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Article

Persistent organochlorine pollutants in cyprinids from the Lake Khanka (Spassky District, Primorsky Krai, Russia)

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Abstract. Present study provides data of organochlorine pesticides (DDT, HCH) and polychlorinated biphenyls (28, 52, 155, 101, 118, 143, 153, 138, 180, 207 congener) content in six carp fish from Lake Khanka. Organochlorine compounds are found in all studied samples. HCHs isomers were dominant, constituting on average 70% of all pesticides detected. α-HCH content varied from 8 to 95%. Among DDTs, *o,p'*- and *p,p'*-DDE were predominant (on average 10–100 and 15–100%, respectively). PCBs content in all fish samples was primarily presented by low-chlorinated 28 and 52 congeners. Detected isomers evidence on the long-term pollution of Lake Khanka by persistent organic pollutants. Compare to southern China and Croatia, fish from Lake Khanka showed significantly higher levels of accumulation of HCH and lower levels of DDT and PCB. When comparing the data with the requirements of regulatory documents in Russia and China, no cases of exceeding maximum permissible concentrations were identified.

Keywords: OCPs, POPs, PCBs, Cyprinidae, freshwater ecosystems, Amur River basin

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Научная статья

Стойкие хлорорганические загрязняющие вещества в карповых рыбах озера Ханка (Спасский район, Приморский край)

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Аннотация. Приведены концентрации хлорорганических пестицидов (ДДТ, ГХЦГ) и полихлорированных бифенилов (28, 52, 155, 101, 118, 143, 153, 138, 180, 207 конгенеры) в шести видах карповых рыб оз. Ханка. Хлорорганические соединения обнаружены во всех исследованных образцах. Доминирующей группой поллютантов были изомеры ГХЦГ, составляющие в среднем 70% от всех обнаруженных пестицидов. Доля α-ГХЦГ варьировала от 8 до 95%. Среди ДДТ и его метаболитов наиболее определяемым были *o,p'*- и *p,p'*-ДДЕ (в среднем – 10–100 и 15–100% соответственно). ПХБ во всех рыбах были представлены в основном низкохлорированными конгенерами – 28 и 52. По обнаруженным изомерам можно предположить давнее загрязнение оз. Ханка. Сравнение с водоемами южного Китая и Хорватии показало значительно более высокие уровни аккумуляции ГХЦГ и более низкие – ДДТ и ПХБ. При сравнении данных с требованиями нормативных документов России и Китая не выявлено случаев превышения допустимых концентраций.

Ключевые слова: СОЗ, ХОП, ПХБ, Cyprinidae, пресноводные экосистемы, бассейн р. Амур

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Introduction

Universality of ambient processes requires international cooperation for environmental protection. Through such interaction, countries ratificate international agreements and establish transboundary protected areas (Petrova et al., 2019). Lake Khanka is an example of such cooperation given that its' territory has received the status of a transboundary international Russian-Chinese nature reserve since 1996.

Lake Khanka is the largest freshwater habitat in the northeastern Asia. The peculiarities of its location and climate predetermine greatly the productivity of local ecosystems, their biological and genetic diversity (Kozhenkova, 2017). Since the middle of XX century, the Khanka Plain has become one of the main agricultural areas of the Russian Far East and Heilujiang Province of China. Yet, there was a significant decrease in the anthropogenic load on this region associated with shutting down a number of agricultural enterprises by the Russian Federation due to economic depression in the 1990s. Since 2000, there has been observed an increase of economic activity in the area adjacent to the lake. The lake waters are affected by both agricultural sector and the local population (formation of landfills, fishing, exposure to sewage and domestic water, parking and passage of vehicles): according to national census in 2017, the population amounted to a total of 605.8 thousand people (Egidarev et al., 2019; Tsybrinskaya, 2009).

Currently, the ecosystem of Lake Khanka is no longer able to manage the decontamination of the incoming volumes of xenobiotics. The greatest damage to the ecosystem is caused by the uncontrolled usage of pesticides, as well as their improper storage. In 2020, significant concentrations of prohibited organochlorine pesticides (OCPs) were detected in the waters of the lake: these were isomers of hexachlorocyclohexane (HCH), dichlorodiphenyltrichloroethane (DDT) and its metabolites (DDD and DDE), hexachlorobenzene, etc. Moreover, a recent entry of DDT into the lake ecosystem was recorded (Lyagusha and Chernyaev, 2020). However, there is practically no data on the content of POPs in the biota of this reservoir, which makes it difficult to track the dynamics of the concentrations of prohibited compounds. In this regard, both the stability of the local ecosystem and the health of coastal residents are threatened due to the presence of OCPs in the aquatic biological resources of the lake.

Our study aims to determine the content of organochlorine pesticides (DDT and HCH) and polychlorinated biphenyls (PCBs) in the organs and tissues of cyprinids from Lake Khanka.

Materials and methods

Six fish species belonging to the Cyprinidae family were studied: Prussian carp *Carassius gibelio* (Bloch, 1782), Amur carp *Cyprinus rubrofuscus* Lacepède, 1803, Yangtze predatory carp *Chanodichthys oxycephalus* (Bleeker, 1871), Ussuri sharpbelly *Hemiculter lucidus* (Dybowski, 1872), Mongolian redfin *Chanodichthys mongolicus* (Basilewsky, 1855), and predatory carp *Chanodichthys erythropterus* (Basilewsky, 1855) (Table 1). Fish were collected from Lake Khanka near the Novoselskoe village during the expedition by the Pacific Geographical Institute of the Far-Eastern Branch of Russian Academy of Sciences (PGI FEB RAS) in 2018 (Fig. 1). Fish were caught at a 1–2-m depth by fishing rods. Individuals were measured, weighed and frozen intact. All collected samples were transported to the PGI FEB RAS. The fish was washed with distilled water and dissected frozen. The samples were stored in a freezer until chemical analysis for persistent organic pollutants (POPs).

The organs were homogenized to solid blend. POPs were extracted from homogenates with a hexane-acetone mixture, followed by destruction of co-extractive components (lipids) with concentrated sulfuric acid and separated into polar (OCPs) and nonpolar (PCBs) phases on chromatographic columns using activated sorbent Florisil© 100–200 mesh (Donets et al., 2021a; 2021b). Standard samples (Dr. Ehrenstorfer and AccuStandard) of the test compounds (α-HCH, β-HCH, γ-HCH, δ-HCH, *p,p′*-DDT, *p,p′*-DDD, *p,p′*-DDE, *o,p′*-DDT, *o,p′*-DDD, *o,p′*-DDE, dieldrin, endrin and a mixture of PCBs' congeners 28, 52, 155, 101, 118, 143, 153, 138, 180, 207), with established metrological characteristics according to a calibration curve developed in regard to the standard solutions.

OCPs and PCBs were determined using a Shimadzu GC-2010 Plus gas chromatography analyzer with an ECD electron capture detector. Capillary column was Shimadzu HiCap CBP 5 (column length of 25 m, internal diameter of 0.32 mm, phase thickness of 0.5 µm). The analyzer was calibrated using standard POP solutions. Identification was based on relative retention time. Quantitative analysis was performed using the internal standard method leveraging standard mixtures of pesticides (Tsygankov and Boyarova, 2015).

Statistical analysis was carried out using IBM SPSS Statistics software for Windows 10. Significance was checked using Kruskal-Wallis test at $p \leq 0.05$. Values exceeding mean ± 3 SD (standard deviation) were considered outliers not taken for comparative analysis and diagram plotting. However, all extremely high concentrations are indicated in the tables and range list without exception. No indication of standard deviation means there were no repetitions, so this value is not discussed. The predominant toxicants are defined as the form of the compound with the greatest contribution to the total concentration and the highest detection frequency. The greatest qualitative diversity is indicated for compounds based on the number of congeners/forms/isomers found for more than three samples.

Table 1. Test objects' size and age characteristics. The range of concentrations is indicated above the line, and the mean ± standard deviation is indicated below the line.

Species	Length (AB), cm	Weight, g	Age, years	N, ind.
Carassius gibelio	25.0–34.0 29 ± 3	310–560 465 ± 84	4–7	19
Cyprinus rubrofuscus	44.2–53.3 47 ± 4	1375–2215 1525 ± 409	6–8	14
Hemiculter lucidus	21.5–24.7 23 ± 2	60–90 72 ± 16	7–8	6
Chanodichthys oxycephalus	27.5–34.2 31 ± 3	165–335 240 ± 63	7–10	15
Chanodichthys mongolicus	43.5–51.0 47 ± 3	630–1235 877 ± 243	7–10	15
Chanodichthys erythropterus	50.8–65.0 56 ± 8	885–2000 1260 ± 640	7–8	10

Fig. 1. Map of the study area. The star indicates the sampling location (Xiangcan and Pingyanh, 2007).

Results

Organochlorine compounds were detected in all studied samples (Tables 2, 3). The average concentrations of ∑OCP (∑DDT + ∑HCH + endrin + dieldrin) and ∑PCB (sum of 28, 52, 155, 101, 118, 143, 153, 138 and 180 congeners) in all samples varied from 0.12 to 120.3 (8.3 ± 17.8) and from 0.11 to 24.2 (2.8 ± 5.1) ng/g wet weight, respectively, which indicates the predominant contribution of pesticides to reservoir pollution.

Among POPs, HCH isomers were prepotent and constituted on average 70% of all detected pesticides. Among the forms of this compound, the α-isomer predominated (8–95%). In general, based on the detected isomers, we assume that Lake Khanka has been contaminated with this compound for a long time (Saadati et al., 2012). Among DDT and its metabolites, *o,p'*- and *p,p'*-DDE were the most detectable (average of 10–100% and 15–100%, respectively), also indicating historical contamination (Saadati et al., 2012). Dieldrin was below detection limits in all samples studied. Endrin was found only in two samples: these were muscles and eggs of *Carassius gibelio* (Table 2).

PCBs in all fish were mainly represented by low-chlorinated PCBs 28 and PCBs 52 (Table 3).

Carassius gibelio

The muscles, liver and eggs of *C. gibelio* were analyzed (Fig. 2). Among HCHs, all studied forms of the compound were found with a predominance of β- and γ-isomers. DDT was presented only as DDE. Endrin was found sporadically in muscles and eggs. Among PCBs, the most low-chlorinated congeners were determined (28 and 52).

No statistically significant differences were found between the concentrations of pollutants (individual and total), which was probably associated with the fairly large variations in concentrations. However, a trend towards greater accumulation of HCH in the liver, as well as DDT and PCBs in the muscles, is visible. It is likely that HCH is the predominant toxicant entering the fish body.

Table 2. Concentrations of POPs in cyprinids from Lake Khanka (mean ± standard deviation), ng/g wet weight. * – found in one sample; < LD – concentration below the detection limits.

Species	Organ	HCH isomers			
		α-	β-	γ-	δ-
Carassius gibelio	Muscles	0.87 ± 0.87	1.6 ± 2.5	1.5 ± 1.7	0.53 ± 0.59
	Liver	3.2 ± 2.2	0.98 ± 0.60	0.59 ± 0.34	0.37 ± 0.35
	Eggs	19.9 ± 34.4	5.6 ± 10.8	1.8 ± 3.1	6.8 ± 8.5
Cyprinus rubrofuscus	Muscles	1.34 ± 0.86	0.73 ± 0.87	0.26 ± 0.15	0.69 ± 0.83
	Liver	3.5 ± 2.4	0.97 ± 0.55	0.51 ± 0.33	1.1 ± 1.3
	Eggs	2.6 ± 18.2	13.7*	< LD	0.29 ± 0.45
	Soft roe	0.38 ± 0.03	0.28*	0.17 ± 0.06	0.16 ± 0.02
Hemiculter lucidus	Muscles	< LD	< LD	0.88*	< LD
Chanodichthys oxycephalus	Muscles	2.1 ± 3.4	0.44 ± 0.15	0.37 ± 0.17	0.58 ± 0.53
	Liver	33.6 ± 30.3	4.8 ± 5.2	5.2 ± 5.7	2.8*
Chanodichthys mongolicus	Muscles	1.2 ± 1.8	0.35*	0.49 ± 0.32	0.49 ± 0.36
	Liver	6.1 ± 4.9	0.36 ± 0.08	0.46 ± 0.08	0.61*
	Eggs	0.85*	0.29*	0.24 ± 0.07	< LD
	Fat	13.1 ± 13.0	1.7 ± 1.3	1.21 ± 0.79	0.87*
Chanodichthys erythropterus	Muscles	1.4 ± 1.6	1.6 ± 1.2	1.6 ± 1.9	0.17*
	Liver	16.2± 22.0	0.93 ± 0.68	0.74*	3.0*
	Soft roe	3.6 ± 4.6	0.73 ± 0.56	0.69 ± 0.25	0.24*
	Fat	4.1 ± 4.2	7.0 ± 9.1	0.37*	0.23 ± 0.15

DDT isomers		DDD isomers		DDE isomers		Dieldrin	Endrin
o,p′-	*p,p′-*	*o,p′-*	*p,p′-*	*o,p′-*	*p,p′-*		
< LD	< LD	< LD	< LD	0.46 ± 0.61	0.61 ± 0.30	< LD	0.25*
< LD	< LD	0.11*	< LD	0.53 ± 0.32	0.20 ± 0.08	< LD	< LD
< LD	< LD	< LD	0.29*	0.63 ± 0.70	0.93*	< LD	4.4*
< LD	< LD	< LD	0.57 ± 0.20	0.24 ± 0.17	0.45 ± 0.29	< LD	< LD
< LD	3.9*	< LD	< LD	0.76*	< LD	< LD	< LD
14.8*	< LD	< LD	< LD	< LD	< LD	< LD	< LD
< LD	< LD	< LD	< LD	0.12*	< LD	< LD	< LD
< LD	< LD	< LD	< LD	0.45 ± 0.27	< LD	< LD	< LD
< LD	< LD	< LD	< LD	0.30 ± 0.21	0.70*	< LD	< LD
< LD	< LD	< LD	< LD	32.9*	< LD	< LD	< LD
< LD	< LD	< LD	< LD	0.38 ± 0.2	< LD	< LD	< LD
< LD	< LD	< LD	0.97 ± 1.12	0.50*	0.20*	< LD	< LD
< LD	< LD	< LD	< LD	0.79*	< LD	< LD	< LD
< LD	< LD	1.04 ± 0.11	3.89 ± 0.85	< LD	2.9*	< LD	< LD
0.45*	< LD	< LD	< LD	0.34 ± 0.18	0.51*	< LD	< LD
< LD	< LD	< LD	< LD	< LD	0.99*	< LD	< LD
< LD	1.4*	< LD	< LD	2.9*	1.8*	< LD	< LD
< LD	< LD	1.6*	2.2*	0.68*	25.7*	< LD	< LD

Table 3. Concentrations of PCB congeners in cyprinids from Lake Khanka (mean ± standard deviation), ng/g wet weight. * – found in one sample; < LD – concentration below the detection limits.

Species	Organ	PCB 28	PCB 52	PCB 155	PCB 101	PCB 118	PCB 143	PCB 153	PCB 138	PCB 180
Carassius gibelio	Muscles	1.8 ± 2.0	0.75 ± 0.07	< LD	0.31*	< LD	< LD	2.7*	< LD	< LD
	Liver	0.43 ± 0.15	0.25 ± 0.13	< LD	< LD	< LD	< LD	< LD	< LD	< LD
	Eggs	1.0 ± 1.6	0.24 ± 0.19	< LD	0.50*	0.16*	< LD	< LD	0.13*	< LD
Cyprinus rubrofuscus	Muscles	0.48 ± 0.22	0.51 ± 0.20	< LD	< LD	< LD	< LD	0.24*	< LD	< LD
	Liver	0.90 ± 0.40	0.24*	< LD	< LD	< LD	< LD	< LD	< LD	< LD
	Eggs	< LD	0.20 ± 0.17	0.38*	< LD	0.40*	0.14*	< LD	< LD	0.13*
	Soft roe	0.13*	0.5*	< LD	< LD	< LD	< LD	< LD	< LD	< LD
Hemiculter lucidus	Muscles	0.29 ± 0.30	0.50 ± 0.16	< LD	< LD	0.49 ± 0.23	< LD	< LD	0.46 ± 0.48	< LD
Chanodichthys oxycephalus	Muscles	0.51 ± 0.28	0.46 ± 0.30	< LD	< LD	< LD	< LD	< LD	< LD	< LD
	Liver	1.8*	2.3*	< LD	< LD	1.4*	< LD	2.0*	2.3*	< LD
Chanodichthys mongolicus	Muscles	0.25 ± 0.02	0.74 ± 0.32	< LD	< LD	0.51*	< LD	0.45*	< LD	< LD
	Liver	1.5*	0.39*	< LD	< LD	< LD	< LD	< LD	< LD	< LD
	Eggs	0.49*	< LD	< LD	< LD	< LD	< LD	< LD	< LD	< LD
	Fat	3.17 ± 0.02	1.15 ± 0.81	< LD	1.03 ± 0.13	1.54 ± 0.69	1.73*	2.18 ± 0.73	2.3 ± 0.5	< LD
Chanodichthys erythropterus	Muscles	0.23*	0.43 ± 0.23	< LD	< LD	0.89*	< LD	0.33*	0.27*	< LD
	Liver	0.81 ± 0.45	1.5 ± 1.3	< LD	0.65*	1.3*	< LD	3.20*	1.3*	< LD
	Soft roe	1.7*	0.54*	< LD	< LD	< LD	< LD	1.80*	1.7*	< LD
	Fat	1.43 ± 0.78	1.08 ± 0.57	< LD	2.8 ± 1.4	5.7 ± 1.4	< LD	4.2 ± 4.3	8.77 ± 0.49	< LD

Cyprinus rubrofuscus

The muscles, liver, eggs and male gonads of *C. rubrofuscus* were analyzed (Fig. 3). HCH isomers were predominant among all detected POPs with a prevalence of the most stable α- and β-forms. DDT and its metabolites were represented mainly by *o,p'*- and *p,p'*-isomers DDE and DDD, which were found mainly in fish muscles. In some cases, original DDT was detected in the liver and eggs.

No significant differences were found for the content of the studied pollutants in the organs of *C. rubrofuscus*. At the same time, there is a tendency to higher HCH accumulation in the eggs and DDT accumulation in the liver. The distribution of PCBs across organs was quite homogeneous in muscle–liver and eggs–soft roe pairs.

Hemiculter lucidus

Among POPs, only *o,p'*-DDE and γ-HCH (lindane) were detected (in one sample). PCBs had great heterogeneity in their qualitative composition: along with low-chlorinated congeners 28 and 52, PCBs 118 and 138 were found. Statistical analysis was not applied due to the study of only one type of organ of *H. lucidus*.

Chanodichthys oxycephalus

HCH was dominant in both organs studied (Fig. 4). Among its isomers, the α-form predominated. DDT in muscles and liver was almost entirely present by *o,p'*-DDE. Among PCBs, congener 28 was predominantly found in muscles, and only some representatives of these pollutants were found in liver (all in one sample only).

In the liver of *C. oxycephalus*, the total content of α-, β- and γ-HCH was significantly ($p \leq 0.05$) higher than in the muscles (Fig. 4). For other pollutants, only a similar trend was observed. It is also worth noting the slightly higher total concentrations of HCH in the liver of *C. erythropterus* compared to other fish.

Chanodichthys mongolicus

Muscle, liver, eggs and visceral fat were analyzed in *C. mongolicus* (Fig. 5). All studied organs were characterized by the predominance of α-HCH over other forms of the compound. In muscles, only DDE

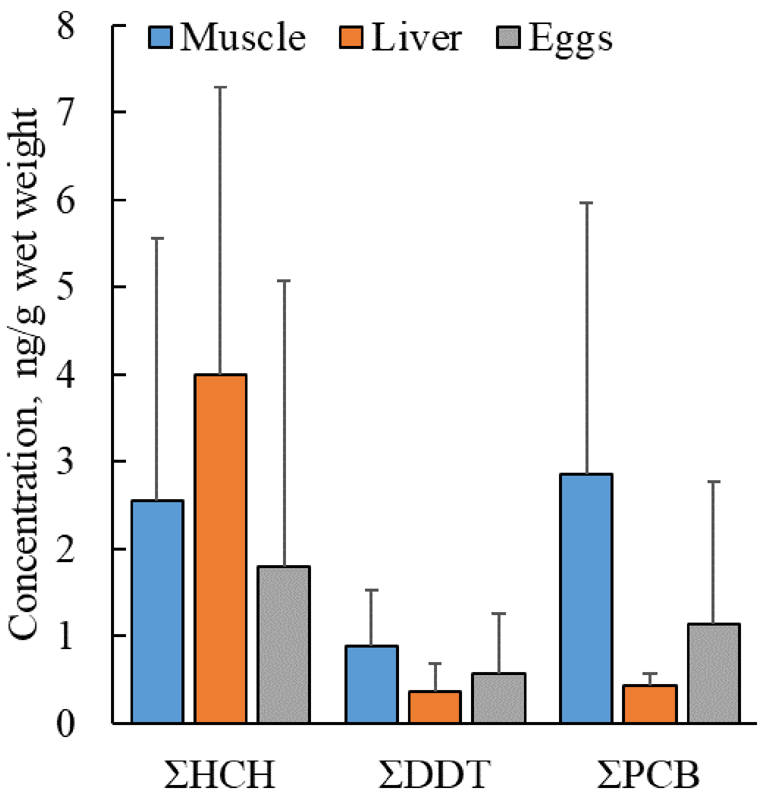

Fig. 2. Average concentrations of POPs in the organs of *Carassius gibelio* (here and after: vertical bars indicate standard deviation).

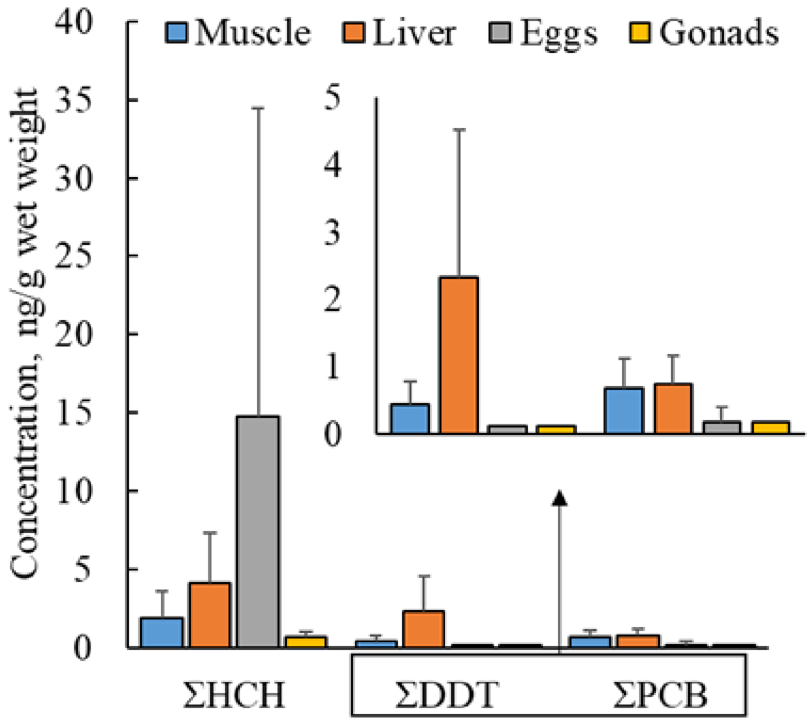

Fig. 3. Average concentrations of POPs in the organs of *Cyprinus rubrofuscus*.

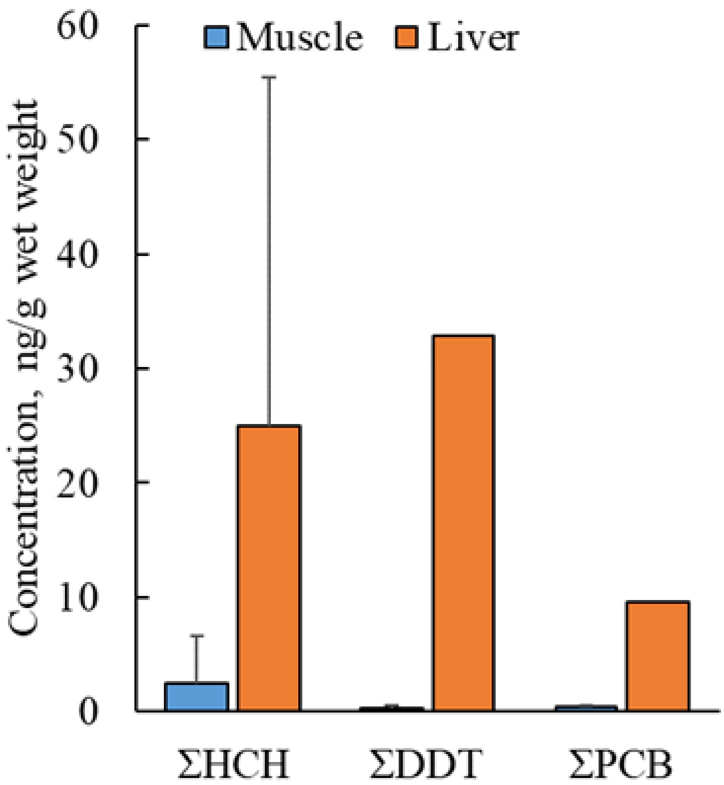

Fig. 4. Average concentrations of POPs in the organs of *Chanodichthys oxycephalus*.

was found among DDT metabolites, in liver and fat, DDD and DDE, in eggs, *o,p'*-DDE (singly). Similar to other studied fish species, PCBs in *C. mongolicus* were predominantly represented by 28 and 52 congeners, except in fat. The highest variability and concentration of toxicants were found in visceral fat, which contained a large amount of lipids (Fig. 5).

Despite the variable composition of pollutants in the studied organs, no significant differences in the concentrations of xenobiotics were found. There is only a tendency for toxicant concentrations to increase in the row fat > liver > eggs ~ muscles.

Chanodichthys erythropterus

Muscles, liver, male gonads, and visceral fat of *C. erythropterus* were collected for the analysis (Fig. 6). Among HCH isomers, the α-form predominated. β- and γ-HCH levels were similar in muscle and male gonads; in addition, the γ-form was detected only sporadically in the liver. This may indicate a chronic effect of pollutants on these fish. Among DDT and its metabolites, decomposition products of the original compound (DDD, DDE) were often identified.

No significant differences between the concentrations of pollutants were found in the organs of *C. erythropterus*. There was a tendency towards greater accumulation of DDT and PCBs in the fat of this fish, and HCH in the fat and liver (Fig. 6).

Discussion

Differences in the accumulation of POPs in the studied fish

C. gibelio is a typical euryphagous fish, widely distributed throughout the world. This species is resistant to extreme conditions, such as low oxygen levels, high acidity, etc. (Erdogan et al., 2014; De Giosa et al., 2014; Promyslvye..., 1949). *Carassius gibelio* is able to change its food specialization when faces tough competition for a specific resource (Carassius..., 2021). Typically, this species feeds mainly on detritus and benthos (Bugaev et al., 2007), which may contribute to a higher accumulation of POPs in the fish body preconditioned by concomitant ingestion of bottom sediments.

Cyprinus rubrofuscus and *Carassius gibelio* are the closest in their biological characteristics. They are competitors due to their similarity in type of feeding and prey items. Benthic organisms constitute a substantial part in *Cyprinus rubrofuscus*'s food spectrum, specifically, chironomid larvae (Burik, 2010). In addition, *Cyprinus rubrofuscus* prefers soft silty soils, saturated with organic matter and absorbing pollutants well. Therefore, a probable source of POPs for these carps might be associated with the concomitant ingestion of contaminated sludge particles during feeding.

In Russia, *Hemiculter lucidus* is rarely exploited as a commercial species. At the same time, in China, closely related species are an important object of commercial fishing. Its diet is based on small crustaceans, mollusks, insect larvae, mayflies, plant seeds, and blue-green algae (Promyslovye..., 1949). Due to their short life cycles, all these organisms are slightly susceptible to the effects of POPs that circulate for a long time in the environment. Accordingly, they accumulate pollutants either due to exposure to atmospheric transport or recent use. In this case, toxicants entering the ecosystem settle on organic particles (and are subsequently ingested) or are sorbed on the surface of the feeding objects of *Hemiculter lucidus*. Due to its ecological characteristics, this species lives mainly in the pelagic zone of the reservoir, which significantly reduces contact with contaminated bottom sediments (unlike crucian carp and common carp). This is indicated by low levels of POPs in the muscles of the belly and their qualitative composition (*o,p'*-DDE, individual PCB congeners). It is likely that atmospheric transport does not have a significant effect on the content of POPs in Lake Hanka.

Chanodichthys oxycephalus is a typical predator. Its food spectrum consists of shrimp, mysids, daphnias, and small fish (especially *Hemiculter lucidus*) (Berg, 1949). Therefore, it has no close contact with bottom sediments, and the accumulation of pollutants occurs due to biomagnification, e.g., the process of increasing the concentration of a chemical substance in the tissues of organisms along the trophic chain (primarily when one organism is eaten by another) (Anderson et al., 2008). As a result of this process, the actual levels of pollutants accumulation exceed those expected under specific conditions (Alexander et al., 1999), and toxicants are again and again in the matter cycle in the ecosystem, even without external introduction (due to the release of pollutants from the carcasses of dead fish).

Like most representatives of genus *Chanodichthys*, *C. mongolicus* is a predator feeding on crustaceans, small fish, and eggs of other cyprinids (especially *Cyprinus rubrofuscus* or common carp).

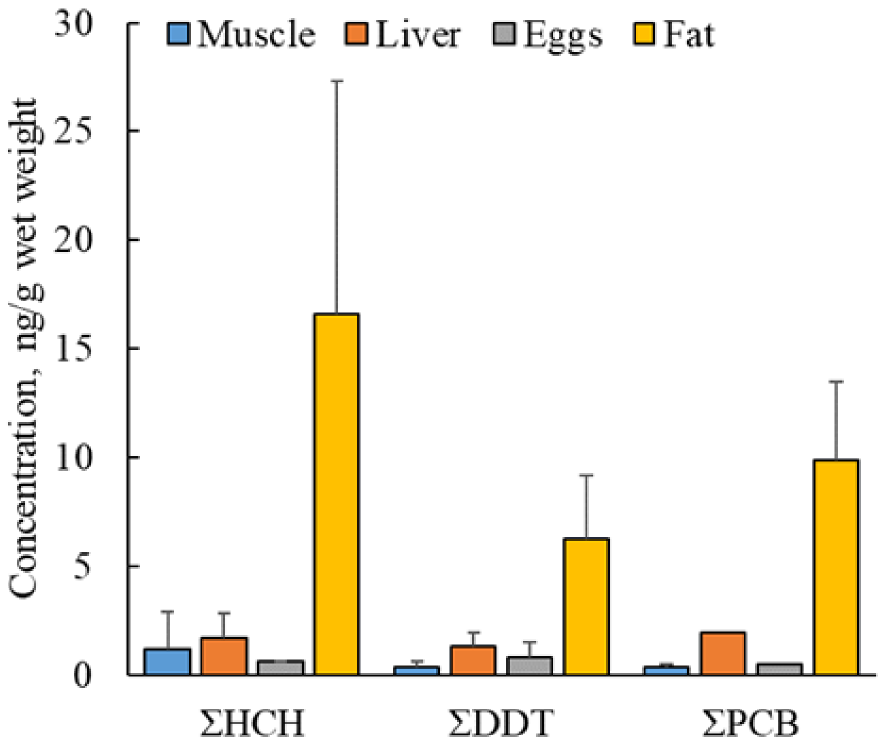

Fig. 5. Average concentrations of POPs in the organs of *Chanodichthys mongolicus*.

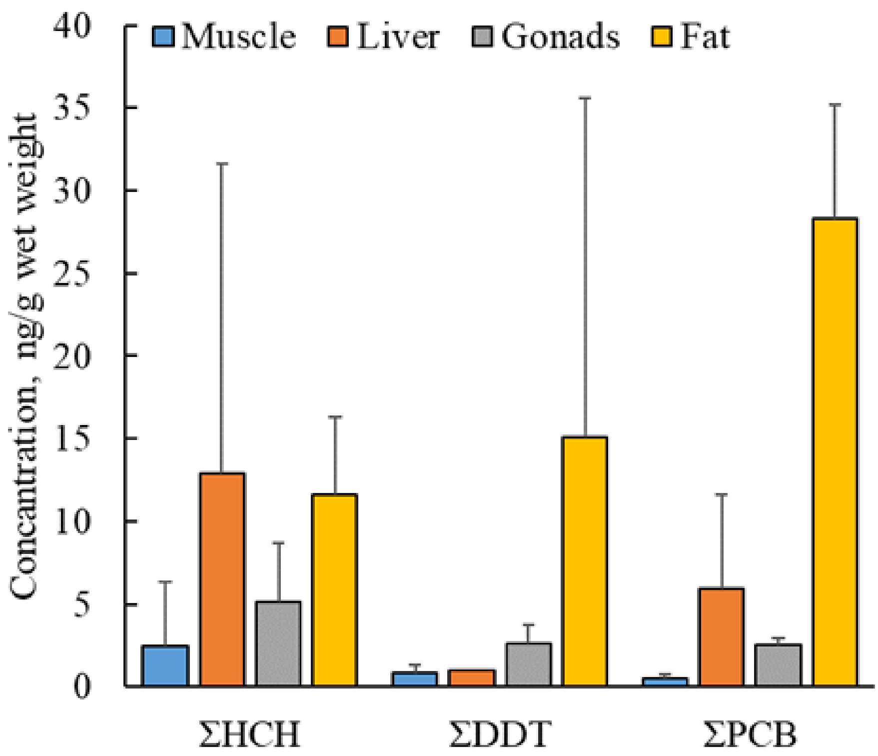

Fig. 6. Average concentrations of POPs in the organs of *Chanodichthys erythropterus*.

Upon reaching age of 5 years, it switches to feeding exclusively on fish (Promyslovye..., 1949). The widest range of studied compounds was found in the organs of these fish, especially in visceral fat, along with *Chanodichthys erythropterus*, which reflects the biomagnification properties of POPs.

In terms of its feeding spectrum, *Chanodichthys erythropterus* is very similar to *Chanodichthys mongolicus*: it hunts mainly the juveniles of the other fish. The found pollutants show the greatest qualitative diversity, especially of PCB congeners.

When comparing the levels of xenobiotics in all studied fish, we have only identified a tendency towards greater accumulation of PCBs in the muscles of *Carassius gibelio* and HCH in the liver of *Chanodichthys oxycephalus* (Fig. 7). POPs are able to enter ecosystems through various pathways. Their qualitative composition depends on the source, which makes it possible to distinguish between recent use and secondary pollution due to long-term circulation of pollutants (including within the framework of atmospheric transfer). If HCH is recently introduced into the ecosystem, the dominant form of the compound is lindane (γ-HCH). During its degradation, this substance undergoes a series of transformations of one isomer into another ($\gamma \rightarrow \alpha \rightarrow \delta \rightarrow \beta$) increasing the chemical stability in the ecosystem (Lu et al., 2019). Thus, the ratio of decomposition products and the original compound may indicate the persistence of the pollutant circulation in the environment. When the concentration of the original substance is greater than its breakdown products, recent use of POPs can be confirmed. For the studied fish, the ratio (α-+β-HCH)/γ-HCH exceeds 1.0, which indicates that this pesticide has been recently introduced. DDT is the main active ingredient in pesticide preparations based on it (Saadati et al., 2012). Under aerobic conditions, this compound is converted to DDE; under anaerobic conditions, it is converted to DDD, which can also be transformed into DDE (Huang et al., 2018). The (DDD+DDE)/DDT ratio in the studied samples exceeds 1.0, which indicates long-term circulation of this toxicant.

Therefore, based on the qualitative and quantitative composition of POPs in fish organs, only the indicators of long-lasting POP contamination were observed in Lake Khanka 2018. A single determination of initial POP compounds does not indicate the existence of sources of original substances entry into the lake's ecosystem.

Possible sources of POPs entering the waters of Lake Khanka

According to Lake Khanka's water quality research held in 2020, the concentration ranges of γ-HCH and DDT were 24.9–49.6 and 3.0–14.9 ng/L, respectively (MPC of 1 and 10 ng/L, respectively) (Lyagusha and Chernyaev, 2020). This means that HCH entered the ecosystem a long time ago, while DDT can be used most likely at the present time, despite the ratification of the Stockholm Convention[1] by Russia and China. In addition, significant excess of MPC of DDT and HCH were found in the rivers feeding the lake, for example, in the Astrakhanka River (by 315 and 38 times, respectively). Unlike Lake Khanka, the release of HCH into the river is believed to be recent and may indicate a dispersion from storage of this banned compound or from its intentional use in agriculture. There is also an unimpeded flow of water from cultivated areas into the river network that feeds the lake. As a result, in some rivers entering the Lake Khanka basin, only the parent compound is found among DDT metabolites ranging from 43.7 to 61.9 ng/L. At the same time, information on the content of POPs in the northern part of the lake (Chinese territory) is virtually absent.

Heilongjiang Province is one of the central rice growing regions in China. At the same time, the main production of agricultural products is carried out here by small farming enterprises, which independently make decisions about the planted and processing methods. This promotes the use of prohibited pesticides due to the transfer of knowledge from older generations, who still experience the active use of POPs (Yang et al., 2007). Up to date, works by Chinese authors (Hu et al., 2019) note a significant overabundance of pesticides, but do not specify their qualitative composition. On average, pesticide applications for rice cultivation in northeastern China are 1.2–2.3 times higher than required (Sun et al., 2020). Thus, DDT and HCH could potentially be utilized by farmers' households in Heilongjiang Province and thus be released into the waters of Lake Hanka.

In all studied samples of fish, low-chlorinated congeners (28 and 52) of polychlorinated biphenyls dominated. This may indicate the predominant intake of PCBs as a result of the navigation activities of various vessels (UNEP, 2001; Urbaniak, 2007). Higher chlorinated congeners make an insignificant contribution to the overall POPs pollution of Lake Khanka and most likely practically do not enter the reservoir.

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[1] UNEP, 2001. Stockholm Convention on persistent organic pollutants. URL: http://chm.pops.int/ (accessed: 13.05.2020).

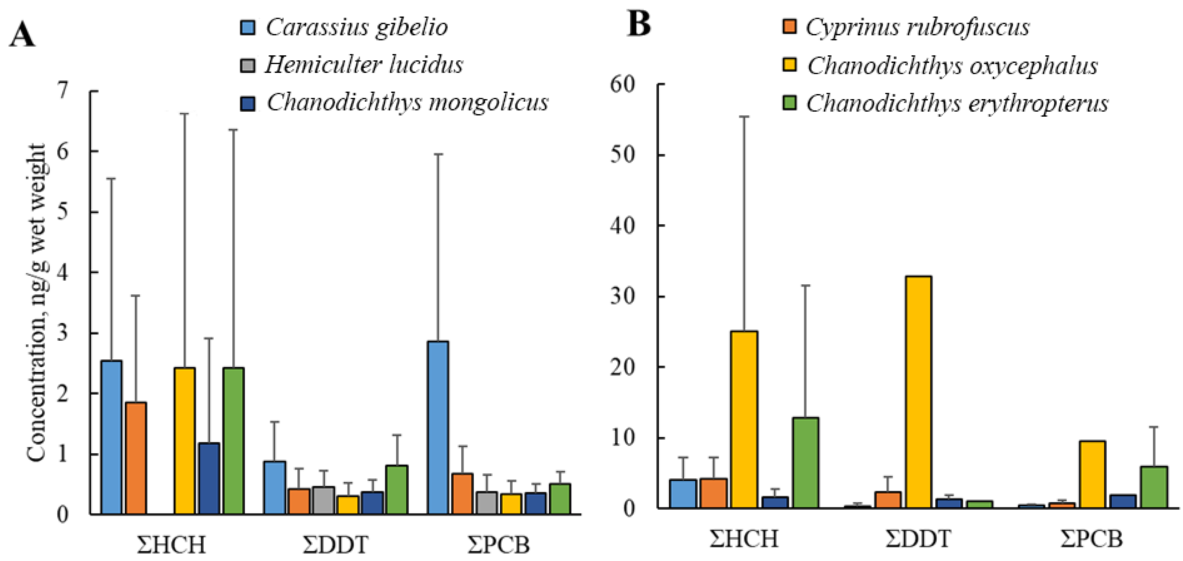

Fig. 7. Comparison of the POPs total concentrations of in the muscles (**A**) and liver (**B**) of the fish studied.

Comparison of POP concentrations in fish organs with other regions of the world

When comparing POPs levels in muscles of the studied fish and the data from other regions, significantly higher levels of HCH are registered in the samples from Lake Khanka (Table 4). The only exception is Lake Dianshan (China), which is exposed to strong POPs contamination due to combined pollution with the discharge of industrial and domestic wastewater, aquaculture and ship transport (Yang et al., 2019). At the same time, the DDT content is generally lower compared to other countries (Wang et al., 2012; Yang et al., 2019; Yu et al., 2012; Zhang et al., 2014).

The high levels of DDT accumulation in fish in southern China might be explained by the use of this pesticide to control vectors of dangerous diseases (malaria, typhus, and scabies), the widespread utilization of this pesticide to kill agricultural pests, and its production in the country in the past (Grung et al., 2015; Li et al., 2018).

High accumulation of HCH in fish from Lake Khanka might be associated with more extensive use of this pesticide in the southeastern part of Russia and the northwestern part of China compared to other regions. Remarkably, HCH levels are generally higher than that of DDT at the Pacific Asian coast and in adjacent seas, which probably reflect the effects of global atmospheric transport and the physicochemical characteristics of these pesticides (Donets et al., 2021a, b; Lukyanova et al., 2018; Tsygankov et al., 2016).

Average concentrations of polychlorinated biphenyls in *Carassius gibelio* and *Cyprinus rubrofuscus* from Lake Hanka are higher than in closely related species from Lake Taihu, China (Wang et al., 2012; Yu et al., 2012) and Lake Vransko, Croatia (Romanić et al., 2018), but lower than in Lake Dianshan, China (Yang et al., 2019). In other cases, PCB concentrations in examined fish samples were lower than those in other regions. We assume that in the studied region, PCBs are supplied in extremely limited quantities, and bottom sediments are the main source of their release nowadays.

POP concentrations and the requirements of regulatory documentation in Russia and China

Hygienic requirements for the content of POPs and PCBs in fish and non-fishery species are presented in Table 5. In Russia, the regulated POPs are *p,p′*-isomers of DDT, DDD and DDE, α-, β-, and γ-isomers of HCH, indicator congeners of PCBs (28, 52, 101, 118, 138, 153, 180) and dioxins (TR CU 021/2011, 2011[2]; TR EAEU 040/2016, 2016[3]). At the same time, China also takes into account δ-HCH, *o,p′*-isomers of DDT and its metabolites, the same PCB congeners, and more than 200 types of pesticides included in the POPs group (GB 2763-2016, 2016[4]; GB 2762-2017, 2017[5]).

When comparing obtained concentrations and MPC requirements adopted in Russia and China, no cases of excess have been found. This indicates the relative safety for consuming fish from Lake Khanka. For a more detailed and accurate analysis, it is necessary to assess the risk to human health and identify safe consumption levels of fish from this reservoir.

Table 4. Average concentrations of POPs in the muscles of cyprinids from various lakes. [1]Sum of α-, β- and γ-HCH; [2]sum of *p,p'*-DDE, *p.p'*-DDD and *p,p'*-DDT; [3]sum of indicator PCBs (28, 52, 101, 138, 153, 180); "–" – no data.

Species	Reservoir	Year of sampling	Concentration, ng/g wet weight			References
			∑HCH	∑DDT	∑PCB	
Carassius carassius	Lake Taihu	2009	0.77	9.75	1.08	Wang et al., 2012; Yu et al., 2012
Carassius gibelio	Lake Vransko	2014	0.19[1]	0.87[2]	1.15[3]	Romanić et al., 2018
	Lake Khanka	2018	2.92	0.81	2.95	Present study
Carassius auratus	Lake Nansihu	2011	1.90	2.61	–	Zhang et al., 2014
	Lake Dianshan	2016	10.1	10.6	16.4	Yang et al., 2019
Cyprinus carpio	Lake Taihu	2009	0.51	6.56	0.58	Wang et al., 2012; Yu et al., 2012
	Lake Nansihu	2011	2.46	3.58	–	Zhang et al., 2014
	Lake Vransko	2014	0.23[1]	1.37[2]	2.56[3]	Romanić et al., 2018
	Lake Dianshan	2016	8.20	12.7	15.3	Yang et al., 2019
Cyprinus rubrofuscus	Lake Khanka	2018	2.15	1.07	0.79	Present study
Chanodichthys mongolicus	Lake Taihu	2009	0.59	4.90	1.17	Wang et al., 2012; Yu et al., 2012
	Lake Khanka	2018	1.63	0.38	0.80	Present study
Chanodichthys erythropterus	Lake Taihu	2009	0.55	9.50	1.59	Wang et al., 2012; Yu et al., 2012
	Lake Dianshan	2016	7.80	8.70	17.0	Yang et al., 2019
	Lake Khanka	2018	1.63	0.38	0.80	Present study

[2] TR CU 021/2011, 2011. Technical regulations of the Customs Union “On the safety of food products”.

[3] TR EAEU 040/2016, 2016. Technical regulations of the Eurasian Economic Union “On the safety of fish and fish products.”

[4] GB 2763-2016, 2016. National food safety standard for Maximum Residue Limits for Pesticides in Foods.

[5] GB 2762-2017, 2017. National Food Safety Standard for Maximum Levels of Contaminants in Foods.

Table 5. POP and PCB content hygienic requirements for freshwater fish safety in Russia and China (MPC), ng/g wet weight.

Country	Pollutant	MPC	Note	References
Russia	HCH	30	All types of freshwater fish products, except liver, eggs, soft roe, cod liver oil, dried and other ready-to-eat products	TR CU 021/2011, 2011; TR EAEU 040/2016, 2016
		200	Fish eggs and soft roe and products	
		1000	Fish liver and products	
	DDT and its metabolites	300	All types of freshwater fish products, except liver, eggs, soft roe, cod liver oil, dried and other ready-to-eat products	
		3000	Fish liver and products	
		200	Cod liver oil	
		400	Fish eggs, soft roe and products	
	PCB	2000	All types of fish products (except liver and cod liver oil)	
		5000	Fish liver and products	
		3000	Cod liver oil	
China	DDT	500	Products from aquatic organisms	GB 2763-2016
	HCH	100	Products from aquatic organisms	
	PCB	500	Aquatic organisms and products	GB 2762-2017

Conclusions

Despite the international protected status of Lake Khanka, its basin is subjected to anthropogenic impact. The area around the reservoir is abundantly used for agriculture. At the same time, there is an excessive utilization of pesticides by China and the probable usage of prohibited compounds in Russia. In general, based on the qualitative composition of POPs in the fish from Lake Khanka, long-standing pollution of the reservoir has been recorded in 2018. Significant levels of HCH were found in the studied samples, their concentrations exceeded those in fish from water bodies of southern China and Europe. At the same time, the DDT content in fish from Lake Khanka was generally lower than in compared areas. Polychlorinated biphenyls, reflecting anthropogenic pollution, tended to accumulate less than POPs, indicating the influence of agricultural activities. At the same time, PCBs accumulated in higher quantities in fish ingesting silt while feeding being a benthos species. It is also worth noting that biomagnification is one of the main sources of POPs entering fish organisms. When comparing the obtained data with the requirements of regulatory documents of Russia and China, no cases of exceeding MPC were identified. It is necessary to reduce the anthropogenic impact on the lake waters and to identify the sources of prohibited substances.

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