions of organochlorine compounds, including its isomers and metabolites, are considered to be a useful tool for understanding the formulation types, origin and transport pathways, etc. The concentrations of OCP isomers in air are shown in Table 1. The organochlorine compounds contamination pattern found in rank concentrations were the DDT>HCH>endosulfans. The value of HCH residues in air was found to be between 9 and 52 pg·m^{-3}. The DDT residues in air range between 17 and 159 pg·m^{-3}. Furthermore, the ratio of *p,p′*-DDE/*p,p′*-DDT in air was 0.4, indicating the recent used to DDT. However, *o,p′*-DDT/*p,p′*-DDT ratio was 1.5, implying that dicofol was used in surrounding Honghu Lake.

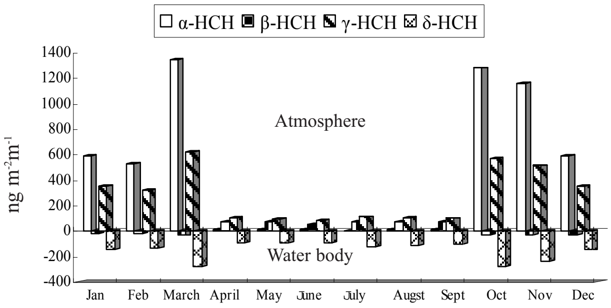
Endosulfan is persistent in the environment [29], and its isomers can transport through air from one place to another. The concentration of α-Endosulfan and β-Endosulfan was 20 and 103 pg·m^{-3}, respectively. The source of endosulfan may be attributed to its intensive use in rice fields. And other studies found the high emission and residues of α, β-endosulfan in agricultural soil in China [30].

Concentration of OCP in Wet and Dry Deposition

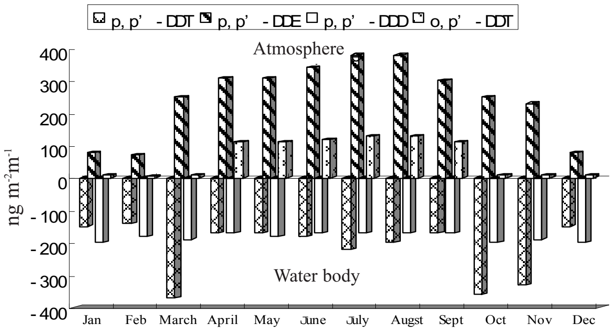
According to the phase state and process, there are 3 manners of OCP exchange between water and atmosphere i.e. grained phase deposition (dry deposition plus wet deposition), dissolved wet deposition, and water-gas interface exchange. The former is controlled by atmosphere grain and rainfall belongs to a non-dispersion process, while the later is caused by molecular random movement belonging to a dispersion process. Table 2 shows the organochlorine pesticides flux concentration in wet and dry depositions in a year. After calculating these values, it clearly shows the amount of OCPs in dry particulate matter and also in wet deposition form. Flux from dry and wet deposition was calculated using the wind speed and temperature collected from Hubei meteorological bureau. Honghu Lake area of 348.2 km^2 whole year rainfall was 1,114 mm during 2005-06. Total HCH concentrations were higher in wet deposition (1,893 ng/m^2) than dry deposition (425.7 ng/m^2), clearly showing that during the rainfall time atmospheric OCPs deposit to the water body. Therefore, DDT and endosulfan concentration was also higher in wet deposition.

Exchange Quantity

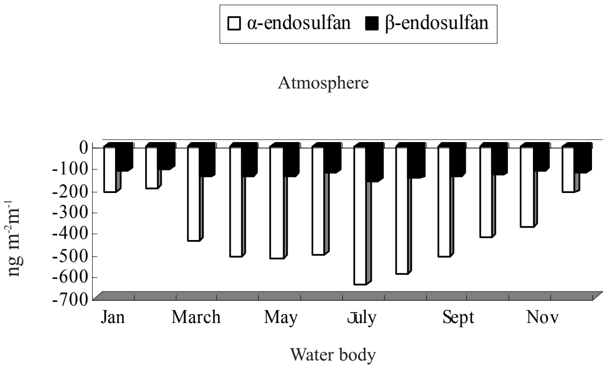
Air and water are the planet's two most important fluids, the atmosphere and water layers playing a complementary role in the migration and transformation of compounds. The contact between air and water interface is huge. The exchange of semi-volatile organic compounds in the natural environment is an important behavior of gaseous substances in the water-air interface. This process consists of two completely opposite effects, i.e. absorption of OCPs by water and volatilization of OCPs by the atmosphere. When volatilization is greater than absorption, the lake will be the atmospheric pollution. On the contrary, absorption is stronger than volatilization; the atmosphere will be the lake's pollution. In china after the ban of technical HCH 20 years ago, the air concentrations vs. flux of α-HCH in the



(a) HCHs



(b) DDTs



(c) Endosulfan

Fig. 2. OCP migration quantity flux in Honghu Lake (a) HCHs, (b) DDTs, (c) Endosulfan. (-) indicates deposition in water body, (+) indicates volatilization in atmosphere.

Honghu Lake region were higher during March (1,348 ng $m^{-2}\cdot m^{-1}$), October (1,277 ng $m^{-2}\cdot m^{-1}$), and November (1,160 ng $m^{-2}\cdot m^{-1}$) as shown in Fig. 2a. However, δ-HCH was more deposited in the water body compared to other HCH isomers.

The present study showed that more volatilization vs. flux of *p,p'*-DDE metabolite in Honghu Lake atmosphere concentration was 380 ng $m^{-2}\cdot m^{-1}$ (Fig. 2b). It shows the recent uses of DDT and dicofol in and around Honghu Lake. This observation indicated that after the implementation of Chinese chemical industry standards for dicofol, it still is found in the environment [12].

α-Endosulfan (640 ng $m^{-2}\cdot m^{-1}$) was found to be more deposited in Honghu Lake (Fig. 2c) and the present study results are similar to another study [31]. The more exchange of OCPs may happened due to the geographical proximity of the tropical countries, which still have large amounts of residues of these pesticides. Hence, these may removed rapidly from the surface water bodies to the atmosphere due to volatilization.

Conclusion

The present study clearly demonstrates that exchange of organochlorine pesticides (OCPs) takes place between the atmosphere and water body in Honghu Lake. The higher transportation rate of DDTs revealed a stronger tendency to transfer DDT contaminants from water to air. This implies that Honghu Lake water bodies principally act as a source of OCPs, while Honghu Lake passively received OCPs from the atmosphere.

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