Acta Oceanol. Sin., 2024, Vol. 43, No. 6, P. 32–37
https://doi.org/10.1007/s13131-023-2267-6
http://www.aosocean.com
E-mail: ocean2@hyxb.org.cn

Tetrabromobisphenol A and hexabromocyclododecane in sediments from the Zhujiang (Pearl) River Estuary and South China Sea

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Received 18 April 2023; accepted 11 September 2023

Abstract

Marine sediments collected from the Zhujiang (Pearl) River Estuary (ZRE) and South China Sea (SCS) were utilized to study the occurrence and spatial distribution of tetrabromobisphenol A (TBBPA) and hexabromocyclododecane (HBCDD). The levels of TBBPA and HBCDD in sediments ranged from not detected (nd) to 6.14 ng/g dry weight (dw) and nd to 0.42 ng/g dw. TBBPA concentrations in marine sediments were substantially higher than HBCDD. The concentrations of TBBPA and HBCDD in the ZRE sediments were significantly greater than those in the SCS. α-HBCDD (48.7%) and γ-HBCDD (46.2%) were the two main diastereoisomers of HBCDD in sediments from the ZRE, with minor contribution of β-HBCDD (5.1%). HBCDD were only found in one sample from the northern SCS. The enantiomeric fraction of α-HBCDD in sediments from the ZRE was obviously greater than 0.5, indicating an accumulation of (+)-α-HBCDD. The enantiomers of HBCDD were not measured in sediments from the SCS. This work highlighted the environmental behaviors of TBBPA and HBCDD in marine sediments.

Key words: tetrabromobisphenol A, hexabromocyclododecane, marine sediments, Zhujiang River Estuary, South China Sea

Citation: Long Chuyue, Yang Weiyan, Lu Jiaxun, Cheng Yuanyue, Qiu Ning, Du Sen, Zhang Li, Chen Shejun, Sun Yuxin. 2024. Tetrabromobisphenol A and hexabromocyclododecane in sediments from the Zhujiang (Pearl) River Estuary and South China Sea. Acta Oceanologica Sinica, 43(6): 32–37, doi: 10.1007/s13131-023-2267-6

1 Introduction

Tetrabromobisphenol A (TBBPA) and hexabromocyclododecane (HBCDD) are commonly used as brominated flame retardants (BFRs) in the world (Xiong et al., 2019). More than ninety percent of TBBPA is employed as a reactive BFR in polymer resins for manufacturing printed circuit boards, and the remaining ten percent of TBBPA is utilized as an additive BFR in engineered plastics for electrical and electronics products (BSEF, 2020). TBBPA was listed in the Toxics Release Inventory by the Environmental Protection Agency of the United States and the Restriction of Hazardous Substances by the Europe Union because it can cause hepatic, renal, neural, cardiac, and reproductive toxicities to animals and human beings (Abdallah, 2016; Yu et al., 2019; Zhou et al., 2020). HBCDD is primarily used as an additive BFR in expanded and extruded polystyrene foams for thermal insulation of construction, industrial packaging, textiles, adhesives, coatings, and other products (Koch et al., 2015; Cao et al., 2018). HBCDD has the advantages of low additive amount, high flame retardant efficiency and small impact on polymer properties (Baek et al., 2017). In May 2013, HBCDD was listed as the Annex A of persistent organic pollutants (POPs) under the *Stockholm Convention* (POPRC-8/3, 2013). In the manufacture, utilization, recovery and disposal of products, TBBPA and HBCDD can be released and observed in diverse environmental substrates such as sediment, water, air, as well as living organisms (Chokwe et al., 2015; Zhang et al., 2018; Gao et al., 2019; Li et al., 2021).

Marine sediments are important sinks and sources of organic pollutants and play a very important role in the geochemical cycle of organic pollutants (Avellan et al., 2022). Large amounts of organic contaminants from land-based sources accumulate in marine sediments through various physical, chemical and biological processes. In addition, the re-releasing of organic contaminants from marine sediments may cause adverse effects on marine ecosystems and human health (Sanganyado et al., 2021).

Foundation item: The Guangdong Basic and Applied Basic Research Foundation under contract Nos 2021B1515020040 and 2021A1515011526; the National Natural Science Foundation of China under contract Nos 42277246 and U2244221; the Hainan Provincial Natural Science Foundation of China under contract No. 422CXTD533; the Key Special Project for Introduced Talents Team of Southern Marine Science and Engineering Guangdong Laboratory (Guangzhou) under contract No. 2019BT02H594.
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Monitoring organic contaminants in marine sediments is therefore critical to understand their distribution in the marine environment.

The Zhujiang (Pearl) River Estuary (ZRE) is situated in the Guangdong-Hong Kong-Macao Greater Bay Area (GBA), which has become one of the most economically developed and urbanized areas globally. The ZRE is surrounded by several developed cities, including Guangzhou, Shenzhen, Zhuhai, Dongguan, Zhongshan, Hong Kong and Macao. POPs can accumulate in ZRE and cause adverse environmental effects due to the discharge of urban sewage and industrial wastewater from the surrounding developed cities (Han and Currell, 2017). POPs in the environment of ZRE can migrate into the South China Sea (SCS) by seawater exchange, atmospheric deposition and surface runoff, which can be hazardous for marine biota (Sanganyado et al., 2018). Thus, there is an urgent need to study the environmental behavior of TBBPA and HBCDD in sediments from the ZRE and SCS. TBBPA and HBCDD were widely determined in various environmental matrices (He et al., 2013; Ruan et al., 2018; Hu et al., 2019; Li et al., 2019). TBBPA and HBCDD were investigated in the ZRE sediments collected in 2010 (Feng et al., 2012). HBCDD was also reported in sediments from the ZRE in 2013 and in marine mammals (*Neophocaena phocaenoides* and *Sousa chinensis*) from the SCS collected from 2005 to 2015 (Ruan et al., 2018; Zhu et al., 2018). In general, there are few data available on the environmental behavior of TBBPA and HBCDD in environmental media from the ZRE and SCS.

We collected surface sediments samples from the ZRE and SCS and analyzed for TBBPA and HBCDD. The aims of this work were to reveal the occurrence of TBBPA and HBCDD in sediments from the ZRE and SCS, and to investigate the diastereoisomer- and enantiomer-specific profiles of HBCDD in sediments from ZRE and SCS.

2 Materials and methods

2.1 *Sample collection*

Twenty and ten surface sediment samples were collected from the ZRE in November 2021 (S1–S20) and the SCS in March 2021 (S21–S30), respectively. The detailed sampling sites are presented in Fig. 1. The surface sediments (0–5 cm depth) were sampled onboard by a stainless steel grab sampler, packed in the aluminum foil and stored at –20℃ before chemical analysis.

2.2 *Sample preparation and cleanup*

The extraction procedure for sediments was described in our previous study (Li et al., 2021). Briefly, approximately 20 g sediment samples were spiked with surrogate standards, including $^{13}C_{12}$-TBBPA, $^{13}C_{12}$-α-HBCDD, $^{13}C_{12}$-β-HBCDD, and $^{13}C_{12}$-γ-HBCDD, and extracted by a microwave-assisted extractor with 90 mL acetone/hexane (v/v = 1:1) for 1 hour. The extraction solutions were concentrated about 1 mL in hexane, purified through a silica column packed with sulfuric acid silica (8 cm), neutral silica (8 cm), and anhydrous sodium sulfate (1 cm) and then eluted by 40 mL dichloromethane/hexane (v/v = 1:1). The eluate was concentrated, reconstituted in 200 μL of methanol, and spiked with internal standard (d_{18}-α, β, γ-HBCDD) before instrumental analysis.

2.3 *Instrumental analysis*

An Agilent 1290 liquid chromatography coupled to an Agilent 6470 triple quadrupole mass spectrometer was used to analyze TBBPA and HBCDD. TBBPA and diastereoisomers of HBCDD were separated by an XDB-C18 column (4.6 mm × 50 mm, 1.8 μm, Agilent). The mobile phases A and B were methanol/water (v/v = 9 : 1) and acetonitrile. The enantiomers of HBCDD were separated by a β-cyclodextrin column (4.0 mm × 200 mm, 5 μm, Macherey-Nagel). The mobile phases were methanol/water (v/v = 3 : 7, A), and methanol/acetonitrile (v/v = 3 : 7, B). The ion transitions (m/z) were monitored at 542.7→79 for TBBPA, 554.7→79 for $^{13}C_{12}$-TBBPA, 640.7→79 for HBCDD, 652.7→79 for $^{13}C_{12}$-HBCDD, and 657.7→79 for d_{18}-HBCDD, respectively. The analytes were quantified by the relative response to the isotopically labeled standard in a five-point calibration curve.

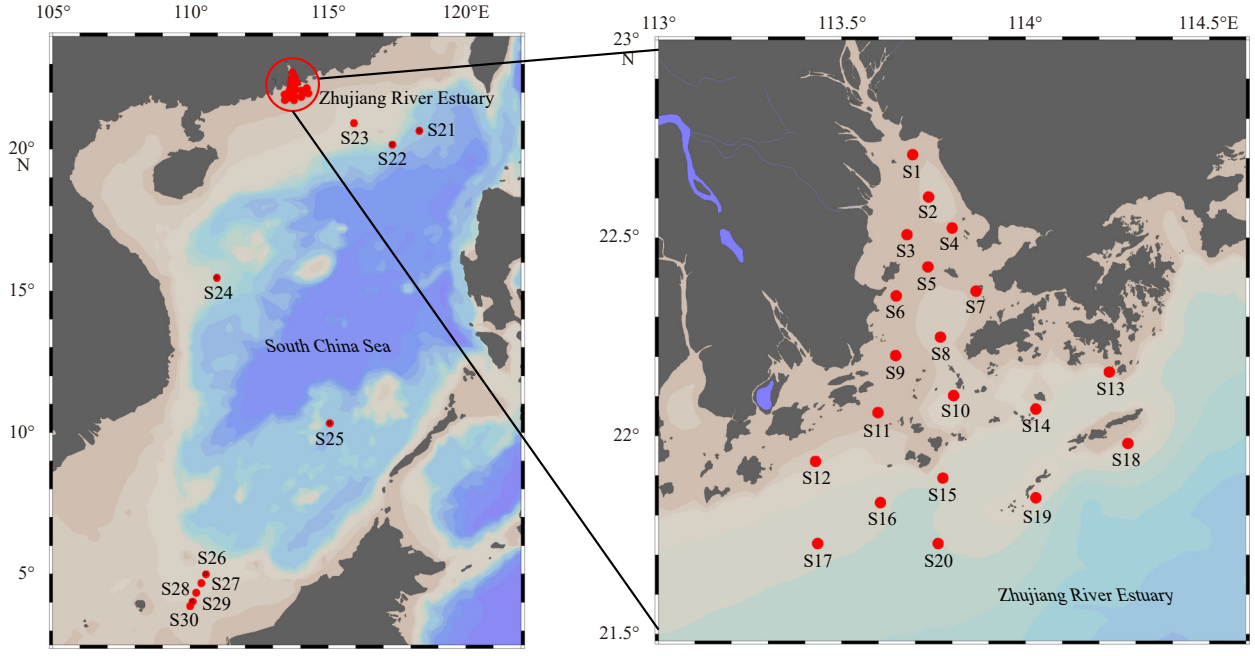

Fig. 1. Sampling sites distribution.

2.4 *Quality assurance/quality control*

Each batch of 11 samples is processed with one procedural blank. Recoveries in matrices and solutions were evaluated by spiking TBBPA, α-HBCDD, β-HBCDD and γ-HBCDD. The mean recoveries (mean ± SE) of TBBPA, α-HBCDD, β-HBCDD and γ-HBCDD in spiked blanks (n = 3) were 94.1% ± 9.6%, 98.7% ± 2.5%, 97.2% ± 5.4%, 102.8% ± 2.7% and in spiked matrices (n = 3) were 81.0% ± 3.4%, 81.8% ± 7.2%, 70.3% ± 5.1%, 64.1% ± 5.6%, respectively. The average recoveries of $^{13}C_{12}$-TBBPA, $^{13}C_{12}$-α-HBCDD, $^{13}C_{12}$-β-HBCDD and $^{13}C_{12}$-γ-HBCDD in all sediment samples (n = 30) were 94.6% ± 2.7%, 89.9% ± 5.8%, 83.3% ± 2.4% and 107.7% ± 3.9%, respectively. The procedural blanks had no detection of TBBPA and HBCDD. The instrument limit of detection (IDL) was set as five times the noise level. The IDLs for TBBPA, α-HBCDD, β-HBCDD and γ-HBCDD were 0.10 ng/mL, 0.83 ng/mL, 0.21 ng/mL and 1.09 ng/mL, respectively. Based on the average dry weight of the sediment samples, the method detection limits (MDLs) for TBBPA, α-HBCDD, β-HBCDD, and γ-HBCDD were 0.001 ng/g, 0.008 ng/g, 0.002 ng/g and 0.011 ng/g dry weight (dw), respectively.

2.5 *Data analysis*

Origin 2021 (OriginLab Corporation, USA) and Ocean Data View 5.5.1 (Alfred Wegener Institute, Helmholtz Centre for Polar and Marine Research) software were used to draw figures and map. SPSS 26.0 software (SPSS Inc. Illinois, USA) was used to perform statistical analysis. Spatial difference in TBBPA and HBCDD concentrations between the ZRE and SCS was analyzed by the Independent-samples T-test. Relationship between TBBPA and HBCDD in marine sediments was tested by linear regression analysis. The significance level was accepted as $p < 0.05$.

3 Results and discussion

3.1 *Distribution of TBBPA in marine sediments*

TBBPA was found in all sediment samples from the ZRE, and its concentration ranged from 0.11 ng/g to 6.14 ng/g dw, with an average value of 0.57 ng/g dw. The highest concentration of TBBPA (6.14 ng/g dw) was found in sediment at Station S1, which located in the upper reaches of the ZRE and was influenced by the two developed cities of Guangzhou and Dongguan. Station S2 is situated in the downstream of Station S1 and also contained higher level of TBBPA (1.12 ng/g dw). TBBPA concentrations in our study were substantially greater than the ZRE sediments sampled in 2010 with range and mean concentrations of 0.06–1.39 ng/g dw and 0.47 ng/g dw (Feng et al., 2012), indicating that TBBPA was extensively used in the GBA of China. The gross domestic product (GDP) of the GBA in 2021 were 12.6 trillion RMB, with a population of 86.7 million. It has become one of the most economical and urban-dynamic regions all over the world. The rapid industrialization and urbanization of this region may result in TBBPA contamination in the ZRE. TBBPA was only determined in three sediment samples at Stations S23, S24 and S26 from the SCS with concentrations of 0.14 ng/g, 0.16 ng/g and 0.11 ng/g dw, respectively (Fig. 2). TBBPA levels in the SCS sediments were conspicuously lower than the ZRE (0.11–6.14 ng/g dw) (p = 0.027). Up to now, the use and production of TBBPA is not restricted in China. The contamination of TBBPA in the ZRE should be continuously focused on.

3.2 *Distribution of HBCDD in marine sediments*

HBCDD were detected in 19 of 20 sediments from the ZRE except Station S5 (Fig. 3). The total concentrations of HBCDD in sediments from the ZRE ranged from not detected (nd) to 0.42 ng/g dw, with an average of 0.19 ng/g dw. The highest level of ΣHBCDD was also found in sediment at Station S1. The content of ΣHBCDD in our study were obviously lower than those in the ZRE sediments sampled in 2010 with range and mean concentrations of nd to 1.08 ng/g dw and 0.50 ng/g dw (Feng et al., 2012). The use and production of HBCDD were prohibited in China from December 2016, but HBCDD can be only applied as

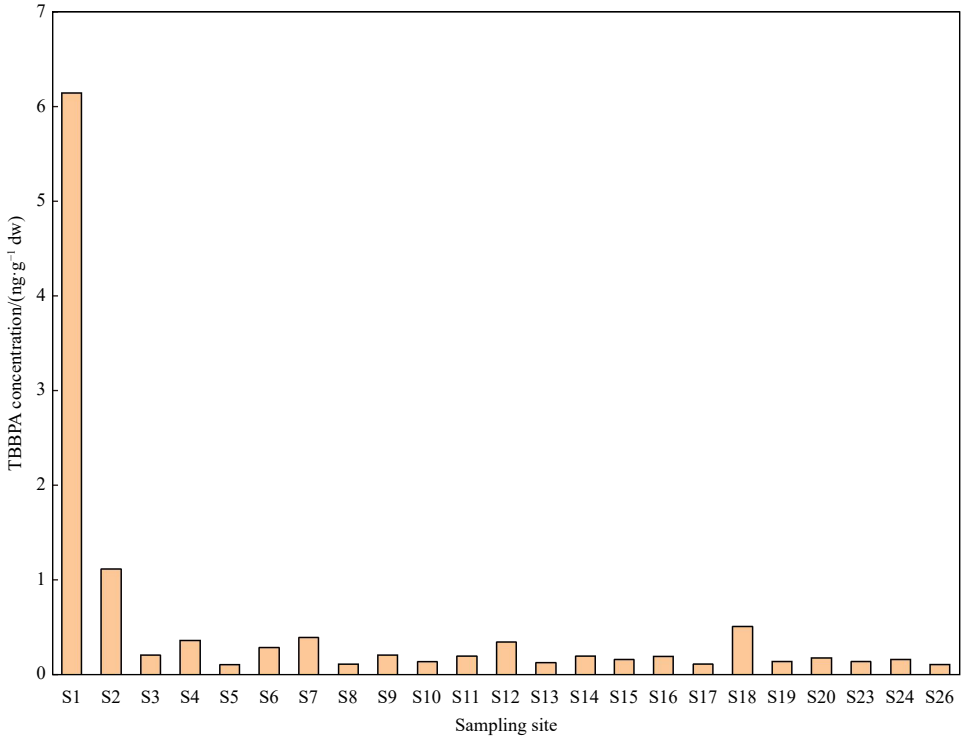

Fig. 2. TBBPA concentrations in sediments from the ZRE and SCS.

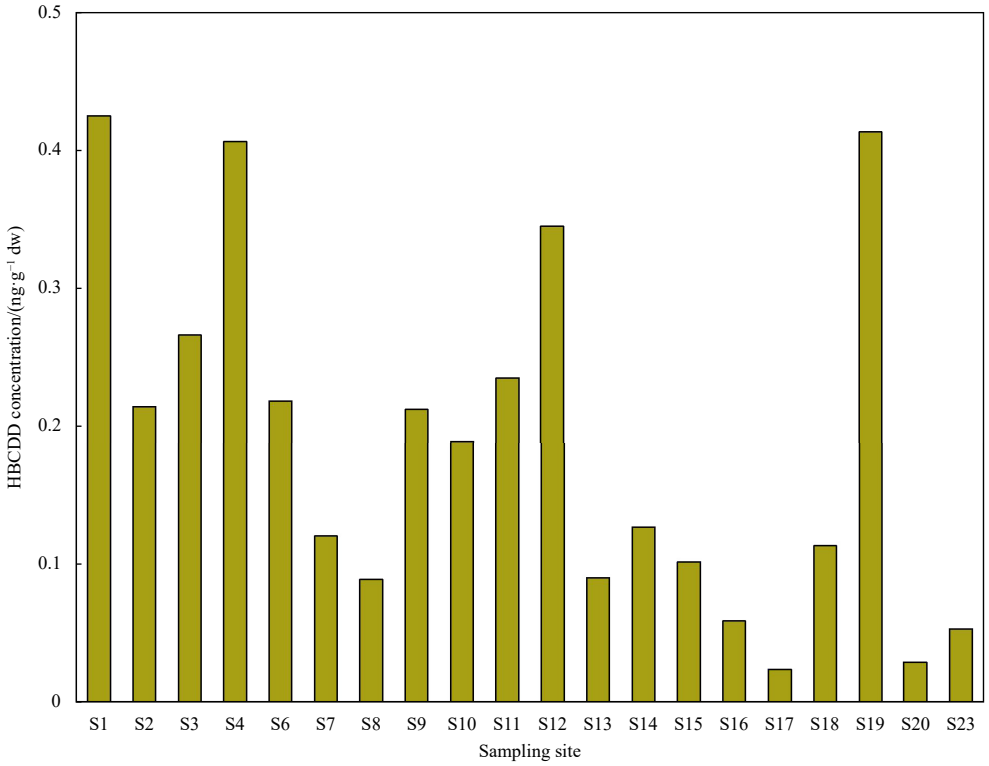

Fig. 3. HBCDD concentrations in sediments from the ZRE and SCS.

expanded polystyrene and extruded polystyrene in building materials from December 2016 to December 2021 (Ministry of Ecology and Environment of the People's Republic of China, 2021). The decrease of HBCDD concentrations in sediments of the ZRE from 2010 to 2021 can be ascribed to the phasing out of HBCDD in China. For the SCS, HBCDD was only detected in sediment at Station S23 with concentration of 0.05 ng/g dw (Fig. 3), which was conspicuously lower than the ZRE sediments. HBCDD levels were significantly associated with TBBPA concentrations in sediments from the ZRE and SCS (p = 0.042), indicating that these two BFRs had similar environmental behavior and/or sources.

3.3 *Diastereoisomer profiles of HBCDD in marine sediments*

The diastereoisomer compositions of HBCDD in sediments from the ZRE and SCS are displayed in Fig. 4, α-HBCDD and γ-HBCDD which were the two main diastereoisomers of HBCDD in sediments from the ZRE with proportions ranging from 13.9% to 74.2% and 21.6% to 84.2%, with average contributions of 48.7% and 46.2%. The percentage contribution of β-HBCDD varied from 1.8% to 12.0%, with a mean of 5.1%, which was much lower than that of α-HBCDD and γ-HBCDD. HBCDD was only detected in one sediment sample at Station S23 from the SCS with proportions of 40.6%, 2.0% and 57.4%, respectively. The fractions of α-, β- and γ-HBCDD in commercial products measured in our laboratory were 6.3%, 8.8% and 84.9%, respectively (Sun et al., 2012). The percentage of α-HBCDD increased and the fractions of γ-HBCDD decreased in sediments from the ZRE and SCS by comparison with the commercial products, suggesting that microbial transformation and/or degradation of HBCDD diastereoisomers probably exist in sediments from the ZRE and SCS (Davis et al., 2006).

3.4 *Enantiomeric fractions of HBCDD in marine sediments*

The enantiomer fraction (EF) was calculated by the peak areas of enantiomer pairs using the following formula (Li et al., 2019):

$$\mathrm{EF} = \frac{\left(\frac{A_+}{A_{+d_{18}}}\right) \times C_{+d_{18}}}{\left(\frac{A_+}{A_{+d_{18}}}\right) \times C_{+d_{18}} + \left(\frac{A_-}{A_{-d_{18}}}\right) \times C_{-d_{18}}},$$

where A_+, A_-, $A_{+d_{18}}$ and $A_{-d_{18}}$ stand for the peak areas of the (+) enantiomer, (–) enantiomer, d_{18}-labeled-(+) enantiomer and d_{18}-labeled-(–) enantiomer; $C_{+d_{18}}$ and $C_{-d_{18}}$ were the levels of d_{18}-labeled-(+) enantiomer and d_{18}-labeled-(–) enantiomers. The EF values of α-HBCDD, β-HBCDD, and γ-HBCDD in the ZRE sediments were in the range of 0.508–0.723, 0.451–0.585, and 0.467–0.566, with average values of 0.563 ± 0.011, 0.503 ± 0.009, and 0.507 ± 0.006, respectively (Fig. 5). The EF values of α-HBCDD in the ZRE sediments were substantially greater than 0.5 (p < 0.001), indicating a preferential accumulation of (+)-α-HBCDD. There were no significant difference between the EF values of β-HBCDD (p = 0.74) and γ-HBCDD (p = 0.24) in the ZRE sediments and those in the commercial products, which indicates that sediment samples were racemic mixture. The enantiomers of α-HBCDD, β-HBCDD and γ-HBCDD in sediments from the SCS were not measured due to the low concentrations and detection frequency. Difference in EF values of HBCDD in sediments may be related to the distinct biological processes, such as degradation/transformation.

4 Conclusions

TBBPA and HBCDD have been measured in sediments from the ZRE and SCS. The concentrations of TBBPA in sediments were substantially higher than that of HBCDD. TBBPA concentrations in sediments from the ZRE increased from 2010 to 2021, while the concentrations of HBCDD decreased. α-HBCDD was

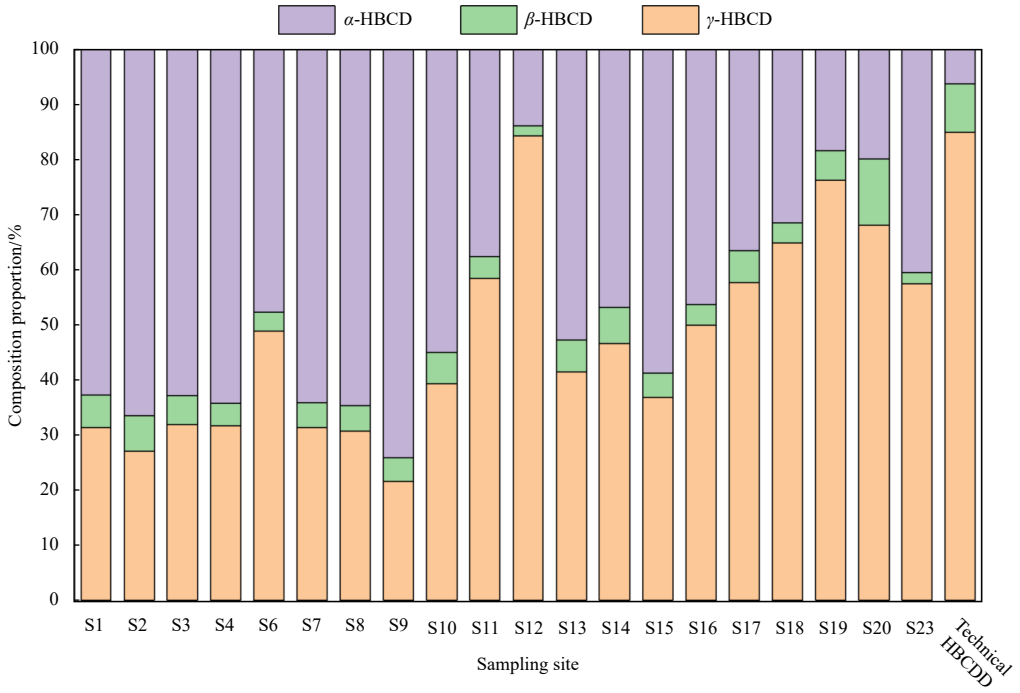

Fig. 4. Diastereoisomer compositions of HBCDD in sediments from the ZRE and SCS.

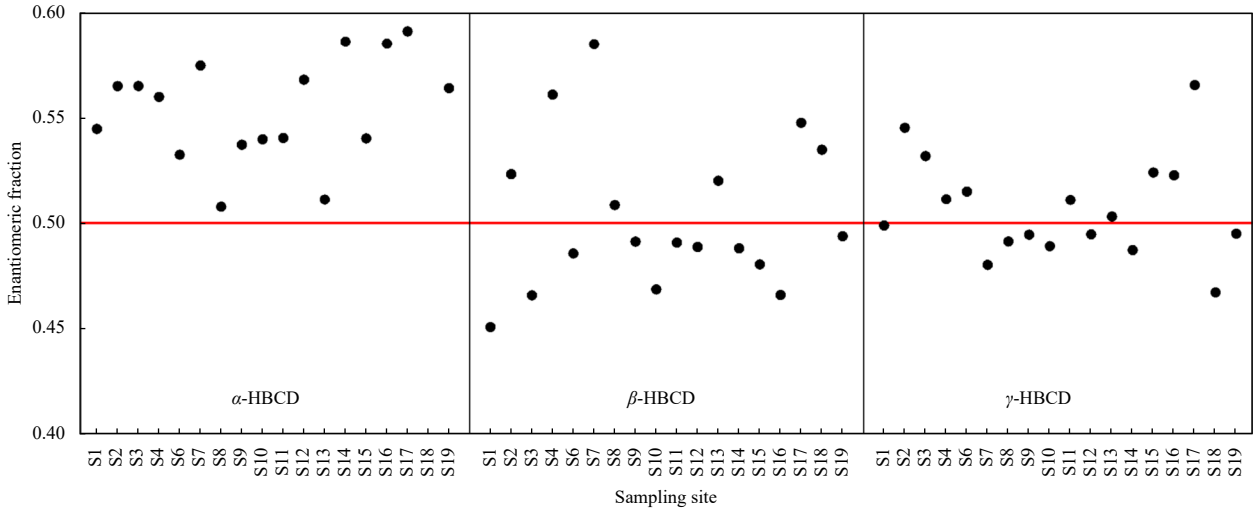

Fig. 5. Enantiomer fractions of HBCDD in sediments from the ZRE.

the dominant diastereoisomer of HBCDD in sediments from the ZRE (48.7%), followed by γ-HBCDD (46.2%), with minor contribution of β-HBCDD (5.1%). The EF values of α-HBCDD in the ZRE sediments were obviously greater than those the commercial products, showing a preferential enrichment of (+)-α-HBCDD relative to (−)-α-HBCDD. The enantiomers of α-, β- and γ-HBCDD were not measured in sediments from the SCS.

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