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HYDROCHEMISTRY, HYDROBIOLOGY: ENVIRONMENTAL ASPECTS

Concentrations, Possible Sources and Influence Factors of Dissolved Polychlorinated Biphenyls in the Water of Yangzhuang Coal Mining Subsidence Area, China[1]

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Abstract—To understand the content, composition and other pollution states of dissolved PCBs in the waters of coal mining subsidence area, the research object is determined as the water of Yangzhuang mining subsidence area. Seven point locations are selected there and three layers of water samples are collected in each of the point locations so as to detect and analyze PCBs with GC-MS and make sources recognition and analysis of relative factors. Results show that other PCB congeners all have been detected except PCB138 congener among the 14 detected PCBs congeners. PCBs mainly consist of trichlorodiphenyl and pentachlorodiphenyl. These two substances in each site occupy 59 to 79% of total PCBs. The pollution sources are primarily two sorts: the atmospheric transmission source and the unintended source. PCBs content has a moderate linear positive correlation with DOC and water temperature and a moderate linear negative correlation with DO. PCBs concentration is in a lower-middle level.

Keywords: coal mining subsidence area, waters, PCBs, concentrations, relative factors

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INTRODUCTION

In China, coal-driven energy holds over 70% of the total primary energy production. By the year of 2015, China's coal production has readily reached nearly 3.69 billion tons as a consequence of increasing demand. Major environmental challenges are faced by regions with coal mines, such as air, soil and water pollutions and the generation of solid wastes, like coal gangue and fly ash. Particularly, land subsidence is of great concern among all these issues. In the coal mines of east-central China, due to the regional hydrological and geological conditions, extensive water-rich landscapes have been formed by land subsidence. These low-lying landscapes are distinguished by thick Quaternary sediments and a densely knitted network of rivers. The Huainan Coal Mines in Anhui Province represent a typical case, which is one of the largest coal producing areas in China, generating 75.68 million tons of raw coal in 2014. One of the severe and profound influences of land subsidence is the loss of farmland which further affected the livelihoods of local people. Moreover, in many large-scale mining areas, land subsidence has even changed the local hydrology and the ecological environment. Small lakes are the dominant landscapes in the mining areas. The government of Huainan Municipality has initiated to regulate these areas as large plain reservoirs, lakes and fishponds, performing multiple functions as flooding buffer zones and aquatic ecological rehabilitation.

Persistent Organic Pollutants (POPs) are gaining people's attention because of intense carcinogenesis, tetratogenesis and mutagenesis. POPs, a kind of organic pollutants with severe harm which grow naturally or are artificially synthesized, can hardly degrade in environmental media while can undergo long-range atmospheric transport and also bioaccumulate through the food chain, imposing threats to humans' physical health and ecological environment [18]. As one of the 12 kinds of POPs under top-priority control in *Stockholm Convention on Persistent Organic Pollutants*, Polychlorinated Biphenyls (PCBs) are a sort of organic compound of nonpolar semi-volatility and incombustibility. They also does not dissolve in water [10]. PCBs are a generic term describing a series of chlorinated biphenyl compounds [2]. There are 209 kinds of PCBs congeners totally [20], according to the different substituent number and position of Cl atom in the benzene ring. With certain thermostability and mobility, PCBs extensively exist in natural environment and have been detected in places that are immensely influenced by human activities and polar

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areas [3, 6, 16]. China had started to produce PCBs since 1965 and called a halt in the early of 1980s [13]. However, the residues of PCBs still remain abundant, greatly threatening humans' physical health and ecological system. Researches at home and abroad on the residues and pollution characteristics of PCBs concentrate in the waters of offshore, riverway and lake areas but scarcely report the pollution characteristics in the specific waters of coal mining subsidence area [21, 23]. Thus, this research takes Yangzhuang subsidence area in Huainan Coal Mines as the research object, analyzes the content level and compositional characteristics of dissolved PCBs in water, detects relative physical and chemical indicators and deduces possible pollution sources. All these lay a foundation for the comprehensive assessment of the water quality and environmental risk in coal mining subsidence areas.

MATERIALS AND METHODS

Study Area

Located in the north of Huainan municipality, China, Panyi coal mining subsidence area faces Cihuaixinhe River in north and borders on Huaihe River in south. There are multiple subsidence waters and dense-covered rivers. Yangzhuang subsidence area is the largest subsidence water in Panyi (116°49′30″–116°52′00″ E, 32°47′00″–32°48′30″ N), with surface water covering nearly for the longest years—nearly 20 years. According to the average deposition rate of common lakes, Yangzhuang subsidence area will emerge as a lacustrine deposit after about 20 years [17], covering the original plough layer of farmland. The sediment in water retains certain soil properties. The average water depth reaches 3.6 m with the maximum depth reaching more than 6 m. Now, Yangzhuang subsidence area has already been developed into a separate fishing area for farmers who lost their land to earn their living. The needed nutrient for its ecological system mainly relies on exogenous supply or its own inside environment. Mud River is the major exogenous river in this area, linking to this water area after crossing multiple residential areas. It flows into the subsidence water from southwest and flows out from southeast, during which a large amount of sanitary sewage and industrial wastewaters of Panyi mine was brought here. It is reported that Yangzhuang subsidence water area encounters severe eutrophication problem and some other ecological problems [26]. From all mentioned above, researches on organic pollutions like PCBs in this area are typical and representative.

Sampling

In August, 2014, 7 sampling sites were arranged in Yangzhuang coal mining subsidence area of Panyi mine with GPS. In each site, 3 water samples were collected respectively 0.5 m above the bottom of the water, in the half of the water and 0.5 m under the water surface and recorded as *a*, *b* and *c*. Due to the vastness of the research area, the layout of the sampling sites should be kept even. Meanwhile, such unique points as the confluence with the Mud River, shoresides and the middle of fishpond should all be collected. Research area and sampling sites are shown in Fig. 1. Water samples should be brought back to the laboratory as soon as possible and analyzed within 24 h.

Materials and Reagents

Materials primarily include C18 column of solid-phase extraction (Waters Sep-Pak), glass fiber filters, anhydrous sodium sulfate and sodium hydroxide. Reagents primarily include n-hexane, dichloromethane and methyl alcohol (all are chromatographically grade and made by the U.S. TEDIA Company). The 17 kinds of mixed standard substances of PCBs include PCB18, PCB28, PCB31, PCB44, PCB52, PCB101, PCB105, PCB118, PCB138, PCB149, PCB153, PCB174, PCB180, PCB194, PCB65, PCB155, and PCB204 (all are made by the U.S. Accustandard Company except PCB204, the internal standard, which is made by the U.S. O2si Corporation, PCB65 and PCB155). The standard reagents of recovery rate indicators include PCB65 (made by the U.S. O2si Corporation) and PCB155 (made by the Germany Dr. Ehrenstorfer Company).

Dissolved Oxygen (DO) and water temperature are determined by the Portable Dissolved Oxygen Meter (DO200, made by the U.S. YSI Company) in sampling sites. pH is determined by the Portable pH Meter (pH100, made by the U.S. YSI Company). As water samples are brought back to the laboratory, 10 mL are taken out to detect the total organic carbon (TOC). Then, GF/F glass fiber filter is used to have samples filtered. After that, 10 mL of the filtered samples are taken out to detect dissolved organic carbon (DOC). And lastly, another 1 L of the filtered water samples is taken out to conduct solid-phase extraction.

Sample Extraction

There are four steps of solid-phase extraction: (1) Activated column: 3 mL of dichloromethane are added into the columns in twice. Then, 3 mL of methyl alcohol and 3 mL of ultrapure water are processed in the same manner. (2) Sample loading and enrichment: add 10 mL of methyl alcohol and recovery rate indicators into 1000 mL of water samples by transfer pipette, then flow the solution through the activated column. (3) Drying and elution: pump up for about 15 min to keep SPE column dry after the columns are leached by 5 mL of ultrapure water. Then, 8 mL of dichloromethane/n-hexane (7 : 3, V/V) eluant are used to steep the dried columns for 5 min.

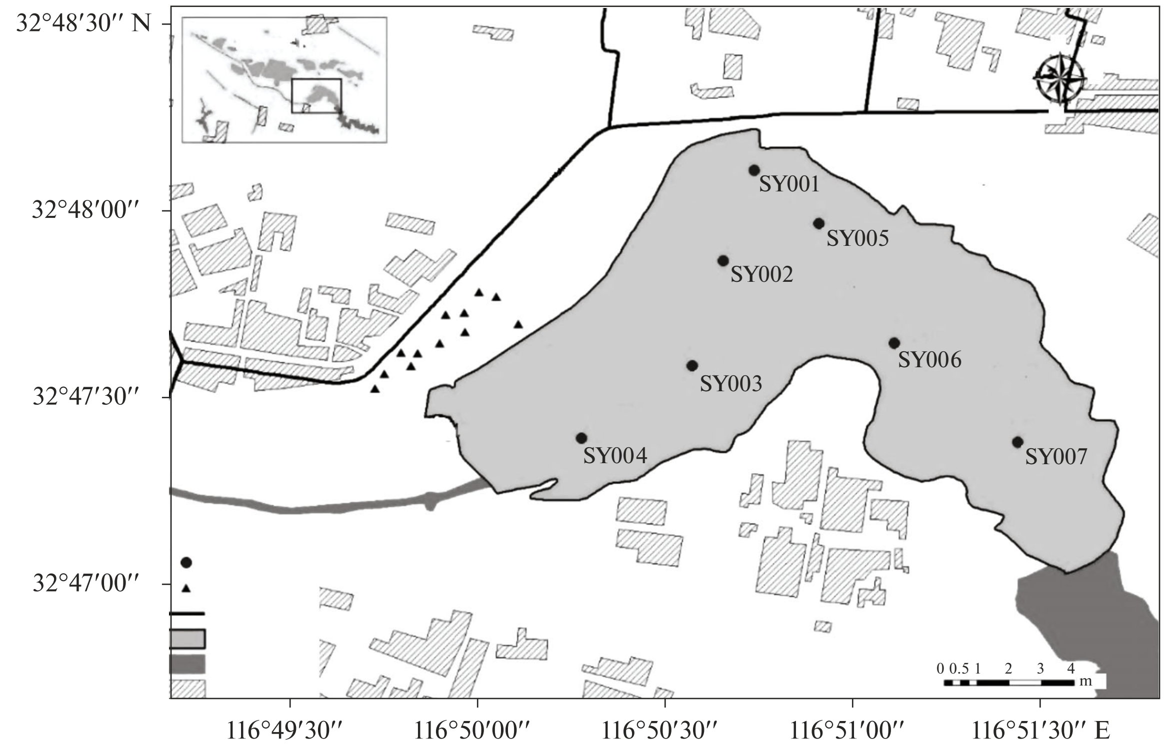

Fig. 1. The scattergram of research area and sampling sites.

(4) Dehydration: pass the collected eluant through a 4 cm dry column of anhydrous sodium sulfate.

As the solid-phase extraction of water samples is finished, the eluants should be concentrated, exchange solvents and be kept in a constant volume. Then, transfer all solvents into the vials of autosamplers and put internal standards of PCBs onto the vials. Following that, wait for GC-MS to analyze.

Instrumental Analysis

Instruments: the Clarus SQ 8 GC-MS made by the U.S. PerkinElmer Corporation includes two parts: gaschromatograph (Clarus 680) and mass spectrometer (SQ 8 MS). Chromatographic column is Elite-5/MS. The internal standard method should be adopted to conduct quantitative analysis, PCBs material composition should be reached through the retention time, peak heights and peak areas of each chromatographic peak. Qualitative analysis of substances can be conducted through the comparison of the appearance time of each chromatographic peak of standard samples.

Quality Assurance/Quality Control (QA/QC)

Quality assurance/quality control (QA&QC) measures adapt the monitoring method recommended by the U.S. EPA to set up parallel samples, standard sample recovery and methods in blank. During the earlier stage of experiment, the recovery rates of the 14 kinds of mixed standard substances are all within the range of 70–140% that the method has ruled. The recovery rates of the two recovery indicators, PCB65 and PCB155, are all in the range of 70–130%. The relative standard deviation among parallel samples is less than 5%. There is no target contaminant has been detected in the blank samples. All these show that the analytical method conforms to the requirements and the data is reliable.

RESULTS AND DISCUSSION

Distributional and Compositional Characteristics

Each of the 7 sampling sites is divided into three layers so that there are totally 21 water samples. Among them, the content of the *a*-layer $\sum_{14}$PCBs which is 0.5 m above the bottom of water is 16.00–26.36 ng/L with an average concentration of 19.93 ng/L; the content of *b*-layer $\sum_{14}$PCBs which is in the half of the water is 16.97–32.01 ng/L with an average concentration of 21.46 ng/L; the content of *c*-layer $\sum_{14}$PCBs which is 0.5 m under the water surface is 16.05–30.56 ng/L with an average concentration of 21.93 ng/L. The column diagram of PCBs concentration in different water layers of each sampling site is in Fig. 2. The sampling point with the least PCBs concentration lies in point SY001, the north point of the coal mining subsidence waters, the average PCBs con-

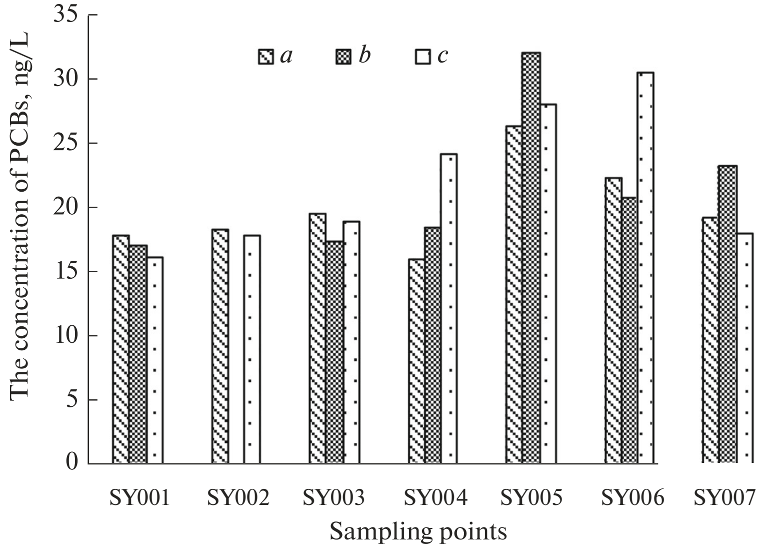

Fig. 2. The concentration histogram of PCBs in different layers of sampling sites.

centration of which in different layers is 16.95 ng/L. The sampling point with the most PCBs concentration lies in point SY005, the east point of the waters, the average PCBs concentration of which in different layers is 28.79 ng/L. This point is in the bed of the Mud River. A mass of agricultural and industrial waste waters flow into the Mud River resulting higher PCBs concentration in SY005, SY006 and SY007 than that in other sampling sites in spatial distribution. The range of PCBs concentration in different layers of sampling sites is 16.00–32.01 ng/L. The minimum value is in SY004a. The maximum value is in SY005b. 14 PCB congeners have all been detected except PCB138 congener. Among them, PCB18, which belongs to trichlorodiphenyl, has the highest concentration in each sampling point. The second is PCB118, which belongs to pentachlorodiphenyl. The one has the least concentration is PCB105.

PCBs with different number of chlorine atoms have different physicochemical properties and different sources. Therefore, the composition of PCBs can be analyzed through the number of chlorine atoms. Further, the distribution characteristics of PCBs contaminants can also be analyzed. PCBs generally can be divided into different kinds of chlorinated substituted benzene. In this research, the trichlorodiphenyl includes PCBl8, PCB28 and PCB31; the tetrachlorobiphenyl includes PCB44 and PCB52; the pentachlorodiphenyl includes PCBl01, PCB105 and PCB118; the hexachlorobiphenyl includes PCB138, PCB149 and PCB153; the heptachlorobiphenyl includes PCB174 and PCB180; the octachlorobiphenyl includes PCB194. The composition percentage of the 14 indicative PCBs is shown in Fig. 3. From the figure, the dissolved PCBs in water are mainly such low chlorinated substituted benzenes as trichlorodiphenyl and pentachlorodiphenyl. The sum of the two substances in each sampling site accounts for 59–79% of the total. This conforms to the PCBs substances that are primarily produced and used in China and is also similar to the mixed pollution characteristics of trichlorodiphenyl and pentachlorodiphenyl that are shown in the researches of most districts. The PCBs that had been produced in China in 1960s to 1970s were mainly No. 1 PCBs and No. 2 PCBs. No. 1 PCBs, which were mainly trichlorodiphenyl, were used as power transformers and the dielectric medium of condenser in a closed type. However, currently, many of them had already be abandoned. No. 2 PCBs, which were mainly pentachlorodiphenyl, were used as paint additives in an open type. Data shows that the total productions of the no. 1 PCBs and no. 2 PCBs in China reached respectively 9000 tons and 1000 tons. The main compositions of the nos. 1 and 2 PCBs were basically similar to those of such PCBs products as Aroclor 1242 and Aroclor 1254 made by the U.S. Mansanto Company. According to the research of Frame, Aroclor 1242 mixture is mainly low chlorinated PCBs. Aroclor 1254 mixture mainly consist of high chlorinated PCBs [27].

From Fig. 3, the trichlorodiphenyl has the highest concentration with an average contribution rate of 50%. SY003 contributes the most, reaching 69%; following that is pentachlorodiphenyl which has a contribution rate of 21%. SY007 contributes the most, reaching 49%. The general researches show that the environmental behaviors of PCBs have certain relations with the substituent number of chlorine atoms. The hexachlorobiphenyl PCB138 that has high relevance ratio in areas with advanced industry and high-level electronic waste dismantling has not been detected in coal mining subsidence water. Thus, the

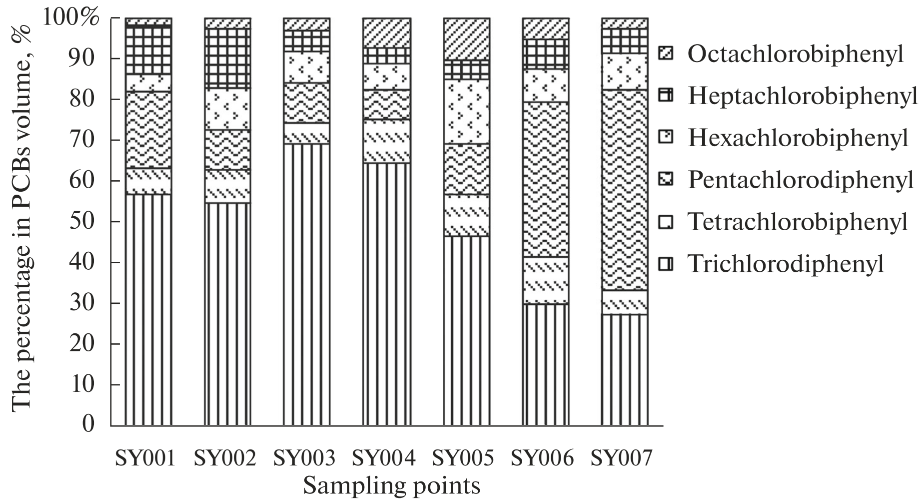

Fig. 3. The 100% stacked column chart of PCBs compositional content in sampling sites.

main source of PCBs is not the dismantling of electronic waste and the use of chemicals. The trichlorodiphenyl congeners in subsidence area come from the impregnant of condensers and the oil of transformers. The pentachlorodiphenyl comes from the open use of paint additives. According to relative reports, the leaks of PCBs had occurred in many condensers' sealing points.

Possible Sources

Therefore, it can be initially presumed that there are three possible sources of PCBs in Yangzhuang coal mining subsidence waters: (1) The leaks of condenser oil in some factories of surrounding areas and the emission of wastewaters from factories such as paper mills, coal-fired power plants and coal-picking industry will contaminate coal mining subsidence area through the transportation of the Mud River; (2) Subsidence waters used to be farmlands and villages. The coal mining has led to ground degradation, making the subsidence exacerbated. Moreover, the condensers in villages were abandoned and dismantled. The oil of transformers and the lost impregnant of waste condensers around the coal mining areas converged into the subsidence water by rains, distillation effect and atmospheric deposition [5, 12, 19]; (3) low-chlorinated PCBs like trichlorodiphenyl and pentachlorodiphenyl are of strong volatility and are very easy to volatilize into air and subside into water with the long-distance flowing and drifting of atmosphere. This indicates that there are two kinds of pollution sources of the dissolved PCBs in the water of coal mining subsidence area: the source of atmospheric transport and the unintended source.

Influence Factors

Research indicates that the contents of PCBs in almost all natural waters have been severely influenced by human activities. For example, seas that are far away from land always have low PCBs concentration, while areas that are near to the land are much more polluted because of busy human activities and shipping and a considerable amount of industrial and tertiary waste. In addition, the content of PCBs in waters are also influenced by factors like the change of seasons, the size of water flow and suspended solids, temperature, pH and organic carbon content level. This research analyzes the correlation between the total content of $\sum_{14}$PCBs in each sampling site and the six indicators: temperature, the content of DO, pH, chemical oxygen demand (COD_{Cr}), TOC and DOC that have been detected in the waters. The data has also been fitted. Results indicate that the content of dissolved PCBs in water basically has no linear correlation with the pH of waters, low linear correlation with COD and TOC and moderate linear correlation with DOC, DO and water temperature (p = 0.01). Among them, the DOC, DO and the water temperature have the highest correlation with the content of PCBs. The reason why these factors have influences on the content of PCBs should be attached with great importance.

The content of $\sum_{14}$PCBs in water has significant positive correlation with DOC, and the coefficient index is 0.6935. The scatter diagram and the fitting straight line can be seen in Fig. 4. Some colloidal substances (organic) in natural waters account for more than 90% of the DOC in water. Meanwhile, organic colloidal substances have the property of high active surface area and better adsorption capacity of organic pollutants like PCBs [11]. Thus, the content of DOC in water restricts the interphase distribution of PCBs

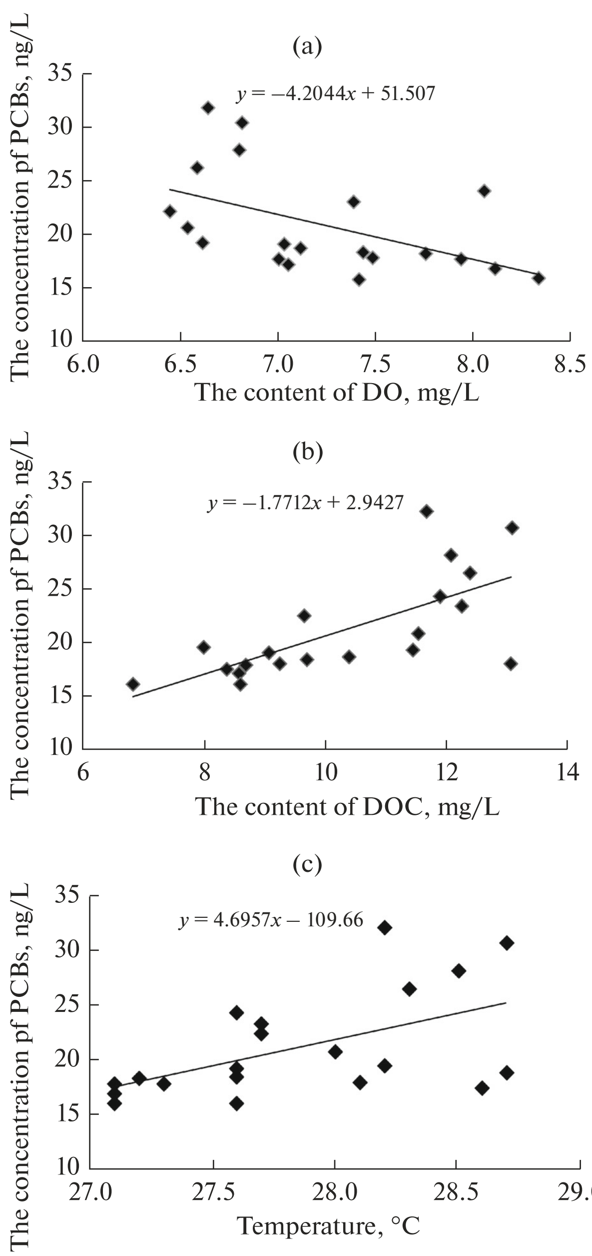

Fig. 4. The scatter diagrams and the fitting straight lines of DOC, DO, temperature and PCBs in water.

to a certain extent. After water samples are filtered by fiber filter membrane, major particle matters can be removed while macromolecule organic colloidal matters such as protein and humic acid are hard to be filtered completely. The fitting result shows that the content of DOC in water has positive correlation with the environmental behaviors of PCBs, which means that as the concentration of DOC grows, the content of the dissolved PCBs in water becomes higher.

The content of $\sum_{14}$PCBs in water has significant negative correlation with DO, and the coefficient index reaches 0.5058. The scatter diagram and the fitting straight line can be seen in Fig. 4. The dissolved oxygen level reflects the pollution level of substances in water like PCBs in some degree. In low oxygen or anaerobic environment, if organic matters decompose into water, the circulation of materials will all be restrained, which lead the concentration of organic matters in water to grow higher and the water quality to deteriorate. The fitting result shows that as the concentration of DO decreases, the content of dissolved PCBs in water grows higher.

The content of $\sum_{14}$PCBs in water has significant positive correlation with the water temperature. The coefficient index is 0.5339. The scatter diagram and fitting straight line can be seen in Fig. 4. On the one hand, as the water temperature grows, the solubility of PCBs in water becomes higher. On the other hand, as the water temperature grows, the dissolved oxygen level in water drops, and the concentration of PCBs in water increases [8, 14]. Secondly, PCBs are adsorbed by suspended solids and colloid, leading to exothermic entropy change. Physical reactions play the main role in the adsorption process [4, 22], during which as the water temperature increases, the molecular distance of particles is enlarged, and the interaction force between the molecules is weakened, causing the change of adsorption activities and the reduction of adsorbing capacity. As a result, the content of PCBs in suspended phase will be reduced while the content of PCBs in aqueous phase will increase accordingly with the increase of water temperature.

Ecotoxicological Concerns

Referring to the ecotoxicological concerns, PCBs are toxic to aquatic organisms. Especially, Yangzhuang subsidence area has already been developed into separate fishing zones for farmers who lost their land to earn their living. To evaluate the ecotoxicological aspect of PCB contamination, some U.S. EPA quality guidelines have been published. According to the evaluation criteria of the U.S. EPA on the PCBs in water, the concentration of PCBs should be less than 14 ng/L for water, which is considered to have no hazard for aquatic and human health. However, the Maximum Contaminant Level (MCL) of PCBs in drinking water set by the U.S. EPA in National Primary Drinking Water Regulations is 500 ng/L. Based on the quality standards of surface water published by the Ministry of Environmental Protection of the People's Republic of China (GB3838-2002); the PCBs level is 20 ng/L. The comparison between the result of the data analysis of the dissolved PCBs in this research area and that in other areas of the world can be seen in Table 1 [1, 7, 9, 15, 24, 25, 28, 29].

The content of the PCBs (16–32 ng/L) in the subsidence area has exceeded 14 ng/L (U.S. EPA) and 20 ng/L (GB3838-2002) but met the MCL (U.S. EPA). It is also higher than the lower level of the domestic inland waters (the Three Gorges Reservoir

Table 1. The concentration comparison of the dissolved PCBs in this research area and in other waters, ng/L

Location	Time of sampling	Variety	Range	Mean	References
Venice Lagoon, Italy	2003	54	0.5–11	–	[15]
Baltimore Harbor, USA	1997–1998	24	0.1–1.5	0.5	[1]
Northern Chesapeake Bay, USA	1997–1998	24	0.2–1.0	0.4	[1]
Mississippi River, USA	2004	28	22–163	–	[28]
North Western Mediterranean	2001	41	2.4–71	–	[7]
The Three Gorge Reservoir, China	2008	18	0.1–0.5	0.2	[24]
Minjiang River Estuary, China	2003	21	204–2473	985	[29]
Meizhou Bay, China	2013	28	5.3–43	19	[9]
Bohai Bay, China	2004	12	60–710	210	[25]
Haihe River, China	2004	12	310–3110	760	[25]
Daya Bay, China	2001	12	91–1355	310	[30]
Yangzhuang coal mining subsidence Area	2014	14	16–32	21	This study

of China, Northern Chesapeake Bay of USA and etc.), but far less than the higher level of some areas such as offshore, bay and estuary (Bohai Bay, Minjiang River Estuary and etc.). This indicates the concentration of PCBs in the water of Yangzhuang subsidence area is in a medium-lower level, and the concentrations of PCBs observed have low ecotoxicological effects on aquatic organisms and human health.

CONCLUSIONS

This research reflects the distribution of PCBs in the area that has been influenced by exogenous human activities. Among the 14 detected PCBs congeners, other PCB monomers had all been detected except PCB138. The dissolved PCBs in the water of Yangzhuang coal mining subsidence mainly consist of such low-chlorinated substituted benzene as trichlorodiphenyl and pentachlorodiphenyl. It can be initially presumed that there are two pollution sources of the PCBs: the source of atmospheric transport and the unintended source. From the comparison between the analytical results of the data on the dissolved PCBs in this research area and those in other main research waters of the world, the concentration of PCBs in this water is in medium-lower level, and the water has already been slightly contaminated by POPs like PCBs. Therefore, precautions still should be taken to prevent potential pollutions in later period.

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