Organochlorine Pesticides in Consumer Fish and Mollusks of Liaoning Province, China: Distribution and Human Exposure Implications

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Abstract Fish and mollusk samples were collected from markets located in 12 cities in Liaoning province, China, during August and September 2007, and 22 organochlorine pesticides (OCPs) were detected. DDT, HCH, endosulfan, chlordane, and HCB were the dominating OCPs, with mean concentrations and ranges of, respectively, 15.41 and 0.57 to 177.56 ng/g, 0.84 and below detection limit (BDL) to 22.99 ng/g, 1.31 and BDL to 13.1 ng/g, 1.05 and BDL to 15.68 ng/g, and 0.63 and BDL to 9.21 ng/g in all fish and mollusk samples. The concentrations of other OCPs generally were low and were detectable in a minority of samples, reflecting the low levels of these OCPs in the study region. In general, OCP concentrations were obviously higher in fish than in mollusks, and higher in freshwater fish than in marine fish, which indicated, first, that freshwater fish are more easily influenced than seawater fish and mollusks by OCP residues in agricultural areas and, second, that there are different biota accumulation factors for OCPs between fish and mollusk. To learn the consumption of fish and mollusk, 256 questionnaires were sent to families in 12 cities of Liaoning province. Using the contamination data, average estimated daily intakes of OCPs via fish and mollusk consumption were calculated, which were used for exposure assessment. The public health risks caused by exposure to OCPs in the course of fish and mollusk consumption were compared to noncancer benchmarks and cancer benchmarks.

Persistent organic contaminants (POCs) are among the agrochemicals and technochemistry that have been used extensively for decades in China. These compounds include organochlorine pesticides (OCPs), PCBs, PBDEs, and PCDDs. Since the 1950s, many such substances have been brought into use in large quantities in China, such as DDT and HCH; consequently, pollutant discharge to the environment has grown rapidly and caused undesirable side effects. The environmental behavior of OCPs and PCBs has been investigated for many years. As early as 1962, Rachel Carson's *Silent Spring* concluded that DDT and other pesticides, designated OCPs today, had insidious effects, harmed birds and other animals and had contaminated the entire world food supply.

Food consumption represents an important pathway for exposure to contaminants from a variety of sources, including pesticide application and industrial processing. Recent studies have indicated that exposures to contaminants in food may pose a public health risk (NRC 1993; MacIntosh et al. 1996). For example, MacIntosh et al. (1996) found that some portion of the adult population may be exposed to individual contaminants in food at concentrations above thresholds of concern. Reports from the U.S. National Research Council of the National Academy of Sciences (NRC) and the Environmental Working Group have also found that pesticide exposures to children could be high enough to cause immediate adverse health outcomes (NRC 1993; Wiles et al. 1998).

Assessments of risks to human health have been undertaken worldwide to examine the potential health risk due to exposure to toxic contaminants in various environmental media and foodstuff (NRC 1993). Large-scale farming and increasing market demand for seafood products should focus more attention on the health consequences via seafood consumption, because historical

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discharge and possible new sources of OCPs are likely to impact adversely the quality of seafood products. This study comprehensively investigates OCP contamination in fish and mollusks in Liaoning province, China. The data enabled us to assess risks related to consumption of the target fish and mollusk products. The goal of this work is to provide baseline information on levels of OCP residues in fish and mollusks in the study area and estimated dietary exposures to certain pesticides through selected food samples that are representative of the diet of the people in the study area.

Methodology

Sample Collection

Fish and mollusk samples were collected from markets in 12 cities in Liaoning province, northeastern China, during August and September 2007; the locations of sampling sites are shown in Fig. 1. For fish samples, a clean and acetone-rinsed bistoury was used to take off haslets and muscle, which were then packed in a solvent-rinsed glass bottle with a Teflon-lined cap; for mollusk samples, edible parts of shells were cut off with an acetone-rinsed bistoury and packed in a solvent-rinsed glass bottle with a Teflon-lined cap. All samples were stored at -20°C until required for extraction.

A total of 259 fish (111 marine fish and 148 freshwater fish) and 444 mollusk individuals were collected; sample species included three marine fish [hairtail (*Trichiurus haumela*), small yellow croaker (*Pseudosciaena polyactis*), and Spanish mackerel (*Scomberomorus niphonius*)], four freshwater fish [bighead carp (*Aristichthys nobilis*), common carp (*Cyprinus carpio*), silver carp (*Hypophthalmichthys molitrix*), and crucian carp (*Carassius auratus*)], and

four marine mollusks [oyster (*Crassostrea gigas*), mussel (*Mytilus edulis*), clam (*Ruditapes philippinarum*), and scallop (*Chlamys farreri*)]. For fish samples, one individual was treated as one sample, and for mollusk samples, three individuals were treated as one sample. So the final quantities of samples for marine fish, freshwater fish, and mollusks were 111, 148, and 148, respectively. Information on samples, by species, including numbers, length/height, ages, habitat use, and feeding ecology, are given in Table 1.

Extraction and Analysis

Five grams of homogenized sample and 10 g of anhydrous sodium sulfate were accurately measured into a precleaned extraction thimble, spiked with a surrogate standard mixture of CB155 and D_8 - p,p' -DDT (purchased from Supelco, USA). After sufficient mixture, the samples were then Soxhlet extracted for 18 h with 200 ml mixed solvent (hexane/acetone, 1:1, v/v). Ten percent of the extracts were used to determine lipid content gravimetrically (results according to species are given in Table 1); the remaining extracts (90%) were bathed three times using 98% H_2SO_4 and rotary-evaporated to 1 ml. Extracts were passed through a 5.5-g silica gel column (Silica 60; Merck, Germany), after pre-rinse with 25 ml of hexane, eluted with 25 ml of hexane for PCBs, HCB, and aldrin, and 50 ml of a hexane/dichloromethane mixture (1:1, v/v) for other OCPs. Elutions were rotary-evaporated to 2 ml and then reduced to 1 ml under a gentler nitrogen gas flow. All OCPs were quantified with GC-ECD (Agilent 6820). DB-5 MS of 0.25-mm ID and 30-m length was used for analysis of OCPs. The column oven temperature was programmed at an increasing rate of $15^{\circ}/\text{min}$ from an initial temperature of 80°C to a temperature of 160°C and then, at a rate of $3^{\circ}/\text{min}$, to 280°C (5-min hold). Injector and ECD temperatures were 250°C and 300°C , respectively.

Quality Assurance/Quality Control

A mixture of 22 OCP standards, including α -HCH, β -HCH, γ -HCH, δ -HCH, HCB, p,p' -DDT, o,p' -DDT, p,p' -DDE, o,p' -DDE, p,p' -DDD, o,p' -DDD, *cis*-chlordane (CC), *trans*-chlordane (TC), *trans*-nonachlor (TN), α -endosulfan, β -endosulfan, endosulfan sulfate, heptachlor, heptachlor epoxide (isomer β), aldrin, endrin, and dieldrin, was purchased from Supelco, Inc. (USA).

All samples were spiked with a labeled recovery standard containing CB155 and D_8 - p,p' -DDT prior to extraction. The surrogate standards of CB155 and D_8 - p,p' -DDT recoveries were 89 ± 18 and 105 ± 16 , respectively. Spike and blank samples were included at a rate of 1 for every 10 samples extracted, and the average spike

Fig. 1 Location of sampling sites

Table 1 Information on fish and mollusk samples

Species	Scientific name	No.	Length/ height ^a	Age	Lipid content (%)	Habitat	Feeding
Marine fish							
Hairtail	<i>Trichiurus haumela</i>	37	65 ± 18	3–4	2.15 ± 1.07	Ocean, offshore	Carnivore, little fish
Small yellow croaker	<i>Pseudosciaena polyactis</i>	37	12 ± 4	2–3	1.87 ± 0.92	Ocean, offshore	Omnivore, little fish, & shrimp
Spanish mackerel	<i>Scomberomorus niphonius</i>	37	31 ± 6	2–4	2.00 ± 1.00	Ocean, offshore	Carnivore, little fish
Freshwater fish							
Bighead carp	<i>Aristichthys nobilis</i>	37	45 ± 10	3–5	1.62 ± 0.86	Pool, lake, & river	Filter-feeding, plankton
Common carp	<i>Cyprinus carpio</i>	37	35 ± 8	2–4	1.92 ± 0.97	Pool, lake, & river	Omnivore, zoobenthos
Silver carp	<i>Hypophthalmichthys molitrix</i>	37	41 ± 7	3–4	1.94 ± 1.05	Pool, lake, & river	Filter-feeding, plankton
Crucian carp	<i>Carassius auratus</i>	37	21 ± 5	2–3	2.09 ± 1.16	Pool, lake, & river	Omnivore, organic detritus, & zoobenthos
Marine mollusks							
Oyster	<i>Crassostrea gigas</i>	111	5.3 ± 2.1	3–5	1.64 ± 0.71	Tideland	Filter-feeding, unicellular algae
Mussel	<i>Mytilus edulis</i>	111	3.1 ± 1.4	2–3	1.76 ± 0.79	Bottom-dwelling	Filter-feeding, unicellular algae
Clam	<i>Ruditapes philippinarum</i>	111	4.5 ± 2.3	2–3	1.58 ± 0.79	Bottom-dwelling	Filter-feeding, unicellular algae
Scallop	<i>Chlamys farreri</i>	111	6.3 ± 3.2	2–3	1.65 ± 0.77	Bottom-dwelling	Filter-feeding, unicellular algae

^a Length is used here for fish samples; it is from the front of the head to the end of the tail. Height is used for mollusk samples; it is from the center of opening sites to the closed end

recoveries fell in the range of 75–98%, except for *p,p'*-DDE (63%). The instrument detection limits (IDLs) were determined by assessing the injection amount that corresponded to a signal-to-noise ratio of 3:1. For α -HCH, β -HCH, γ -HCH, δ -HCH, HCB, aldrin, CC, TC, α -endosulfan, *trans*-nonachlor, and *p,p'*-DDE, IDL values were 1 pg, and the values for other OCPs reached 5 pg. Results are reported only if the signal exceeded three times the baseline noise. All blanks were below the IDL.

Results and Discussion

Analysis of General Contamination

Preliminary analysis indicated that DDT (sum of *p,p'*-DDT, *o,p'*-DDT, *p,p'*-DDE, *o,p'*-DDE, *p,p'*-DDD, and *o,p'*-DDD), HCH (sum of α -HCH, β -HCH, γ -HCH, and δ -HCH), endosulfan (sum of α -endosulfan, β -endosulfan, and endosulfan sulfate), chlordane (sum of *cis*-chlordane, *trans*-chlordane, and *trans*-nonachlor), and HCB were the predominant and ubiquitous OCPs residues in the fish and mollusk samples. The mean concentrations, concentration ranges, and occurrences of these organochlorine pesticides by species are listed in Table 2. In all samples (including fish and mollusks), DDT concentrations ranged from 0.57 to 177.56 ng/g [all normalized to wet weight (ww) except where indicated], and occurrence frequencies were 100%. The concentration ranges and occurrence frequencies, respectively, of HCH, endosulfan, chlordane, and HCB were BDL (below detection limit) to 22.99 ng/g and 95%, BDL to

13.1 ng/g and 67%, BDL to 15.68 ng/g and 40%, and BDL to 9.21 ng/g and 53%, respectively; details are discussed in the following. For other OCPs, such as heptachlor, heptachlor epoxide, aldrin, endrin, and dieldrin, the occurrence frequencies were 5, 11, 4, 3, and 3% (Table 3), respectively; this is not discussed further here, because only a small portion of the samples was analyzed. The residue levels of these seldom detected OCPs are also listed in Table 3.

Analysis of OCP Concentrations

DDT

DDT showed the highest concentration of all OCPs analyzed. Concentrations of DDT were 1–2 orders of magnitude higher than those of HCH, endosulfan, chlordane, and HCB. Higher concentrations of DDT in fish and mollusks have also been reported by other groups (Nakata et al. 2002; Yang et al. 2006; Guo et al. 2007; Wang et al. 2007) in other regions of China, which indicates that these pollutants are ubiquitous in the biosphere of China. Two possible reasons for the higher DDT concentrations in the study area are the heavy historical usage and the existence of a potential new source. DDT was in widespread use in China from the 1950 s to 1983 (Li et al. 1999), and a recent study reported that DDT is still being produced in China for use in malaria control and is also present as an impurity in dicofol, an insecticide currently used in China (Qiu et al. 2005). The mean concentration of DDT in all fish samples was 18.75 ng/g in this study, higher than the 6 ng/g reported by Meng et al. for Guangdong province, China (2007). The

Table 2 Concentrations [mean $\pm$ SD (range) ng/g, ww] and occurrences (n =) of major detected organochlorine pesticides in fish and mollusk species in 12 cities in Liaoning province

Species	<i>p,p'</i> -DDT	<i>o,p'</i> -DDT	<i>p,p'</i> -DDE	<i>o,p'</i> -DDE	<i>p,p'</i> -DDD	<i>o,p'</i> -DDD	α -HCH	β -HCH	γ -HCH
Hairtail	3.51 $\pm$ 3.38 (BDL–11.4)	3.94 $\pm$ 5.59 (BDL–32.11)	4.96 $\pm$ 10.19 (BDL–60.6)	3.91 $\pm$ 10.92 (BDL–65.2)	3.38 $\pm$ 10.68 (BDL–65.65)	2.52 $\pm$ 2.72 (BDL–10.56)	0.09 $\pm$ 0.09 (BDL–0.43)	0.26 $\pm$ 0.21 (BDL–0.99)	0.09 $\pm$ 0.10 (BDL–0.51)
	<i>n</i> = 36	<i>n</i> = 36	<i>n</i> = 33	<i>n</i> = 36	<i>n</i> = 35	<i>n</i> = 32	<i>n</i> = 32	<i>n</i> = 35	<i>n</i> = 30
	Small yellow croaker	3.52 $\pm$ 2.98 (0.24–9.79)	4.06 $\pm$ 8.84 (BDL–54.99)	5.70 $\pm$ 32.65 (BDL–128.26)	3.01 $\pm$ 6.53 (BDL–40.00)	3.84 $\pm$ 8.40 (BDL–51.5)	4.94 $\pm$ 13.63 (BDL–60.54)	1.05 $\pm$ 1.30 (BDL–6.50)	0.21 $\pm$ 0.16 (BDL–0.65)
Spanish mackerel	4.31 $\pm$ 12.94 (0.07–80.29)	2.79 $\pm$ 1.89 (0.13–9.43)	6.94 $\pm$ 11.23 (BDL–51.2)	2.99 $\pm$ 8.82 (BDL–54.10)	2.44 $\pm$ 6.55 (BDL–39.86)	3.99 $\pm$ 12.69 (BDL–76.77)	1.03 $\pm$ 2.78 (BDL–16.78)	0.28 $\pm$ 0.34 (BDL–1.82)	0.11 $\pm$ 0.27 (BDL–1.64)
	<i>n</i> = 37	<i>n</i> = 37	<i>n</i> = 35	<i>n</i> = 35	<i>n</i> = 32	<i>n</i> = 30	<i>n</i> = 31	<i>n</i> = 35	<i>n</i> = 30
	Bighead carp	6.17 $\pm$ 19.89 (BDL–111.83)	5.07 $\pm$ 7.27 (0.09–35.5)	9.29 $\pm$ 22.04 (BDL–98.7)	4.86 $\pm$ 7.21 (BDL–39.86)	3.02 $\pm$ 3.13 (BDL–12.94)	5.81 $\pm$ 13.57 (BDL–65.65)	0.30 $\pm$ 0.43 (BDL–2.32)	0.59 $\pm$ 0.80 (BDL–4.01)
Common carp	7.22 $\pm$ 21.75 (BDL–98.04)	5.82 $\pm$ 12.23 (0.41–54.99)	8.68 $\pm$ 18.72 (BDL–98.7)	5.51 $\pm$ 11.82 (BDL–67.5)	4.50 $\pm$ 7.34 (BDL–45)	5.28 $\pm$ 9.37 (BDL–51.5)	0.29 $\pm$ 0.46 (BDL–2.65)	0.45 $\pm$ 0.42 (BDL–2.14)	0.22 $\pm$ 0.22 (BDL–0.90)
	<i>n</i> = 36	<i>n</i> = 37	<i>n</i> = 33	<i>n</i> = 34	<i>n</i> = 34	<i>n</i> = 34	<i>n</i> = 34	<i>n</i> = 33	<i>n</i> = 33
	Silver carp	4.67 $\pm$ 12.83 (BDL–78.34)	4.28 $\pm$ 3.65 (0.13–18.41)	8.71 $\pm$ 13.84 (BDL–55.44)	5.10 $\pm$ 10.20 (BDL–50.09)	4.94 $\pm$ 8.13 (BDL–43.43)	4.53 $\pm$ 7.71 (BDL–43.4)	0.34 $\pm$ 0.72 (BDL–4.34)	0.43 $\pm$ 0.53 (BDL–3.01)
Crucian carp	3.76 $\pm$ 9.13 (BDL–56.43)	5.87 $\pm$ 11.38 (0.23–54.1)	7.97 $\pm$ 20.43 (BDL–90.76)	5.18 $\pm$ 12.25 (BDL–55.44)	4.67 $\pm$ 10.88 (BDL–66.61)	5.61 $\pm$ 12.12 (BDL–54.4)	0.55 $\pm$ 1.22 (BDL–5.44)	0.73 $\pm$ 1.20 (BDL–6.76)	0.31 $\pm$ 0.89 (BDL–5.41)
	<i>n</i> = 36	<i>n</i> = 37	<i>n</i> = 33	<i>n</i> = 34	<i>n</i> = 36	<i>n</i> = 32	<i>n</i> = 29	<i>n</i> = 31	<i>n</i> = 33
	Oyster	2.63 $\pm$ 2.90 (0.15–10.31)	2.54 $\pm$ 3.83 (BDL–21.21)	1.92 $\pm$ 2.47 (BDL–10.41)	2.92 $\pm$ 7.38 (BDL–40.81)	1.82 $\pm$ 2.32 (BDL–10.75)	1.94 $\pm$ 2.40 (BDL–10.75)	0.13 $\pm$ 0.20 (BDL–1.02)	0.16 $\pm$ 0.19 (BDL–0.88)
Mussel	2.90 $\pm$ 2.08 (0.38–9.01)	1.63 $\pm$ 1.78 (0.03–5.67)	2.72 $\pm$ 6.83 (BDL–41.4)	1.70 $\pm$ 1.96 (BDL–5.67)	2.03 $\pm$ 3.89 (BDL–23.01)	2.64 $\pm$ 4.08 (BDL–23.01)	0.12 $\pm$ 0.15 (BDL–0.53)	0.21 $\pm$ 0.19 (BDL–0.68)	0.08 $\pm$ 0.14 (BDL–0.54)
	<i>n</i> = 37	<i>n</i> = 37	<i>n</i> = 32	<i>n</i> = 33	<i>n</i> = 33	<i>n</i> = 33	<i>n</i> = 28	<i>n</i> = 29	<i>n</i> = 27
	Clam	2.18 $\pm$ 1.81 (0.11–7.98)	2.54 $\pm$ 2.57 (BDL–10.56)	3.35 $\pm$ 4.43 (BDL–20.12)	1.86 $\pm$ 2.21 (BDL–6.56)	2.15 $\pm$ 2.41 (BDL–9.41)	2.28 $\pm$ 2.58 (BDL–9.79)	0.15 $\pm$ 0.19 (BDL–0.88)	0.26 $\pm$ 0.31 (BDL–1.10)
Scallop	2.89 $\pm$ 3.86 (0.15–21.2)	1.59 $\pm$ 1.78 (BDL–7.65)	2.68 $\pm$ 3.78 (BDL–18.41)	1.07 $\pm$ 1.28 (BDL–4.56)	1.41 $\pm$ 1.66 (BDL–7.65)	1.74 $\pm$ 1.84 (BDL–6.5)	0.11 $\pm$ 0.12 (BDL–0.34)	0.20 $\pm$ 0.23 (BDL–0.99)	0.08 $\pm$ 0.10 (BDL–0.4)
	<i>n</i> = 37	<i>n</i> = 34	<i>n</i> = 33	<i>n</i> = 28	<i>n</i> = 32	<i>n</i> = 32	<i>n</i> = 24	<i>n</i> = 28	<i>n</i> = 29
	Species	δ -HCH	α -Endosulfan	β -Endosulfan	Endosulfan sulfate	<i>cis</i> -Chlordane	<i>trans</i> -Chlordane	<i>trans</i> -Nonachlor	HCB
Hairtail	0.11 $\pm$ 0.20 (BDL–0.81)	0.80 $\pm$ 1.63 (BDL–6.41)	0.25 $\pm$ 0.51 (BDL–2.07)	0.14 $\pm$ 0.42 (BDL–2.10)	0.73 $\pm$ 1.05 (BDL–4.14)	0.37 $\pm$ 0.62 (BDL–3.32)	0.28 $\pm$ 0.60 (BDL–3.32)	0.40 $\pm$ 0.95 (BDL–4.14)	
	<i>n</i> = 22	<i>n</i> = 17	<i>n</i> = 14	<i>n</i> = 7	<i>n</i> = 25	<i>n</i> = 19	<i>n</i> = 14	<i>n</i> = 18	
	Small yellow croaker	0.09 $\pm$ 0.17 (BDL–0.99)	0.61 $\pm$ 1.70 (BDL–9.87)	0.36 $\pm$ 1.26 (BDL–6.50)	0.20 $\pm$ 0.54 (BDL–2.10)	0.40 $\pm$ 0.77 (BDL–4.14)	0.22 $\pm$ 0.39 (BDL–1.35)	0.19 $\pm$ 0.36 (BDL–1.35)	0.40 $\pm$ 0.77 (BDL–3.19)
Spanish mackerel	0.14 $\pm$ 0.23 (BDL–1.12)	0.93 $\pm$ 1.48 (BDL–6.41)	0.22 $\pm$ 0.56 (BDL–2.97)	0.01 $\pm$ 0.04 (BDL–0.21)	0.44 $\pm$ 0.89 (BDL–4.13)	0.32 $\pm$ 0.65 (BDL–3.40)	0.28 $\pm$ 0.62 (BDL–3.40)	0.58 $\pm$ 1.09 (BDL–4.34)	
	<i>n</i> = 29	<i>n</i> = 24	<i>n</i> = 13	<i>n</i> = 3	<i>n</i> = 16	<i>n</i> = 14	<i>n</i> = 13	<i>n</i> = 22	
	Bighead carp	0.21 $\pm$ 0.29 (BDL–1.03)	1.94 $\pm$ 2.91 (BDL–10.31)	0.26 $\pm$ 0.98 (BDL–5.65)	0.15 $\pm$ 0.69 (BDL–4.12)	0.39 $\pm$ 0.78 (BDL–3.4)	0.25 $\pm$ 0.66 (BDL–3.40)	0.07 $\pm$ 0.28 (BDL–1.39)	1.37 $\pm$ 2.15 (BDL–9.21)
Common carp	0.10 $\pm$ 0.22 (BDL–1.01)	0.93 $\pm$ 2.25 (BDL–10.08)	0.48 $\pm$ 1.07 (BDL–5.43)	0.01 $\pm$ 0.04 (BDL–0.21)	1.02 $\pm$ 2.21 (BDL–10.41)	0.75 $\pm$ 1.50 (BDL–5.80)	0.30 $\pm$ 0.76 (BDL–4.08)	0.47 $\pm$ 1.36 (BDL–6.41)	
	<i>n</i> = 23	<i>n</i> = 16	<i>n</i> = 12	<i>n</i> = 5	<i>n</i> = 14	<i>n</i> = 13	<i>n</i> = 8	<i>n</i> = 14	

Table 2 continued

Species	δ -HCH	α -Endosulfan	β -Endosulfan	Endosulfan sulfate	<i>cis</i> -Chlordane	<i>trans</i> -Chlordane	<i>trans</i> -Nonachlor	HCB
Silver carp	0.11 $\pm$ 0.21 (BDL–1.01) <i>n</i> = 24	0.76 $\pm$ 1.96 (BDL–10.09) <i>n</i> = 16	0.34 $\pm$ 0.84 (BDL–4.50) <i>n</i> = 9	0.14 $\pm$ 0.59 (BDL–3.56) <i>n</i> = 7	0.64 $\pm$ 1.33 (BDL–4.56) <i>n</i> = 8	0.68 $\pm$ 1.42 (BDL–4.57) <i>n</i> = 9	0.29 $\pm$ 0.73 (BDL–3.05) <i>n</i> = 6	0.44 $\pm$ 0.95 (BDL–4.24) <i>n</i> = 16
Crucian carp	0.29 $\pm$ 0.89 (BDL–5.41) <i>n</i> = 30	1.28 $\pm$ 2.26 (BDL–11.2) <i>n</i> = 24	0.38 $\pm$ 1.26 (BDL–6.89) <i>n</i> = 9	0.28 $\pm$ 0.83 (BDL–4.41) <i>n</i> = 12	0.48 $\pm$ 1.04 (BDL–5.44) <i>n</i> = 13	0.38 $\pm$ 0.92 (BDL–4.31) <i>n</i> = 11	0.26 $\pm$ 0.63 (BDL–3.14) <i>n</i> = 10	0.98 $\pm$ 1.55 (BDL–6.41) <i>n</i> = 27
Oyster	0.13 $\pm$ 0.17 (BDL–0.92) <i>n</i> = 33	1.07 $\pm$ 1.72 (BDL–9.21) <i>n</i> = 25	0.23 $\pm$ 0.66 (BDL–3.06) <i>n</i> = 10	0.06 $\pm$ 0.18 (BDL–0.97) <i>n</i> = 5	0.62 $\pm$ 1.33 (BDL–5.41) <i>n</i> = 16	0.43 $\pm$ 0.99 (BDL–5.41) <i>n</i> = 13	0.19 $\pm$ 0.38 (BDL–1.31) <i>n</i> = 10	0.71 $\pm$ 0.98 (BDL–3.13) <i>n</i> = 23
Mussel	0.08 $\pm$ 0.14 (BDL–0.54) <i>n</i> = 27	0.38 $\pm$ 1.08 (BDL–5.41) <i>n</i> = 12	0.12 $\pm$ 0.39 (BDL–2.09) <i>n</i> = 6	0.04 $\pm$ 0.15 (BDL–0.89) <i>n</i> = 7	0.39 $\pm$ 1.07 (BDL–5.11) <i>n</i> = 14	0.12 $\pm$ 0.22 (BDL–0.78) <i>n</i> = 11	0.13 $\pm$ 0.27 (BDL–1.13) <i>n</i> = 11	0.59 $\pm$ 1.17 (BDL–4.06) <i>n</i> = 19
Clam	0.09 $\pm$ 0.10 (BDL–0.32) <i>n</i> = 29	0.44 $\pm$ 0.72 (BDL–2.35) <i>n</i> = 17	0.34 $\pm$ 0.97 (BDL–4.35) <i>n</i> = 9	0.21 $\pm$ 0.70 (BDL–3.08) <i>n</i> = 6	0.17 $\pm$ 0.54 (BDL–3.12) <i>n</i> = 10	0.15 $\pm$ 0.39 (BDL–2.09) <i>n</i> = 10	0.14 $\pm$ 0.38 (BDL–1.98) <i>n</i> = 9	0.54 $\pm$ 0.83 (BDL–2.56) <i>n</i> = 19
Scallop	0.08 $\pm$ 0.10 (BDL–0.40) <i>n</i> = 27	0.64 $\pm$ 1.07 (BDL–4.00) <i>n</i> = 19	0.26 $\pm$ 0.72 (BDL–3.21) <i>n</i> = 9	0.24 $\pm$ 0.67 (BDL–2.67) <i>n</i> = 7	0.29 $\pm$ 0.81 (BDL–4.13) <i>n</i> = 11	0.17 $\pm$ 0.45 (BDL–2.11) <i>n</i> = 9	0.04 $\pm$ 0.12 (BDL–0.56) <i>n</i> = 5	0.51 $\pm$ 0.92 (BDL–4.00) <i>n</i> = 19

For each species, the total number of samples is 37

ww wet weight

Table 3 Minor detected organochlorine pesticide residues and their occurrence in fish and mollusks

Compound	Marine fish		Fresh water fish		Marine mollusk	
	Range	Occurrence	Range	Occurrence	Range	Occurrence
Heptachlor	BDL–1.28	3	BDL–0.98	7	BDL–0.76	8
Heptachlor epoxide	BDL–0.98	13	BDL–0.91	17	BDL–0.97	13
Aldrin	BDL–0.20	6	BDL–0.91	5	BDL–0.97	5
Endrin	BDL–0.34	2	BDL–0.98	8	BDL–0.11	1
Dieldrin	BDL–0.76	4	BDL–0.19	5	BDL–0.96	4

Note: Total numbers of samples are 111, 148, and 148 for marine fish, freshwater fish, and marine mollusks, respectively

DDT concentrations in fish and mollusk samples ranged from 0.84 to 212.16 ng/g in Dalian, China (Yang et al. 2006), and from 0.5 to 220 ng/g in Xiamen, China (Klump et al. 2002); these values are comparable to our concentrations, which ranged from 0.57 to 177.56 ng/g. DDT concentrations in fish from around the world have been reported (Kannan et al. 1995; Sapozhnikova et al. 2004; Yim et al. 2005; Darnerud et al. 2006). Generally, the median concentration of DDT (18.54 ng/g) in Liaoning province was lower than that in Vietnam (26 ng/g), Indonesia (28 ng/g), and Australia (22 ng/g) but higher than that in India (15 ng/g), Thailand (6.2 ng/g), Papua New Guinea (0.43 ng/g) Solomon Islands (4.8 ng/g), Korea (8.96 ng/g), Sweden (7.02 ng/g), and Salton Sea, USA (17.6 ng/g). It is known that DDT can be biodegraded to DDE under aerobic conditions and to DDD under anaerobic conditions, and a value of DDT/(DDE + DDD) > 1 can be used as an

indicator of possible new sources. In this study, the median value of DDT/(DDE + DDD) was 0.48, and it was >1 in 26% of samples. The results suggest that DDT concentrations in fish and mollusks are mainly due to historical use, but point sources of DDT exist currently in the study areas.

HCH

The HCH concentration ranged from BDL to 22.99 ng/g in all fish and mollusk samples in this study; this range is comparable to that reported in fish in Guangdong province (0.13–24.06 ng/g) (Meng et al. 2007) but wider than those reported in other areas of China, including Dalian (fish and mollusks, 0.15–4.25 ng/g), Tianjin (fish and mollusks, 0.35–0.54 ng/g), Shanghai (fish and mollusks, 0.044–2.64 ng/g) (Yang et al. 2006), and Zhoushan (fish, < 0.1 ng/g) (Jiang et al. 2005). In the case of fish

samples, the mean value (1.02 ng/g) in this study was similar to those in Sweden (0.96 ng/g) (Darnerud et al. 2006), Thailand (0.82 ng/g), Australia (0.34 ng/g), Indonesia (0.73 ng/g), Papua New Guinea (0.57 ng/g), the Solomon Islands (0.53 ng/g) (Kannan et al. 1995), and Korea (0.94 ng/g) (Yim et al. 2005) but lower than those in India (28 ng/g) and Vietnam (1.8 ng/g) (Kannan et al. 1995). Historically, the quantity of technical HCH used in China (4500 kt) was much higher than that of DDT (270 kt) (Li et al. 1998, 1999, 2001), but according to the residue data, a much lower concentration of HCH was observed. There could be two reasons for this: first, HCH has a higher biodegradability and a lower lipophilicity than DDT (Guo et al. 2007); and second, DDT has been released to the environment since 1983 due to the use of dicofol and other purposes (Qiu et al. 2005).

Chlordane

As an insecticide, chlordane has been widely used in China for termite control (Zhao 2005). In this study, the occurrence frequencies of chlordane were 42% in fish, ranging from BDL to 15.68 ng/g, with a mean concentration of 1.25 ng/g, and 36% in mollusks, ranging from BDL to 10.82 ng/g, with a mean concentration of 0.71 ng/g. The mean concentration in mollusk was comparable to those reported in Dalian (0.82 ng/g) (Yang et al. 2006) and Fuzhou (0.96 ng/g) (Monirith et al. 2003) and lower than those in Zhenjiang (3.48 ng/g), Shanghai (3.74 ng/g), Xiamen (2.02 ng/g), and Shenzhen (1.43 ng/g) (Monirith et al. 2003). Compared to other countries, the mean concentration in fish (1.25 ng/g) was higher than that in Vietnam (0.11 ng/g), Papua New Guinea (0.37 ng/g), Indonesia (0.45 ng/g), Solomon Islands (0.57 ng/g), but lower than India (2.4 ng/g), Thailand (2.6 ng/g), and Australia (51 ng/g) (Kannan et al. 1995).

HCB

HCB was used principally as an intermediate to produce pentachlorophenol (PCP) and Na-PCP in closed systems (Hu et al. 2007). The occurrence frequencies of HCB were 53% in fish, ranging from BDL to 9.21 ng/g, with a mean concentration of 1.66 ng/g; these values were much higher than those in other regions of China reported by Monirith et al. (2003), and also higher than those in other countries reported by Kannan et al. (1995), but much lower than those in Australia [range, <0.01–60; mean, 4.2 ng/g (Kannan et al. 1995)].

Endosulfan

Technical endosulfan, which consists of α - and β -isomers, is one of the few cyclodiene pesticides that is still used extensively throughout the world to control a number of

insects on crops. In the environment, the cyclic sulfite group of endosulfan can be oxidized to the corresponding sulfate (endosulfan sulfate) (Chandler et al. 1991; Guerin et al. 1992; Kathpal et al. 1997), which is persistent than its parents (Guerin 2001). In China, endosulfan is currently used as pesticide for cotton, wheat, tea, tobacco, and apple tree (Jia et al. 2009). As for all samples (include fish and mollusks), endosulfan concentrations varied from BDL to 13.1 ng/g, with a mean value of 1.50 ng/g, and the occurrence frequency was 67%. Due to the rarity of reports on endosulfan in fish and mollusk, few comparable data are available. It was reported that the endosulfan concentration in seafood from Guangdong province, China, ranged from 0.04 to 1.89 ng/g (Guo et al. 2007), much lower than that in this study. According to the report by Jia et al. (2009), compared to Guangdong province, endosulfan was more comprehensively used in Liaoning province, due to the need for control of insects on cotton and apple; we conclude that the greater usage led to the higher concentrations in fish and mollusks in Liaoning province.

Comparing OCPs in Different Fish and Mollusk Species

Toxic chemicals released to the environment from point sources such as industrial and municipal discharges and from nonpoint sources such as agricultural runoff and atmospheric deposition have contaminated surface waters across China. Many chemical pollutants concentrate in fish and mollusks by accumulating in fatty tissues or selectively binding to fish muscle tissue. Even extremely low concentrations of bioaccumulative pollutants detected in water or bottom sediments may result in accumulation in fish and shellfish. Thus aquatic organisms are commonly used for environmental monitoring of pollutants such as heavy metals, OCPs, and polychlorinated biphenyls (PCBs). In the aquatic system, fish and mollusks are on different positions in the food chain, thus different fish and mollusk species may lead to different OCPs concentration. Figure 2 displays the mean concentration of OCPs in various fish and mollusk species, which were divided into three groups (I is marine fish, II is freshwater fish, and III is mollusks). In general, OCP concentrations were obviously higher in fish than in mollusks and higher in freshwater fish than in marine fish. These results indicate that OCP accumulation is greater in freshwater fish than in marine fish and mollusk. This probably reflects the impact of contaminant discharge from Liaoning province in northern China. Typically, seawater fish and mollusks are cultured in intertidal zones, small bays, and shallow seas, while freshwater fish are raised in ponds, lakes, reservoirs, and river channels, which are close to agricultural areas. Freshwater fish may be more easily influenced than seawater fish and mollusks are by OCP residues in agricultural areas. In

Fig. 2 Average concentrations and standard deviations of DDT, HCH, endosulfan, chlordane, and HCB in (I) three marine fish, (II) four freshwater fish, and (III) four mollusks. SYC small yellow croaker, SM Spanish mackerel, BC bighead carp, CC-1 common carp, SC silver carp, CC-2 crucian carp

addition, fish are at the top of the food chain in the aquatic system, so due to the biota accumulation feature of OCPs, fish suffer compound accumulation more easily than mollusks; our results reflect these characters.

Estimating Exposure

The analysis estimates exposure to specific contaminants via foods by combining data on contaminant concentrations in foods, which is commonly used to assess human risk. The acceptable daily intake (ADI) recommended by the Food and Agriculture Organization and World Health Organization (FAO/WHO) is usually used to assess human exposure to target contaminants, without consideration of different eating habits and consumption rates. To estimate individual exposure from fish and mollusks, we calculated the estimated daily intake (EDI; ng/kg body weight/day) of OCPs by the following equation:

$$EDI = \frac{FDC \times CC}{BW} \quad (1)$$

where FDC is food daily consumption, CC is the mean contaminant concentration of OCPs, and BW is body weight, for which 60 kg is typical (IPCS 2006).

To determine the consumption of fish and mollusk, 256 questionnaires were sent to the families in 12 cities of Liaoning province. The questionnaires included the information number of people, consumption frequencies and quantities consumed of these foods (researched in this paper), and data on consumption of other fish and mollusks. After statistical calculations, we got the general consumption information on these foods in Liaoning province, and the results are presented in Table 4. Amounts consumed ranged from 20.45 to 50.12 g/day, with a mean value of 34.08 g/day, for marine fish, from 23.45 to 40.35 (mean = 32.78) g/day for freshwater fish, and from 13.24 to 38.47 (mean = 24.12) ng/day for mollusks. The consumption data arrived at are a little lower than the average consumption value of fishery food from the China Statistical Yearbook [38.90 g/day (NBSC 2008)].

Table 4 Consumptions (g/day) of fish and mollusk according to city in Liaoning province

City	No. of questionnaires	Marine fish	Freshwater fish	Mollusk
Dalian	21	48.33 (70%)	23.45 (91%)	29.25 (75%)
Yingkou	22	42.15 (81%)	30.12 (89%)	33.69 (63%)
Dandong	18	50.12 (76%)	32.15 (93%)	38.47 (68%)
Anshan	23	28.14 (78%)	30.12 (83%)	24.13 (70%)
Panjin	22	35.68 (85%)	25.46 (93%)	30.45 (62%)
Benxi	23	35.36 (69%)	36.15 (85%)	18.53 (65%)
Fushun	21	21.63 (72%)	34.66 (87%)	16.46 (71%)
Shenyang	22	30.87 (80%)	40.35 (88%)	15.11 (67%)
Tieling	18	20.45 (65%)	38.79 (90%)	14.21 (58%)
Jinzhou	21	38.43 (70%)	33.49 (85%)	25.78 (57%)
Huludao	22	33.46 (75%)	32.74 (81%)	30.12 (75%)
Chaoyang	23	24.31 (73%)	35.89 (93%)	13.24 (56%)

Note: Percentages in parentheses are the market share of the species studied in this research

Table 5 Human exposures and benchmark concentrations for contaminants in fish and mollusk

Compound	Species	Estimated daily intake (ng/kg body weight/day)	Noncancer benchmark concentration (ng/kg body weight/day) ^a	Cancer benchmark concentration (ng/kg body weight/day) ^b
HCH	Marine fish	0.33	300	1354
	Freshwater fish	0.74	300	1408
	Marine mollusk	0.21	300	1914
DDT	Marine fish	14.79	500	5178
	Freshwater fish	18.64	500	5383
	Marine mollusk	5.34	500	7316
Chlordane	Marine fish	0.61	500	5030
	Freshwater fish	0.76	500	5230
	Marine mollusk	0.29	500	7107
Endosulfan	Marine fish	0.66	6000	–
	Freshwater fish	0.95	6000	–
	Marine mollusk	0.40	6000	–
HCB	Marine fish	0.26	800	1100
	Freshwater fish	0.45	800	1144
	Marine mollusk	0.24	800	1555

^a Risk values derived from chronic RfD, recommended by the USEPA

^b Benchmark concentration for carcinogenic effects equals 10^{-6} divided by the cancer slope factor represents the exposure concentration at which the lifetime cancer risk is 1 in 1 million

Using the data from the present study, we calculated average EDIs of OCPs via fish and mollusk consumption; OCP concentrations use the mean value of marine fish, freshwater fish, and mollusk samples. As reported in Table 5, DDT clearly had the highest EDI values and also the highest values of OCPs for human exposure. In general, it showed the highest level of human exposure to OCPs through freshwater fish consumption, followed by marine fish and marine mollusks. Potential public health risks from exposure to OCPs by fish and mollusk consumption were assessed using two benchmark concentrations. A benchmark concentration represents the daily concentration below which there is a high probability of no adverse health effect. This is different from a benchmark dose, which is a statistically derived value used in setting a reference dose for noncancer health effects. The first benchmark concentrations were U.S. Environmental Protection Agency (EPA) Reference Dose (RfD) values, which were used for non-carcinogenic effects. The second benchmark concentrations were carcinogenic effects, which were derived using U.S. EPA cancer slope factors and represent exposure concentrations at which lifetime cancer risk is 1 in 1 million (USEPA 2006). The benchmark concentration is an estimate, with an uncertainty spanning perhaps an order of magnitude, of the daily exposure of the human population that is likely to be without appreciable risk of deleterious effects during a lifetime (USEPA 1990). Different benchmark concentrations of OCPs are also reported in Table 5. In this study, none of the EDI values for OCPs exceeded the benchmark concentration, which suggests that these aquatic foods are safe to consume based on their contributions to the EDI of organic contaminants. Despite the risk assessment

results showing that it is safe, the potential health risk associated with consumption of these products cannot be neglected. The widespread distribution and higher concentrations of certain compounds deserve further monitoring efforts to ensure the long-term safety of consumers in association with consumption of these food.

To assess geographical exposure, human exposures were calculated based on cities by association with concentration and consumption data. According to the locations, we divided the 12 cities into inland (including Chaoyang, Anshan, Benxi, Fushun, Shenyang, and Tieling) and coastal (included Dalian, Yingkou, Dandong, Panjin, Jinzhou, and Huludao). In general, there was higher exposure to OCPs by consumption of freshwater fish in inland cities than in

Fig. 3 Human exposure to DDT by consumption of fish and mollusks in different cities in Liaoning province

costal cities, and the results were reversed for consumption of marine fish and mollusks (Fig. 3 presents the human exposure to DDT in 12 cities in Liaoning province), which reflects the different compositions of aquatic products consumed in different cities. We also calculated the total exposure of these foods, and one-way analysis (ANOVA) was used to assess the different exposure between coastal cities and inland cities; at the 0.05 level, there was no significant difference between them. The conclusion was that, in geographical terms, residents of both inland and coastal regions were subjected to the same total exposure to OCPs by consumption of fish and mollusks.

Some limitations associated with exposure analysis can lead to uncertainty in the total risk. For example, our risk analysis did not consider different ages, and childhood exposure may have a greater probability of producing tumors than exposure in adulthood. The use of mean fish and mollusk contaminant concentrations to estimate exposure is another limitation that could underestimate risk if an individual regularly consumes fish and mollusks from a contaminated water body. In addition, human body weight and possible interactions among different toxic chemicals could lead to uncertainties. Our study, however, provides a clear picture of the distribution of OCPs in fish and mollusks and a risk assessment of human exposure in Liaoning province, China.

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