

# Levels and Sources of Organochlorine Pesticides in Surface Sediment from Anhui Reach of Huaihe River, China

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**Abstract** Twenty-two organochlorine pesticides (OCPs) were investigated in Anhui reach of Huaihe river, China. Seventeen out of 22 OCPs were detected by GC-MS. The mean concentrations of OCPs followed the order: HCHs > DDTs > HCB > chlordanes > endosulfans. Levels of total HCHs and total DDTs ranged from 2.54 to 13.91 ng g<sup>-1</sup> (mean = 7.52 ng g<sup>-1</sup>) and 0.016 to 2.54 ng g<sup>-1</sup> (mean = 0.45 ng g<sup>-1</sup>), respectively. The concentrations of DDTs were lower than those of HCHs. Compared with the other rivers in China, DDTs and HCHs were relatively lower or similar. Statistical analysis indicated that the OCPs concentration differences were not insignificant between upstream, midstream and downstream. The compound compositions suggested that historical usage of lindane and fresh DDT were the main sources. The regression analysis indicated that TOC has influence on the levels of HCHs and little influence on the levels of DDTs in the sediments.

**Keywords** Organochlorine pesticides · Surface sediments · Total organic carbon · Huaihe river

Organochlorine pesticides (OCPs) have been extensively studied due to their ubiquity, bioaccumulation and

persistence in the environment (Bajwa et al. 2016). The residence time of OCPs in the environment is notably long. The sediment is an important reservoir of OCPs (Li et al. 2015). These compounds can enter marine and freshwater ecosystems through effluent release, atmospheric deposition and runoff, then can be adsorbed on suspended particulate matter due to their high hydrophobicity and low water solubility, thereby accumulating in sediments (Wu et al. 2015).

Huaihe river, one of the seven largest rivers in China, flows through five provinces of Henan, Hubei, Anhui, Jiangsu and Shandong. The river has become vulnerable to water pollution due to increasing discharges of industrial and agriculture waste along with rapid development of the local economy. Wang et al. (2009) reported ecological risk assessment of OCPs in water from Jiangsu reach of Huaihe river. Sun et al. (2010) studied sources of OCPs in sediments from Henan reach of Huaihe river. Meng et al. (2014) have also studied perfluoroalkyl substances and OCPs in sediments in Huaihe watershed from Anhui reach of Huaihe river. However, little information is available for OCPs contamination in Anhui reach of Huaihe river. Investigation on OCPs levels in sediments from Anhui reach of Huaihe river is important in order to better understand the contribution of that to Huaihe river pollution.

The aims of this paper were to: (1) investigate the levels and patterns of OCPs in surface sediments from Anhui reach of Huaihe river, (2) analyze their spatial distribution, (3) measure total organic carbon (TOC) levels and study its influential role in concentrations of OCPs in sediment.

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## Materials and Methods

A total of 16 surface sediment samples were collected with a stainless steel grab from Anhui reach of Huaihe river in November 2015. The sampling sites were shown in Fig. 1, among which sampling sites S1–S5, S6–S10 and S11–S16 were from the upstream, midstream and downstream regions of the Huaihe river, respectively. Approximate 500 g of every sediment sample was collected. After collection, pebbles and plant leaves in sediments were removed manually. Collected sediments from each site were mixed homogeneously. Sixteen sediment samples were collected from Huainan district to Bengbu district. All the samples were wrapped with aluminum foil and mixed separately and stored at  $-20^{\circ}\text{C}$  in pre-washed glass bottles until analysis.

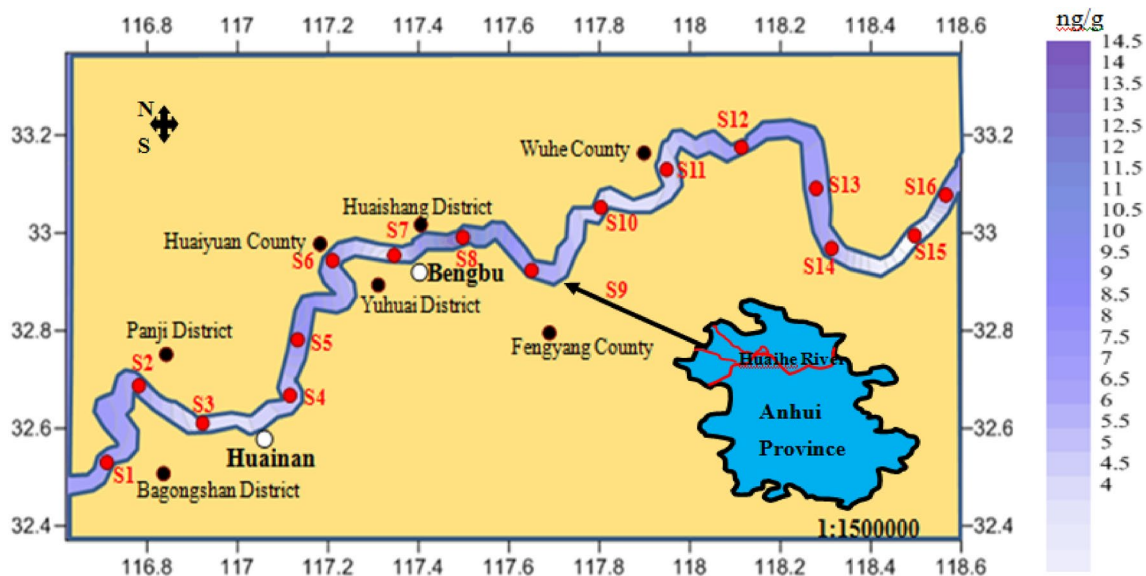
The samples were freeze-dried, hand-sieved ( $200\ \mu\text{m}$ ) and homogenized. Approximately 20 g of homogenized samples were extracted in a Soxhlet apparatus with 200 mL of dichloromethane for 48 h. Activated copper was added for desulfurization. The coppers were cut into squares of 0.5 cm, and washed three times with hydrochloric acid, methylene chloride and n-hexane. The extractor water bath temperature was maintained at  $49^{\circ}\text{C}$ . Extracts were then rotary evaporated to 2 and 10 mL n-hexane was added to solvent-exchange and was further reduced to 1–2 mL by rotary evaporation. The water bath temperature for the rotary evaporation was maintained at  $35^{\circ}\text{C}$ . The alumina/silica ( $v/v=1:2$ ) gel column was eluted with 70 mL of dichloromethane/hexane ( $v/v=3/7$ ) for OCPs. The activation method of the alumina and silica has been reported in our previous article (Da et al.

2014). The eluate was concentrated to 1 mL by rotary evaporation.

The determination of 22 OCPs (DDTs, HCHs, Heptachlor, Heptachlor epoxide, *cis*-chlordane, *trans*-chlordane, endosulfan I, endosulfan II, HCB and mirex, aldrin, dieldrin, endrin, methoxychlor) was quantified with an Agilent 6890 series gas chromatography system connected to an Agilent 5973 network mass spectrometer selective detector (GC-MS). Samples ( $1\ \mu\text{L}$ ) were auto injected in splitless mode, and the separation was performed with a DB-5 MS fused silica capillary column ( $30\ \text{m}\times 0.25\ \text{mm}\times 0.25\ \text{mm}$ ). The carrier gas was helium (99.999%) at flow rate of  $1.0\ \text{mL}\ \text{min}^{-1}$ . The MS was operated in  $\text{EI}^+$  mode with selected ion monitoring, and the electron energy was 70 eV. The column oven temperature was programmed at an increasing rate of  $15^{\circ}\text{C}\ \text{min}^{-1}$  from an initial temperature of  $80^{\circ}\text{C}$  for 1 min, ramping at a rate of  $12^{\circ}\text{C}\ \text{min}^{-1}$  to  $200^{\circ}\text{C}$  for 10 min, to  $220^{\circ}\text{C}$  at  $1^{\circ}\text{C}\ \text{min}^{-1}$  and maintaining for 5 min and to a final temperature of  $290^{\circ}\text{C}$  at  $15^{\circ}\text{C}\ \text{min}^{-1}$  with a final holding time of 5 min.

TOC was analyzed on the basis of the procedure reported by Yadav et al. (2016). Briefly, 3 g sample were dried and mixed with 10% HCl to remove inorganic carbon. Then the samples were washed with deionized water and dried in oven at  $60^{\circ}\text{C}$  overnight. The TOC content was measured with TOC analyzer (Multi N/C 3100 Analytik Jena; Bajwa et al. 2016).

The residue levels of OCPs were quantitatively determined by the internal standard method using peak area. The correlation coefficients of calibration curves of OCPs were all higher than 0.998. Instrumental detection limits (DL) were determined from linear extrapolation from the



**Fig. 1** Sampling sites and spatial distribution of OCPs in the surface sediments

lowest standard in the calibration curve using the area of a peak having a signal-to-noise of 3, which ranged from 0.001 to 0.3 ng·g<sup>-1</sup> for OCPs. The spiked levels were close to or below DL. For every set of ten samples, procedural blanks, spiked blanks and replicate samples were run to check for the interference and cross-contamination. The spiked recoveries of OCPs were in the range of 85%–102% and the relative standard deviation values ranged from 0.2% to 10%. We analyzed three replicate sediment samples for every sediment sample in every sample site. The concentration of OCPs was the average of three parallel samples at each sampling site.

## Results and Discussion

The concentrations of OCPs in sediments are summarized in Table 1. Seventeen (DDTs, HCHs, heptachlor, heptachlor epoxide, *cis*-chlordane, *trans*-chlordane, endosulfan I, endosulfan II, HCB) out of 22 OCPs were detected. The residues of HCHs and DDTs ranged from 2.54 to 13.91 ng g<sup>-1</sup> (mean = 7.52 ng g<sup>-1</sup>) and 0.016 to 2.54 ng g<sup>-1</sup> (mean = 0.45 ng g<sup>-1</sup>), respectively. The detection frequencies for the HCHs and DDTs in the samples were 93.8% and 36.5%, respectively, indicating a wide occurrence of HCHs in the sediments from Anhui reach of Huaihe river. The concentrations of DDTs were much lower than those of the HCHs. A most likely explanation is that the utilization of HCHs in China was significantly larger than that of DDTs in the past (Sun et al. 2010; Meng et al. 2014; Wu et al. 2015). The mean concentration of HCB was 0.05 ng g<sup>-1</sup>, with an intermediate detection rate of 50%. HCB in these studied sediments had higher detection frequencies. HCB was a byproduct in the process of industrial production, and it had never been over used in China (Da et al. 2015). Endosulfan I (mean = 0.03 ng g<sup>-1</sup>) was more abundant than endosulfan II (mean = 0.008 ng g<sup>-1</sup>). The detection rate of endosulfan I (50%) is also higher than the detection rate of endosulfan II. Endosulfans were used to kill pests in the crops in 1990s in China (Da et al. 2014). Heptachlor epoxide and its parent compound heptachlor were detected by a small amount in the sediments. Heptachlor compounds were produced and used from 1960s and 1970s in China. Drins (aldrin, dieldrin and Endrin) were not detected in the studied sediments, relating to their zero production in history in China (Li et al. 2015). Other OCP compounds, such as mirex and methoxychlor, were also not detected. The mean concentrations of different categories of OCPs in samples were in following the order: HCHs > DDTs > HCB > chlordanes > endosulfans.

When compared with the previous studies, the residues of HCHs in this study area were slightly lower than those in the Daling river area (1.1–30 ng g<sup>-1</sup>), the Pearl river

(1.20–17.0 ng g<sup>-1</sup>), and the Haihe estuary (0.997–36.1 ng g<sup>-1</sup>; Zhao et al. 2010) but higher than those in the Chaohu lake (0.04–7.12 ng g<sup>-1</sup>; Li et al. 2015), the Bohai sea (0.16–3.17 ng g<sup>-1</sup>; Li et al. 2009) and Laizhou bay (0.03–6.38 ng g<sup>-1</sup>; Zhong et al. 2011). In the case of DDTs, the concentrations were similar to those in the East China sea (0.06–6.04 ng g<sup>-1</sup>) but lower than those in the Chaohu lake (0.23–85.83 ng g<sup>-1</sup>; Li et al. 2015), Yueqing bay (1.85–16.54 ng g<sup>-1</sup>; Yang et al. 2010), the Pearl river delta estuary (3.8–31.7 ng g<sup>-1</sup>; Wang et al. 2009), and Bohai bay (0.20–11.1 ng g<sup>-1</sup>; Wan et al. 2005). In general, the levels of DDTs and HCHs were relatively lower than or similar to those in other Chinese rivers or lakes.

The ArcGis map (Fig. 1) showed that there was no obvious concentration variation trend of OCPs between upstream (averaged at 8.30 ng g<sup>-1</sup>), midstream (averaged at 8.10 ng g<sup>-1</sup>) and downstream (averaged at 8.21 ng g<sup>-1</sup>). There was also no concentration variation trend of HCHs and DDTs between upstream (HCHs: averaged at 7.5 ng g<sup>-1</sup>; DDTs: averaged at 0.5 ng g<sup>-1</sup>), midstream (HCHs: averaged at 7.9 ng g<sup>-1</sup>; DDTs: averaged at 0.15 ng g<sup>-1</sup>) and downstream (HCHs: averaged at 7.1 ng g<sup>-1</sup>; DDTs: averaged at 0.94 ng g<sup>-1</sup>). Furthermore, the concentration value of per site change is inapparent, it may be considered a high degree of uniformity (Fig. 1; Table 1). The concentration value of OCPs only differs by a factor of 3 which by environmental gradient would suggest a high degree of uniformity. The highest levels of OCPs were found in the samples from S8 and the second highest pollution levels existed in S5. These two sample sites locate in farmland area, so residues of OCPs may be washed away from agricultural soils. In addition, there was a pesticide factory in the past near to S8 in the Fengyang country, which led to the high residues in the nearby river basin. The lowest levels of OCPs were found in the samples from S15 and the second lowest pollution levels existed in S7. S7 is near the dam of Bengbu, thus OCPs were much easier to be washed away by water flow. The lowest concentration was investigated at S15 which suggests there are no significant input sources. Moreover, some possible factors such as historic usage, physicochemical properties may also result in the different OCP concentrations at different sampling locations (Chen et al. 2011).

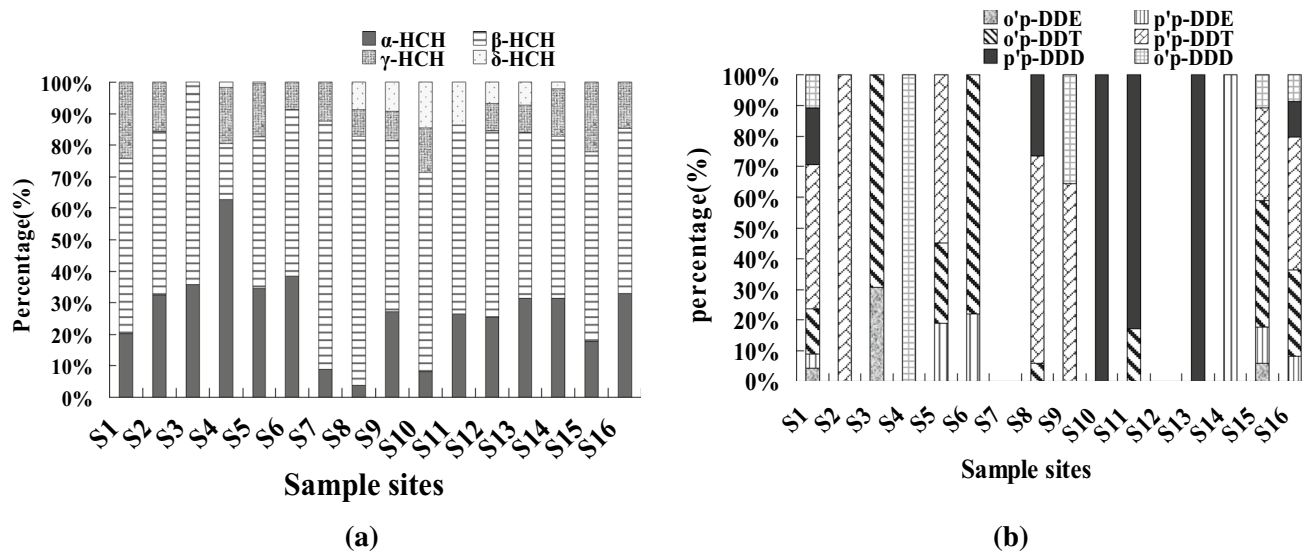
The average percentages of the different isomers are shown in Fig. 2a. The isomers of  $\alpha$ -HCH,  $\beta$ -HCH,  $\gamma$ -HCH and  $\delta$ -HCH were observed to contribute approximately 28.1%, 57.1%, 8.0% and 6.7%, respectively. The high percentage of  $\beta$ -HCH in the sediments was also recorded in other study areas, such as East China sea (Yang et al. 2010), Daliao river estuary (Li et al. 2009), Minziang river estuary, Baiyangdian lake (Dai et al. 2011), South India and the Yangtze river (Tang et al. 2013). Hinojosa-Garro et al. (2016) suggested the phenomenon for high  $\beta$ -HCH

**Table 1** Concentration of OCPs (ng g<sup>-1</sup> dw) in surface sediments

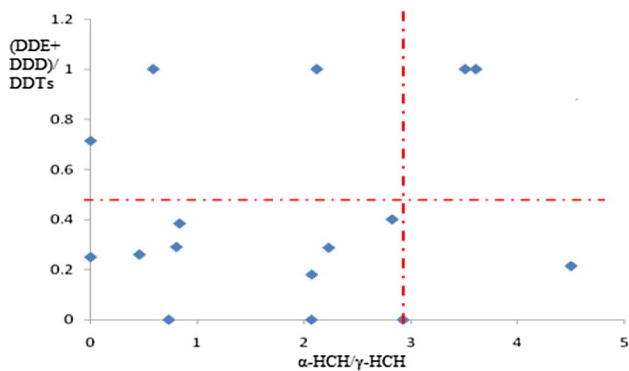
Sample code	α-HCH	β-HCH	γ-HCH	δ-HCH	∑HCH	o'p-DDE	p'p-DDE	o'p-DDT	p'p-DDT	p'p-DDT	o'p-DDD	∑DDT	HCB	Hr	He	Tc	CC	En I	En II	∑OCPs
S1	1.30	3.57	1.56	n.d	6.43	0.10	0.11	0.34	1.09	0.43	0.25	2.32	0.11	n.d	n.d	n.d	0.32	n.d	0.01	9.19
S2	2.73	4.34	1.32	n.d	8.39	n.d	n.d	n.d	0.02	n.d	n.d	0.02	n.d	n.d	n.d	0.08	n.d	0.03	0.03	8.55
S3	1.68	3.02	n.d	n.d	4.70	0.02	n.d	0.05	n.d	n.d	n.d	0.08	n.d	n.d	n.d	n.d	0.11	0.12	n.d	5
S4	4.56	1.28	1.30	0.12	7.26	n.d	n.d	n.d	n.d	n.d	0.11	0.11	n.d	0.08	n.d	n.d	n.d	n.d	0.01	7.46
S5	3.79	5.26	1.83	0.06	10.94	n.d	0.02	0.03	0.06	n.d	n.d	0.115	0.23	n.d	n.d	n.d	n.d	0.01	n.d	11.29
S6	3.42	4.72	0.76	0	8.90	n.d	0.03	0.11	n.d	n.d	n.d	0.14	n.d	n.d	n.d	n.d	n.d	n.d	0.01	9.05
S7	0.41	3.59	0.56	n.d	4.56	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	0.02	n.d	4.58
S8	0.53	11.00	1.16	1.22	13.91	n.d	n.d	0.03	0.28	0.11	n.d	0.42	n.d	0.02	n.d	0.05	n.d	n.d	n.d	14.4
S9	2.40	4.82	0.85	0.8	8.87	n.d	n.d	n.d	0.03	n.d	0.02	0.05	0.10	.005	n.d	n.d	n.d	n.d	n.d	9.02
S10	0.51	3.90	0.87	0.89	6.17	n.d	n.d	n.d	n.d	0.09	n.d	0.09	0.12	n.d	n.d	n.d	n.d	n.d	n.d	6.38
S11	1.31	2.99	n.d	0.67	4.97	n.d	n.d	0.011	n.d	0.05	n.d	0.07	n.d	0.04	n.d	0.01	0.11	n.d	n.d	5.151
S12	2.37	5.50	0.81	0.62	9.30	n.d	n.d	n.d	n.d	n.d	n.d	n.d	0.12	n.d	n.d	n.d	n.d	0.12	n.d	9.54
S13	2.67	4.44	0.74	0.61	8.46	n.d	n.d	n.d	n.d	0.09	n.d	0.09	0.09	n.d	n.d	n.d	0.05	0.11	n.d	8.8
S14	2.88	4.71	1.36	0.20	9.15	n.d	0.03	n.d	n.d	n.d	n.d	0.03	n.d	0.01	0.08	0.11	n.d	0.09	0.01	9.48
S15	0.45	1.53	0.56	n.d	2.54	0.07	0.13	0.45	0.33	n.d	0.12	1.09	0.03	0.01	0.21	0.12	0.13	n.d	0.02	4.16
S16	1.94	3.09	0.87	n.d	5.90	n.d	0.21	0.71	1.10	0.30	0.22	2.54	0.02	0.1	n.d	0.33	0.14	0.01	0.03	9.07

The data was the average of three parallel samples at each sampling site

*Hr* Heptachlor, *He* Heptachlor epoxide, *Tc* *Trans*-chlordane, *CC* *Cis*-chlordane, *En I* Endosulfan I, *En II* Endosulfan II



**Fig. 2** Compositions of HCH and DDT in the surface sediments



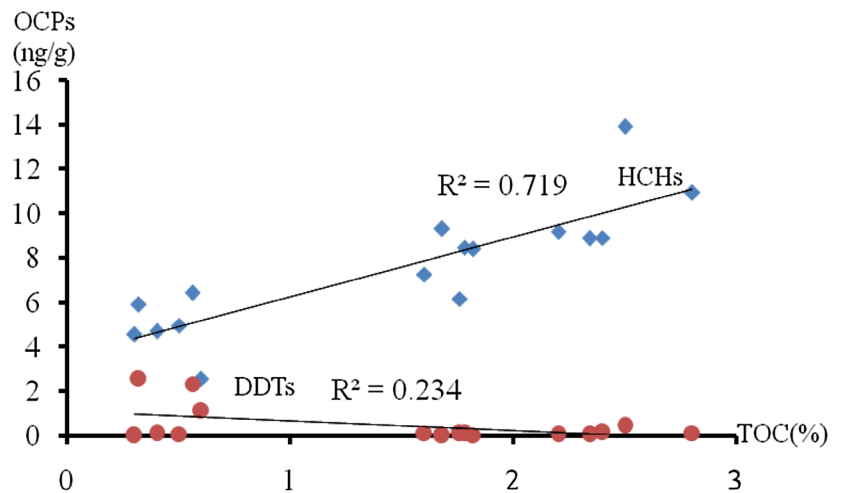
**Fig. 3** The ratio of  $\alpha$ -HCH/ $\gamma$ -HCH and (DDE+DDD)/DDTs

loading of OCPs may be due to the historical usage of HCH. Moreover, some other factors, including the lower vapor pressure, physicochemical stability and the transformation of  $\alpha$ -HCH and  $\gamma$ -HCH into  $\beta$ -HCH over time, may also contribute to the higher loading of  $\beta$ -HCH (Mahmoud et al. 2016). As seen from Fig. 3, the ratios of  $\alpha$ -HCH/ $\gamma$ -HCH in the samples were in the range of 0.58–4.5 with 75% of samples  $<3$ . This result confirmed that lindane was the major source and the use of technical HCHs was the minor source (Tang et al. 2013). It was reported that thousands of tons of lindane were used in China between 1991 and 2000 (Dai et al. 2011). Therefore, it can be concluded that lindane may be used in the upper reach of the Huaihe river. Our result is consistent with the sources of HCHs in sediments from Baiyangdian lake, China (Dai et al. 2011).

The composition of DDTs is shown in Fig. 2b, which shows that the concentration of p'p-DDT was much higher than o'p-DDT in most of samples. Similarly, p'p-DDE

and p'p-DDD were slightly higher than o'p-DDE and o'p-DDD. Moreover, p'p-DDT was the predominant isomer and existed in most of the sampling sites. This is consistent with industrial DDTs composition (Lin et al. 2016). The dominance of p'p-DDT in sediments was also reported from the Nagaon and Dibrugarh, North East India (Mishra et al. 2013), the Yangtze river, China (Tang et al. 2013), and the Candarli Gulf, Eastern Aegean (Pazi et al. 2012). As seen from Fig. 3, the mean ratio of (DDE+DDD)/DDTs were 0.498, with 68.75% of the ratio values less than 0.5, which could indicate fresh DDTs (Adeleye et al. 2016). Several recent studies reported high DDTs residues in human breast and animal tissues, and revealed a new input of DDTs, especially in South China (Zhou et al. 2011). These pollution events usually occur on a relatively small scale and are driven by water runoff.

Here, we performed linear regression analysis to obtain the correlations between OCPs and TOC. The TOC in sediments ranged from 0.3% to 2.8% with the average value of 1.47%. As seen from Fig. 4, linear regression analysis indicated significantly positive correlation between TOC and HCHs ( $r^2 = 0.719$ ) and weak negative correlation between TOC and DDTs ( $r^2 = 0.234$ ), suggesting that TOC has influence on the levels of HCHs and little influence on the levels of DDTs in the sediments (Ogbeide et al. 2015). Previous literature (Malik et al. 2014) has also reported TOC in the sediment could enhance adsorption and deposition of organic compounds. Accordingly, it is not difficult to explain the positive correlation between HCHs and TOC. Similar significant positive correlations were also reported in Owan river of Nigeria (Ogbeide et al. 2015), the Indus river (Bajwa et al. 2016) and lake Chaohu of China (Li et al. 2015). It is worth noting that there is a negative

**Fig. 4** The correlations between OCPs and TOC

correlation between TOC and DDTs in this study, which agreed with the studies by Malik et al. (2014).

A more likely explanation is that HCHs have the highest loadings over a broader area of watershed inputs and thus have been subject to weathering and environmental distribution processes leading to their partitioning behavior into organic phases within the environment.

Seventeen out of 22 OCPs were detected in the sediments collected from Anhui reach of Huaihe river. The mean concentrations of OCPs were in the following order: HCHs > DDTs > HCB > chlordanes > endosulfans. Levels of total HCHs and total DDTs ranged from 2.54 to 13.91 ng g<sup>-1</sup> (mean = 7.52 ng g<sup>-1</sup>) and 0.016 to 2.54 ng g<sup>-1</sup> (mean = 0.45 ng g<sup>-1</sup>), respectively. The concentrations of DDTs were lower than those of HCHs. Compared with the other rivers in China, DDTs and HCHs were relatively lower or similar. The OCPs concentration differences were not insignificant between upstream, midstream and downstream. Moreover, the compound compositions suggested that historical usage of lindane and fresh DDT were the main sources. The regression analysis indicated that TOC has influence on the levels of HCHs and little influence on the levels of DDTs in the sediments.

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