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The Occurrence and Spatial Distribution of Organophosphorous Pesticides in Chinese Surface Water

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Abstract The organophosphorous pesticides (OPPs) contaminations have been reported to occur in some Chinese waters. To describe the contamination status and the spatial distribution of OPPs in the surface water throughout China, samples were collected from over 600 sites located in the surface water of seven major river basins and three main internal rivers drainage areas during 2003 and 2004. The surface water samples were analyzed for the representative OPPs including dichlorvos, demeton, dimethoate, methyl parathion, malathion and parathion. In general, the most frequently detected compound was dichlorvos and demeton, being detected in 89.1% of samples (mean = 17.8 ng/L; range <1.4–1,552.0 ng/L) for dichlorvos, and 78.2% of samples (mean = 35.4 ng/L; range <1.5–2,560.0 ng/L) for demeton. While the detection frequencies of the dimethoate, methyl parathion, malathion, and parathion were all less than 50% in all the surface water samples. Measured concentrations for the six compounds were low and rarely exceed the environment quality standard for surface water of China. The six OPPs compounds were more frequently detected at much higher concentrations in the rivers of north China compared with those of south China. The results of this investigation indicate that OPPs contamination in the Yellow River, Huaihe River, Liaohe River, and Haihe River basins of north China should be of particular concern. When compared with other regions of the world, it appears that the Chinese surface water is some moderately polluted by dimethoate, methyl parathion, malathion, parathion and contaminated by dichlorvos and demeton in certain degree.

Keywords Surface water · China · Dichlorvos · Malathion · Dimethoate

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Over the past few decades, several hundred pesticides of different chemical nature are currently used for agricultural purposes all over the world. It is well known that most of the applied pesticides are subject to many transport and conversion products. Thus, they do not remain at their target site but often enter aquatic environment via soil percolation, air drift or surface runoff, the surface water contamination may have ecotoxicological effects for aquatic flora and fauna as well as for human health if used for public consumption (Forney and Davis 1981; Mulla and Mian 1981; Leonard 1988; Miyamoto et al. 1990), affecting abundance and diversity of non-target species producing complex effects on the ecosystems and altering tropic interactions (Rand et al. 1995). Because of their widespread use, they are detected in various environmental matrices, such as soil, water and air. Pesticides are divided into many classes, of which the most important is organophosphorous compounds. OPPs are among the most potent insecticides known and have been used throughout the world to control pests in agricultural crops, forests, and wetlands for more than four decades (Smith 1987). Their wide use is due to high toxicity and rapid environmental degradation (Eto 1974). Unfortunately, OPPs lack target specificity and can cause severe and persistent population effects on aquatic non-target species, particularly invertebrates (Fulton and Key 2001; Schulz and Liess 1999; Christopher et al. 2001).

As the world's second largest pesticide manufacturer, it has been estimated that China produced 203,000–381,000 metric tons annually of technical-grade pesticides between 1985 and 1996(Huang et al. 2000). The magnitude of contamination of these pesticides pollutants in surface water, however, had not been systematically investigated at the national scale of China. In order to determine the contamination status and the spatial distribution of organophosphorous pesticides in China's surface water, methyl parathion, malathion, parathion, dichlorvos, demeton and dimethoate were chosen as the representative compounds for analysis. Because the six compounds had been used widely in China as the main organophosphorous pesticides, and the analysis results of them can more correctively characterize the contamination status of organophosphorous pesticides, adding their the toxicity and ecological effects, the six compounds were chosen as representative to characterize the pollution status of organophosphorous pesticides in Chinese surface water. Samples collected from over 600 sites in seven major rivers basins and three internal rivers drainage areas were analyzed for methyl parathion, malathion, parathion, dichlorvos, demeton and dimethoate.

Materials and Methods

Little data were available on the occurrence of methyl parathion, malathion, parathion, dichlorvos, demeton and dimethoate at the outset of this investigation, therefore, the sampling sites was chosen randomly along the individual river basin. The 623 sites sampled during 2003–2004 (Fig. 1), including 217 reservoirs and 406 rivers and lakes. Samples were collected from the seven major river basins, including the Yangtze River basin, the Yellow River basin, the Pearl River basin, the Songhua River basin, the Liaohe River basin, the Haihe River basin and the Huaihe River basin. Samples were also collected from rivers in southeast, northwest and southwest internal rivers drainage areas. The global positioning system (GPS) was used to locate the sampling positions. The sites map distribution is shown in Fig. 1.

All samples were collected by hydrological bureau personnel using consistent protocols and procedures designed to obtain a representative sample using standard depth and width integrating techniques (Shelton 1994). At each site, a composite water sample was collected from about 4 to 6 vertical profiles. Water was passed through a 0.45 μm, baked, glass-fiber filter in the field where possible, or else filtration was conducted in the laboratory. Water samples for chemical analysis were stored in pre-cleaned-amber, glass bottles and collected in duplicate. The duplicate samples were used for backup purposes (incase of breakage of the primary sample) and for laboratory

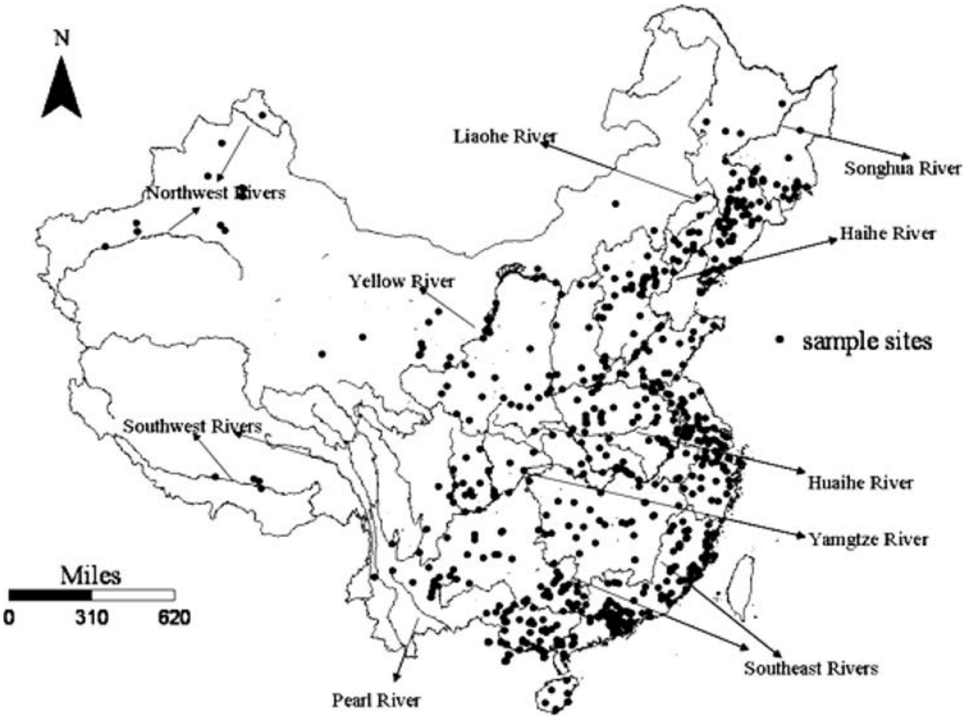

Fig. 1 Map of sampling sites distribution

replicates. Following collection, samples were immediately chilled and sent to the laboratory.

Aliquots of the sample (5.0 L), which were filtered through a 0.45 μm glass fiber membrane under vacuum and then surrogates (simazina-D_5) were added before extraction, were extracted by solid phase extraction (SPE) following published procedures (Zhou et al. 2002; Zhang et al. 2002a, b). Briefly, the SPE cartridges were first conditioned with 10 mL of methanol followed by 2 × 5 mL of deionized water. Water samples were passed through the cartridges at a flow rate of 6 mL/min under vacuum and OPPs are then eluted by passing 10 mL of ethyl acetate through the cartridge, making it possible to pass these compounds from the water phase to an organic phase, residual water was removed by anhydrous Na_2SO_4. The extracts obtained were concentrated with a N_2 flow until an approximate volume of 0.5 mL is reached, Anthracene deuterade (D_{10}) (SUPELCO) is then added to extract for subsequent quantification of OPPs present in the samples. These extracts are analyzed by GC/MS.

All solvents used for sample processing and analysis (ethyl acetate, hexane, acetone methanol) were of HPLC grade. Deionized water was prepared using a Milli-Q system (Millipore, Watford). Chemical standards of OPPs were obtained from Supelco. Working standards were prepared in ethyl acetate and the internal standard Anthracene deuterade (D_{10}) was added to each working standard. These solutions were further diluted with ethyl acetate to prepare calibration solutions.

An Agilent 6890 GC coupled to a model 5973N MS was used for methyl parathion, malathion, parathion, dichlorvos, demeton and dimethoate analysis. Peak confirmation was achieved with an Agilent 6890 GC coupled to a model 5973N MS detector in selected ion mode. The capillary column used was HP-5MS (30 m × 0.25 mm i.d. × 0.25 μm film thickness). All the instruments and capillary column were from Agilent company of America. The carrier gas was helium for MS. The inlet was heated to 220°C. The GC column temperature was programmed as follows: initially at 60°C (equilibrium time 1 min), increased to 140°C at the rate of 10°C/min, then to 230°C at 5°C/min before reaching at 260°C at the rate of 10°C/min and then held for 10 min. The MS temperature was set at 280°C and the electron impact energy was 70 eV.

The residue levels of OPPs were quantitatively determined by the internal standard method using peak area. The method detection limits (MDLs) of OPPs were taken to be 3:1 signal versus noise value (S/N). For every set of 10 samples, a procedural blank and a spiked sample with standards were run to check for the interference and cross-contamination. Table 1 illustrated the Mean recoveries, MDLs and relative standard derivation (RSD%) of the method. The MDLs ranged from 0.8 ng/L for malathion to 1.5 ng/L for demeton. The spiked recoveries of OPPs using 1 ng of composite standards were in the range of 64.6%–102.4% with RSD% values ranging from 2.5% to 7.2%. These parameters confirmed the suitability of the analytical protocols. A field quality assurance protocol was used to determine the effect, if any, of field equipment and procedures on the concentrations of OPPs in water samples. Field blanks, made from laboratory-grade organic free water, were submitted for about 5% of the sites and analyzed for OPPs. Field blanks were subject to the same sample processing, handling, and equipment as the water samples.

Table 1 Recoveries, method detection limits (MDLs), and the relative standard derivation (RSD) of the analytical procedure

Pesticides	Recovery (%)	MDLs (ng/L)	RSD (%) (n = 6)
Dichlorvos	68.4–89.8	1.4	7.2
Demeton	64.6–88.7	1.5	6.3
Dimethoate	72.4–90.3	1.3	2.5
Methyl parathion	68.5–101.6	0.9	6.3
Malathion	76.7–96.5	0.8	4.2
Parathion	70.8–102.4	0.9	5.9

Values for OPPs lower than the method detection limits (<MDL) were substituted with zero prior to statistical analysis. To detect the difference of OPPs concentrations between the north China and south China, the student t-test was employed in this study. Software from Excel Statistics was used in this study.

Results and Discussion

One or more OPPs were found in 91.2% of the 623 sample sites sampled for this study. The reason of the high overall frequency of detection for the OPPs is likely that the six compounds chosen were widely used in China as the main organophosphorous pesticides. In all the surface water samples collected, the detection rates of dichlorvos, demeton, dimethoate, methyl parathion, malathion and parathion were 89.1%, 78.2%, 37.0%, 29.7%, 43.5% and 28.6%, respectively (Tables 2, 3). From the data results, it should be known that the detection rate of dichlorvos was the highest and that of the parathion was the lowest. The mean values of the dichlorvos and demeton were 17.8 ng/L and 35.4 ng/L, with a concentration range from <1.4 to 1,552.0 ng/L for dichlorvos and from <1.5 to 2,560.0 ng/L for demeton. The detection rates of the other four compounds were less than 50%, so the statistical mean values can not be achieved. Among the six organophosphorous pesticides, the detection frequencies of dichlorvos (89.1%) and demeton (78.2%) were the highest, Which indicated

Table 2 The summary results of dichlorvos, demeton and dimethoate concentration statistical analysis

Basins	Sample number	Dichlorvos				Demeton				Dimethoate			
		Rate %	Min	Mean	Max	Rate %	Min	Mean	Max	Rate %	Min	Mean	Max
Songhuajiang River	40	17.5	<1.4	-	20.0	17.5	<1.5	–	110.0	22.5	<1.3	–	180.0
Liaohe River	58	70.7	<1.4	14.5	70.0	70.7	<1.5	122.1	2,560.0	44.8	<1.3	–	480.0
Haihe River	39	100	10.0	25.6	50.0	100.0	<1.5	70.8	50.0	66.7	<1.3	42.8	220.0
Yellow River	50	100	10.0	40.7	140.0	98.0	<1.5	78.9	230.0	68.0	<1.3	91.0	2,660.0
Yangtse River	150	100	2.0	17.9	1,552.0	92.7	<1.5	17.5	254.0	26.0	<1.3		16.0
Huaihe River	39	100	2.0	25.2	140.0	92.3	<1.5	41.5	200.0	59.0	<1.3	34.6	280.0
Pearl River	150	91.3	<1.4	5.6	24.0	74.0	<1.5	9.1	124.0	25.3	<1.3		28.8
Southeast drainage area rivers	74	97.3	<1.4	9.4	86.0	75.7	<1.5	20.7	358.0	23.0	<1.3	–	9.6
Northwest drainage area rivers	18	100	10.0	27.6	70.0	100.0	20.6	104.6	320	44.4	<1.3	–	110.0
Southwest drainage area rivers	5	0	–	–	–	0	<1.5	–	–	0	<1.3	–	–
North China	244	80.3	<1.4	22.4	140.0	78.3	<1.5	73.2	2,560	52.0	<1.3	48.0	2,660.0
South China	379	94.7	<1.4	11.1	1,552.0	80.7	<1.5	14.6	358	24.8	<1.3	–	28.8
Overall China	623	89.1	<1.4	17.8	1,552.0	78.2	<1.5	35.4	2,560.0	37.0	<1.3	–	2,660.0

Table 3 The summary results of methyl parathion, malathion and parathion statistical analysis

Basins	Sample number	Methyl parathion				Malathion				Parathion			
		Rate %	Min	Mean	Max	Rate %	Min	Mean	Max	Rate %	Min	Mean	Max
Songhuajiang River	40	20.0	<0.9	–	130.0	17.5	<0.8	–	1,070.0	17.5	<0.9	–	30.0
Liaohe River	58	70.7	<0.9	25.3	120	70.7	<0.8	59.1	180	72.4	<0.9	14.6	40.0
Haihe River	39	100.0	10.0	32.1	160	100.0	10.0	49.5	130	100.0	10.0	19.0	40.0
Yellow River	50	98.0	<0.9	46.9	480	100.0	10.0	101.2	1,290.0	98.0	<0.9	23.6	150
Yangtse River	150	4.0	<0.9	–	20.0	21.3	<0.8		540.0	5.3	<0.9		20.0
Huaihe River	39	46.2	<0.9	–	60.0	64.1	<0.8	36.5	229	56.4	<0.9	8.3	30.0
Pearl River	150	1.3	<0.9	–	3.1	24.7	<0.8		216	4.0	<0.9		2.0
Southeast drainage area rivers	74	4.0	<0.9	–	16.4	28.4	<0.8	–	262	6.8	<0.9	–	2.0
Northwest drainage area rivers	18	100.0	10.0	16.9	50.0	100.0	10	51.8	180.0	94.4	<0.9	17.2	30.0
Southwest drainage area rivers	5	0	<0.9	–	<0.9	0	<0.8	–	–	0	<0.9	–	<0.9
North China	244	71.3	<0.9	25.7	480	74.6	<0.8	59.5	1,290.0	72.1	<0.9	14.6	150
South China	379	2.9	<0.9	–	20.0	23.7	<0.8	–	540	5.0	<0.9	–	20.0
Overall China	623	29.7	<0.9	–	480.0	43.5	<0.8	–	1,290.0	28.6	<0.9	–	150.0

that the two compounds were the prevalent organophosphorous pesticides pollutants in Chinese surface water. In addition, the measured concentrations of six compounds were generally low (mean detectable concentrations generally <1 μg/L, Tables 2, 3), without any compound exceeding drinking water guidelines, health advisories, or aquatic-life criteria (Tables 2, 3).

As the prevalent organophosphorous pesticides pollutants in Chinese surface water, the similar spatial distribution occurred for the dichlorvos and demeton. For the seven major river basins and three internal rivers drainage areas, the detection rates of dichlorvos and demeton were also from 0% to 100%. Among different river basins, the mean concentration of dichlorvos in the Pearl River basin was the lowest (mean = 5.6 ng/L) and that in the Yellow River basin was the highest (mean = 40.7 ng/L), the second highest mean level occurred in the northwest region (mean = 27.6 ng/L), followed by the Haihe River (mean = 25.6 ng/L), Huaihe River (mean = 25.2 ng/L) and Yangtze River (mean = 17.9 ng/L). While for the demeton, the mean concentration in the Pearl River basin was also the lowest (mean = 9.1 ng/L) and that in the Liaohe River basin was the highest (mean = 122.1 ng/L), the second highest mean level occurred in the northwest internal rivers (mean = 104.6 ng/L), followed by the Yellow River basin (mean = 78.9 ng/L), the Haihe River (mean = 70.8 ng/L) and the Huaihe River (mean = 41.5 ng/L). In addition, the dichlorvos and demeton detection rate of north China (including Songhuajiang River, Liaohe River, Haihe River, Yellow River, Huaihe

River, and northwest drainage area rivers) was 80.3% and 78.3%, with the mean value 22.4 ng/L and 73.2 ng/L, respectively. While the dichlorvos and demeton detection rates of south China (including Yangtse River, Pearl River, southeast drainage area rivers and southwest drainage area rivers) was 94.7% and 80.7%, with the mean value 11.1 ng/L and 14.6 ng/L. According to the concentration distribution results, the river basins with high dichlorvos and demeton concentrations mainly occurred in north China, Which suggests that the high levels of the two compounds in surface water were mainly present in areas of north China, while the more polluted sites of the south China mainly occurred in the Yangtze River. The statistical analysis result showed a significant difference between the north China rivers and the south China rivers concentrations ($t = 1.65$, $p < 0.001$).

The methyl parathion, malathion, parathion, and dimethoate detection frequencies were less than the dichlorvos and demeton detection rates, and there was also similar spatial distribution trend characteristics to the dichlorvos and demeton. For the dimethoate, there were only the Haihe River (66.7%), Yellow River (68.0%) and Huaihe River (59.0%) basins with the detection frequencies higher than 50%, and the three basins mainly occurred in the north China. The dimethoate detection rate of north China was 52.0%, with a mean value 48.0 ng/L, while detection rate of south China was only 24.8%. The dimethoate concentration distribution results suggested that the river basins with high dimethoate concentrations mainly occurred in north China, the similar situations occurred in the methyl parathion, malathion, and parathion. The river basins with the methyl parathion detection higher than 50% were Liaohe River, Haihe River, Yellow River, and the northwest drainage area rivers of the north China, the detection rates of the other river basins were all less than 50%. While for the malathion and parathion, the detection frequencies of Liaohe River, Haihe River, Yellow River, Huaihe River, and the northwest drainage area rivers of north China were all higher than 50%, the other river basins with the detection rates less than 50%. The detection rates of methyl parathion, malathion, parathion, and dimethoate in north China were 71.3%, 74.6%, 14.6% and 52.0%, respectively, much higher than that of methyl parathion, malathion, parathion and dimethoate in south China (2.9%, 23.7%, 5.0% and 24.8%, respectively). From the concentration distribution results, it should be known that the river basins with high methyl parathion, malathion, parathion and dimethoate concentrations mainly occurred in north China, which suggests that the high levels of the four compounds in surface water were mainly present in areas of north China.

Due to their efficiency and inexpensiveness, OPPs had been widely used in agriculture for crop protection and fruit tree treatment as substitute for OCPs. The OPPs have occupied the dominant position in production and use in China since the use of OCPs such as DDT and HCH was banned in 1983. There are over 20 kinds of OPPs in production and it was reported that, 0.11 million tons of OPPs were used in 1989, occupying 52.8% of the total usage of pesticides and 77% of the total usage of insecticide in China (Pan et al. 1997). It was obvious that dichlorvos, demeton had higher occurrence and concentration than other OPPs because of a large amount of application loads. It was reported that in 2002, over 10 thousand tons of dichlorvos, trichlofon and demeton were employed in China, respectively, which were far more than other OPPs usage (Hua and Shan 1999). Furthermore, because of the acute toxicity for human being and other endotherm, parathion and methyl parathion were gradually reduced in use. In 2004, parathion and methyl parathion was decreased by 22.51% and 14.52% in usage compared to in 2003 (Shao and Su 2004). In January 2004, the registration of five highly toxic OPPs including parathion and methyl parathion was stopped and from January. 2007, their use will be completely forbidden in China (Tang et al. 2004). This can give a good explanation of the lower concentration and occurrence of them.

Another conclusion drawn from the data was that there was a significant difference in concentration between the north China and the south China surface water and the high levels of OPPs in surface water were mainly present in areas of north China. The highest concentrations of OPPs were 1,552.0 ng/L for dichlorvos in Yangtze River, 2,560.0 ng/L for demeton in Liaohe River, 2,660.0 ng/L for dimethoate in Yellow River, 480.0 ng/L for methyl-parathion in Yellow River, 1,290.0 ng/L for malathion in Yellow River and 150.0 ng/L for parathion in Yellow River. Except for dichlorvos, the highest concentration of OPPs appeared in the rivers of north China. This can be explained by the special mode of Chinese economy development. In the south China, the highly developed economy made the industry occupying the main proportion of economy total amount, the farming only occupied a small ratio. While in north China, the farming is still the main component of north China economy, OPPs had been widely used in these agriculture regions every year. The Yellow river, Huaihe river, Haihe river, and Liaohe River basins of north China were the important agricultural areas with an extremely large numbers of agricultural farmlands. OPPs used yearly on a regular basis may be a contributing factor for the elevated levels of OPPs concentrations in north China surface water. Actually, there are also several other factors affecting the occurence and persistence of OPPs in surface waters. One is the stability of the residues of the several pesticides which vary widely, another factor which will affect persistence and hence detectable amounts is the climatic differences from North to South, and the differences in environmental conditions between collection sites is also the factor affecting the detectable amounts and distribution. In summary, all the factors mentioned above may together result in the elevated levels of OPPs concentrations in north China surface water.

In this study, the detection rates of dimethoate, methyl parathion, malathion, and parathion were 37.0%, 29.7%, 43.5% and 28.6%, respectively, Which were all below the 50%. The fact of low detection frequency for these compounds indicated that the dimethoate, methyl parathion, and malathion were not the main OPPs pollutants in Chinese surface water. When Compared with the pesticides concentrations in European freshwaters, parathion methyl was detected in the Segre (Spain), Elbe and Rhine rivers (Germany) at concentrations up to 270, 332 ng/L and 40 ng/L, respectively (Planas et al. 1997; Gotz et al. 1998). Malathion was detected in River Arno (Italy), at 170.0 ng/L (Griffini et al. 1997), and dimethoate in the Elbe and Rhine rivers (Germany) at 3,210.0 ng/L and 50.0 ng/L, respectively (Gotz et al. 1998). The concentrations of the dimethoate, methyl parathion, and malathion in this study were much lower, which suggested that the Chinese surface water is also somewhat moderately polluted by dimethoate, methyl parathion, malathion, and parathion. On the contrary, the detection frequency of dichlorvos and demeton was 89.1%, 78.2%, with the mean values of 17.8 ng/L and 35.4 ng/L. Compared with the dimethoate, methyl parathion, malathion, and parathion, the concentrations and frequencies of dichlorvos and demeton were much higher, which indicated that the dichlorvos and demeton were the main OPPs contaminants and the Chinese surface water was contaminated by them in certain degree.

Conclusion

This work investigated contamination status of dichlorvos, demeton, dimethoate, methyl parathion, malathion, and parathion in Chinese surface water and provided data on the levels of the six organophosphorous pesticides. In general, the measured concentrations for the six compounds were low and rarely exceed the Environment quality standard for surface water of China. For the six compounds, the dichlorvos and demeton are the major OPPs contaminants with higher detection frequency and concentration relative to the other four OPPs compounds. Comparison with other places of the world, the surface water of China was somewhat moderately contaminated by dimethoate, methyl parathion, malathion, and parathion, while the surface water in China was polluted by the dichlorvos and demeton in certain degree because of their much higher concentrations and detection frequencies. There was a significant difference in concentration between the north China and the south China surface water and the high levels of OPPs in surface water were mainly present in areas of north China. It is submit that the Yellow River basin, Huaihe River, Liaohe River, and Haihe River basins of the north China should be given priority in terms of the prevention and control of OPPs contamination.

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