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Occurrence, source identification and risk assessment of polybrominated diphenyl ethers in water and sediments of Osun River, Nigeria

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Abstract

Background In the present study, the concentrations of polybrominated diphenyl ethers (PBDEs) in water and sediments of Osun River, Nigeria were determined.

Methods The obtained water samples were subjected to liquid-liquid extraction while the sediment samples were subjected to extraction using n-hexane and acetone. The extracts were quantified using a Gas Chromatography-Mass Spectrometer to assess their distribution, source identification, and ecological risk assessments.

Results The results of the study indicated that BDE-99, BDE-183, and BDE-209 were not detected in the water samples while BDE-28 had the most significant mean contribution (0.057 mg/L w.w) to the total PBDEs in the water samples. The sediment samples were dominated by enhanced concentrations of BDE-100 congener (mean concentration 0.184 mg/kg d.w). The compositional profiles of the PBDE congeners indicated the abundance of higher BDEs in the sediment samples compared to the water samples, a phenomenon attributed to the lower reduction rate and lower water solubility of the higher BDEs. Source identification indicated that commercial deca-BDE mixtures might represent the major source of PBDEs in the studied river. Risk assessment of PBDEs in water samples showed that the mean concentrations of tri-BDE, tetra-BDE, penta-BDE, and hexa-BDE exceeded the acceptable limits, suggesting adverse health risks to the public. Ecological risk assessment of PBDEs in sediment samples showed medium ecological risk by hexa-BDE ($0.1 \leq RQ \leq 1$) and high ecological risks due to tri-, tetra-, penta-, and deca-BDEs ($RQ > 1$).

Conclusion The study concludes that efficient and cost-effective remediation strategies should be developed and employed in the abatement of these persistent organic pollutants from the environment.

Keywords Polybrominated diphenyl ethers, Risk assessment, Sediments, Source identification, Water

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Background

Persistent organic pollutants (POPs) are organic compounds that have a wide occurrence in the environment due to their resistance to environmental degradation. As a result of their high lipophilicity, they can bioaccumulate in the tissues of aquatic organisms upon exposure. Many organic compounds comprising polybrominated diphenyl ethers, organochlorine pesticides, polychlorinated biphenyls, and polycyclic aromatic hydrocarbons belong to this class [1, 2]. While some of these POPs are products of combustion (such as polycyclic aromatic hydrocarbons), others are synthesized for use as agrochemicals

or other industrial purposes (such as organochlorine pesticides and polybrominated diphenyl ethers). Even though they fulfill the intended industrial and economic use, their bioaccumulation in biota and biomagnification up the food chain poses serious threats to humans and other organisms [3].

Polybrominated diphenyl ethers (PBDEs) belong to a class of brominated flame retardants that are usually fused in several products such as electronics, construction products, furniture, and plastics in a bid to reduce their inflammability [4–6]. Although PBDEs have shown enormous resourcefulness in lowering the combustive properties of products, their ability for long-range atmospheric transport, bioaccumulation, persistence, and toxicity have been a concern over the years [7]. Owing to these properties which have negative effects on human health, many developed countries have phased out its use in industrial production. For instance, octa-BDE, penta-BDE, and deca-BDE have been included in the list of persistent organic pollutants due to their ubiquitous nature and potential health risks [8]. Due to the inability of PBDEs to form chemical bonds, they easily get into the environment during their use in production and ultimately waste disposal [9]. For example, BDE-209 is an important component of deca-BDE that undergoes debromination by microorganisms under the influence of light to lowly brominated diphenyl ethers. However, the higher brominated diphenyl ethers are relatively less toxic compared to lowly brominated diphenyl ethers [10].

River sediments are reported to be the final sink for many environmental pollutants including persistent organic pollutants [11]. Exposure to PBDEs can have devastating effects on humans such as neural developmental disorders, cardiovascular injury, thyroid hormone disorders, endothelial dysfunction, and liver deiodinase inhibitor [12, 13].

Therefore, the monitoring of these pollutants in water and sediments is required for a proper understanding of their fate in the environment. Many recent studies have been carried out all over the world to assess the distribution of PBDEs in water bodies [14–18]. However, there is a paucity of environmental studies addressing the PBDE levels in the water and sediments of Nigerian water bodies. The present investigation is the first known attempt at characterizing PBDEs in water and sediments of Osun River, Nigeria. Assessing PBDE levels in the Osun River is critical for determining the extent of contamination and the possible consequences to local ecosystems and those that rely on it for water, food, and recreation. This study provides critical baseline data that can help policymakers and environmental regulators understand the prevalence and concentration levels of PBDEs. This knowledge is critical for creating effective laws and mitigation measures to manage and reduce PBDE contamination. This study contributes data from an underrepresented location, which improves the global database of PBDE contamination. This information is invaluable for conducting comparative research and understanding worldwide patterns and sources of PBDE pollution. In this study, the occurrence and compositional profiles of PBDEs were estimated. The source identification and ecological risk assessment were then evaluated using the PBDE concentrations.

Methods

Study area

The study area is Osun River situated in Osogbo, Osun State, Nigeria. Osogbo is characterized by a tropical rainforest with a population of 156,694 (NPC, 2006). It has an average annual rainfall of 350 mm and a temperature that ranges between 19 °C and 38 °C depending on the season. Osun River flows through several rainforests, farmlands and villages before Osogbo in Osun State and also through many states in Southwestern Nigeria before entering into the Lagos Lagoon and the Atlantic Gulf of Guinea. The sampling points in the river as shown in Fig. 1 encompass Ofatedo, Aregbesola, Isale-Osun, Fountain, Osun-Jela, Kajola and Koka village which were different channels along the river course from upstream to downstream having proximity with community activities.

Sample collection and preparation

Seven regions along the river course were chosen for sampling. Water samples were taken from specific locations in Ofatedo, Aregbesola, Isale-Osun, Fountain, Osun-Jela, Kajola, and Koka between May and July 2022. To ensure sample purity, 1 L amber bottles were pre-cleaned using hexane and acetone to eliminate organic impurities. After thoroughly cleaning with deionized water, sterilised stainless containers were used to collect samples from around 10 cm below the water's surface. Sterilisation was accomplished using autoclaving and UV light exposure. To prevent bacterial activity and volume changes, the samples were stored in sealed, PBDE-free bags at temperatures around 4 °C.

Surface sediments were collected using a stainless-steel grab sampler. The samplers were thoroughly cleaned, including solvent rinsing with hexane and acetone, followed by deionized water rinses and autoclaving. The prepared samplers were kept in a contamination-free area until needed. The grab sampler was used to capture sediment samples from depths of less than 15 cm. After collection, materials were freeze-dried, homogenised, sieved through a 63 µm mesh, and kept at -20 °C in a polythene bag before extraction.

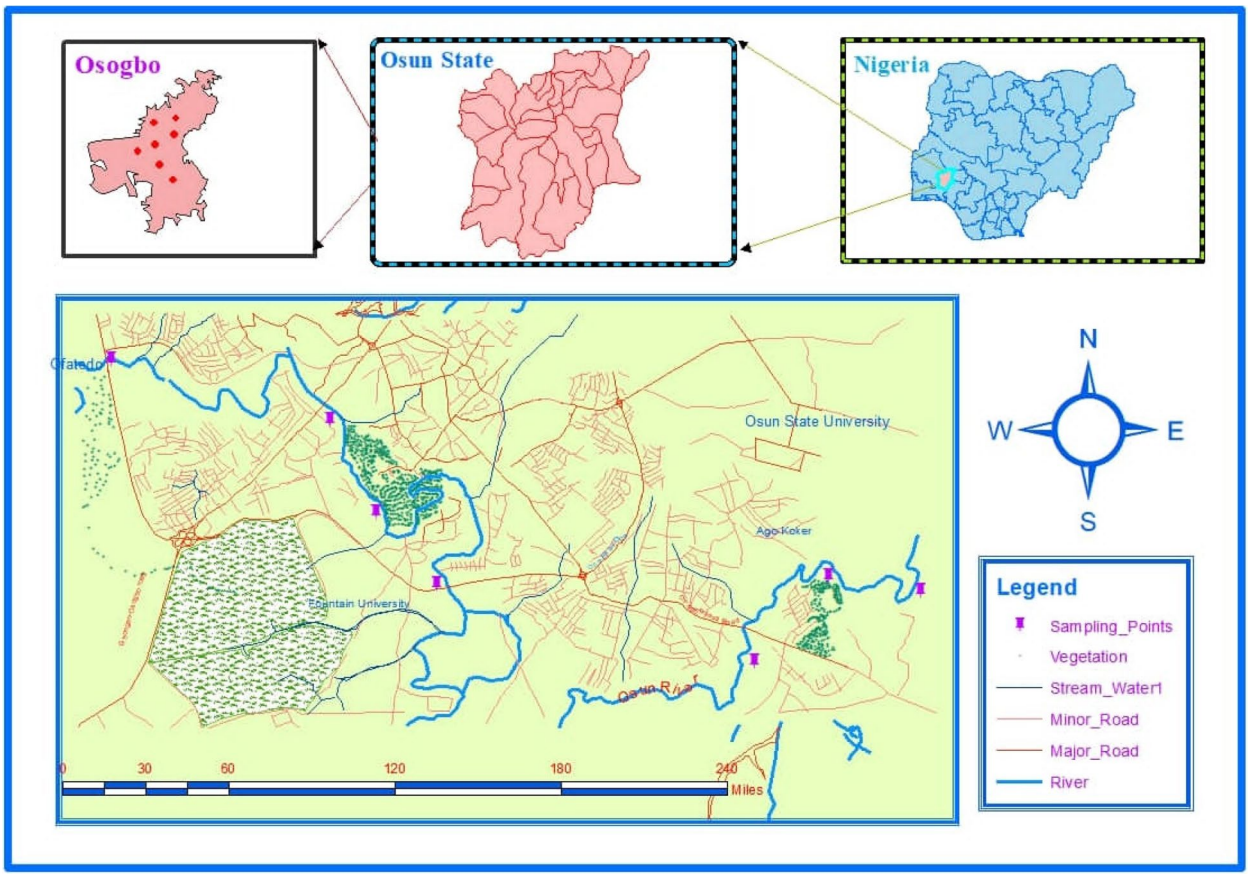

Fig. 1 Map of the study area showing sampling points along the river course

To confirm the absence of PBDE contamination, samples were collected with properly cleaned equipment and analysing them for PBDEs. The results revealed no detectable PBDEs, demonstrating the efficacy of our cleaning techniques. We meticulously assured that all sample collection, transportation, and processing equipment was free of PBDE contamination using stringent protocols. By maintaining this level of cleanliness, we ensured the integrity and dependability of our study's findings.

Chemicals and reagents

The chemicals and reagents used in the present study include acetone (BDH Poole House, England), n-hexane (BDH Poole House, England), silica-gel (MERCK, Germany), anhydrous sodium sulphate (BDH Poole House, England), and dichloromethane (DCM) (Sigma-Aldrich, South Africa).

Extraction of PBDEs, clean up and instrumental analysis

Liquid–liquid extraction techniques were used to extract PDBEs from the water samples by shaking 500 mL of the sample with 30 mL of DCM. Three consecutive extractions were carried out and the extracts were combined and concentrated down to 2 mL using a rotary evaporator. For the sediment samples, 5 g of properly homogenized samples were weighed into beakers and mixed with 10 mL n-Hexane: Acetone in a ratio of 1:1. The beakers were then placed into an ultrasonic bath and sonicated for 20 min. The sonication was carried out thrice. The mixture was then allowed to settle and the solvent layer was decanted and concentrated to 2 mL using a rotary evaporator.

PBDE standard, containing 8 PBDEs components was purchased from AccuStandard. The GC–MS was calibrated using five (5) point serial dilution calibration standards (0.1, 0.31, 0.625, 1.250, 2.500 ppm) which was prepared from the stock solution. The auto-tuning of the mass spectrometer to perfluorotributylamine (PFTBA) was carried out using established criteria to check the abundance of m/z 69, 219, 502 and other instrument's optimal and sensitivity conditions prior to calibration. The concentrations of PBDEs in the respective samples were determined using the selective ion monitoring and scan mode of the GC–MS in a bid to ensure that the target constituents were detected even

at low levels. The instrumental determination of the PBDE concentrations using GC–MS is described by Benson et al. [19]. The determination of target PBDEs in water and sediment samples involved using GC–MS in selective ion monitoring (SIM) and scan mode for sensitive detection. We utilized an Agilent 7820A gas chromatograph coupled to a 5975C inert mass spectrometer with a triple-axis detector and an electron-impact source. The separation occurred on an HP-5 capillary column (30 m length, 0.32 mm diameter, and 0.25 µm film thickness). Helium served as the carrier gas at a constant flow rate of 1.48 mL/min, maintaining an initial nominal pressure of 1.49 psi and an average velocity of 44.22 cm/s. Sample injection (1 µL) was performed in splitless mode at an injection temperature of 300 °C. The mass spectrometer operated at 70 eV in electron-impact ionization mode, with specific ion source, quadrupole, and transfer line temperatures. After calibration, the samples were analysed by comparing the retention times with those of reference standards to obtain the corresponding PBDE concentrations. The determined PBDEs include BDE-28, BDE-47, BDE-99, BDE-100, BDE-153, BDE-154, BDE-183, and BDE-209.

For the clean-up procedure, granular silica gel (Mesh Size 60-200A) was activated by heating at 130^0 C for 16 h and stored in a desiccator. A glass column was packed with 5 g of silica gel and 1 g of anhydrous Na_2SO_4 was added. 20 mL n-hexane was added to the column and eluted into a beaker. The 2 mL sample extract was added to the top of the column quantitatively. Another 10 mL of n-hexane was added to the column and eluted to waste. Before the column head dried out, 10 mL (1 + 1) Dichloromethane + Hexane was added and the eluent was collected. The eluent was then concentrated to 2 mL using a rotary evaporator for PBDE analysis.

Quality assurance/control

For the purpose of quality assurance and control, matrix spiked samples and procedural blanks were determined. Individual PBDE congener concentrations were measured using external calibration procedures based on five-point standard curves. Notably, recoveries of target compounds in matrix spiked samples ranged from 88 to 107% of certified levels. This finding indicates the reliable extraction efficiency and analytical precision of the adopted method. In addition, procedural blanks are commonly assessed in environmental contamination analyses. In this study, trace levels of BDE-28 and BDE-153 were detected in the blanks. To compensate for background contamination, the mean concentrations were subtracted from the sample results. Furthermore, eliminating procedural blank values helps to reduce potential contamination during sample preparation and analysis, in line with widely accepted guidelines.

Statistical analysis and data treatment

Mean and standard deviation were computed to estimate the variability of the data set. One-way analysis of variance was also computed to determine the significant differences between the samples. Origin Version 2022 was the software used for data treatment and plotting of variation diagrams.

The ecological risks of PBDEs in sediments were assessed by risk quotient (RQ) in accordance with Federal Sediment Quality Guidelines (FSeQGs) [20]. The calculation formula was expressed in Eq. (1):

$$RQ = \frac{C_i}{C_{si}} \tag{1}$$

where C_i is the measured concentration of PBDE in sediments, C_{si} is the standard concentration according to FSeQGs, and RQ is the risk quotient of PBDE in the sediments [20]. Four levels of ecological risks are obtainable: $RQ < 0.01$ indicates no ecological risk; $0.01 \leq RQ \leq 0.1$ indicates low ecological risk; $0.1 \leq RQ \leq 1$ indicates

Table 1 Levels (mg/L) of polybrominated diphenyl ethers in water samples

Samples	BDE-28	BDE-47	BDE-100	BDE-99	BDE-154	BDE-153	BDE-183	BDE-209	∑*PBDEs*
A	0.09	0.18	0.10	ND	0.10	0.13	ND	ND	0.60
B	0.08	0.10	0.10	ND	0.11	0.12	ND	ND	0.51
C	0.14	ND	ND	ND	ND	ND	ND	ND	0.14
D	0.08	ND	ND	ND	ND	ND	ND	ND	0.08
E	0.01	ND	ND	ND	ND	ND	ND	ND	0.01
F	ND	ND	ND	ND	ND	ND	ND	ND	ND
G	ND	ND	ND	ND	ND	ND	ND	ND	ND
Mean ± SD	0.057 ± 0.054	0.04 ± 0.072	0.028 ± 0.048	ND	0.03 ± 0.051	0.035 ± 0.061	ND	ND	

Table 2 Levels (mg/kg) of polybrominated diphenyl ethers in sediment samples

Samples	BDE-28	BDE-47	BDE-100	BDE-99	BDE-154	BDE-153	BDE-183	BDE-209	∑*PBDEs*
A	0.09	0.11	0.09	0.14	0.10	0.16	0.11	0.11	0.91
B	0.13	0.14	0.23	0.13	0.12	0.15	0.14	0.12	1.16
C	0.11	0.13	0.18	0.14	0.11	0.16	0.11	0.12	1.06
D	0.09	0.13	0.17	0.15	0.12	0.17	0.11	0.11	1.05
E	0.10	0.11	0.41	0.15	0.11	0.15	0.11	0.11	1.25
F	0.09	0.14	0.10	0.19	0.14	0.21	0.11	0.14	1.12
G	0.09	0.11	0.11	0.14	0.11	0.14	0.10	0.11	0.91
Mean ± SD	0.1 ± 0.015	0.124 ± 0.013	0.184 ± 0.111	0.148 ± 0.019	0.115 ± 0.012	0.162 ± 0.022	0.112 ± 0.012	0.117 ± 0.011	

medium ecological risk; and RQ > 1 indicates high ecological risk [21].

Results

Concentration of PBDEs in Water and Sediments

The levels of polybrominated diphenyl ethers (PBDEs) in the studied water and sediment samples are presented in Tables 1 and 2 respectively. Eight BDEs comprising BDE-28, BDE-47, BDE-99, BDE-100, BDE-153, BDE-154, BDE-183, and BDE-209 were identified in the samples. In the water samples, the mean concentrations of PBDE ranged from ND to 0.057 mg/L. BDE-99, BDE-183, and BDE-209 were not detected in the water samples while the most significant contributions emanated from the levels of BDE-28 (mean concentration of 0.057 mg/L). The mean levels of BDE-47, BDE-100, and BDE-153 were 0.04, 0.028, and 0.035 mg/L respectively.

Also, the sediment samples highlight the presence of eight PBDEs. The mean concentrations of BDE-28, BDE-47, BDE-99, BDE-100, BDE-153, BDE-154, BDE-183, and BDE-209 were 0.1, 0.124, 0.148, 0.184, 0.162, 0.115, 0.112, and 0.117 mg/kg respectively.

Compositional Profiles of PBDEs in Water and Sediments

The respective classes of PBDEs were grouped according to the number of bromine atoms present in each molecule. Based on this classification, six classes of PBDE congeners comprising tri-BDE (BDE-28), tetra-BDE (BDE-47), penta-BDE (BDE-99 and BDE-100), hexa-BDE (BDE-153 and BDE-154), hepta-BDE (BDE-183), and deca-BDE (BDE-209) were identified. The compositional profiles of PBDE congeners in water and sediment samples are shown in Figs. 2 and 3 respectively. Tri-BDE dominated over 42% of the water samples while slightly over 28% of the water samples did not reflect the presence of the PBDE congeners. Similarly, about 28% of the water samples were equally dominated by hexa-BDE and tetra-BDE.

In the sediment samples, penta- and hexa-BDE represented the dominant forms of congeners exhibiting wide occurrence.

Source identification of PBDEs in Water and Sediments

The possible sources of PBDEs in water and sediments of the studied river were estimated using the Pearson correlation matrix (Table 3).

Furthermore, the primary input of PBDEs in sediments can be estimated using the ratios of BDE-47, BDE-99, and BDE-100 [22]. The plot of diagnostic ratios is shown in Fig. 4. The ratios of BDE-47/(BDE-47+BDE-99) ranged from 0.42 – 0.51 with a mean ratio of 0.45 in the sediment samples. The mean ratio is higher than the mean ratio of the technical mixture of penta-BDE, suggesting the degradation of PBDE congeners such as BDE-99 [23]. Similarly, the ratios of BDE-99/BDE-100 ranged from 0.36 – 1.9 with a mean ratio of 1.04, and this was equally higher than the mean ratio of penta-BDE.

Ecological risk assessment of PBDEs in water and sediment samples

The contamination associated with PBDEs in the water samples was estimated via comparison with the acceptable levels set by the Federal Environmental Quality Guidelines [24]. The acceptable levels of tri-BDE, tetra-BDE, penta-BDE, hexa-BDE, and hepta-BDE are 46, 24, 0.2, 120, and 17 ng/L respectively [25].

The ecological risk assessment of PBDEs in sediment samples was estimated using risk quotient (RQ) on non-target organisms [26, 27]. The calculated RQ values are shown in Fig. 5.

Discussion

The observed concentration of PBDEs in the studied water samples are relatively higher than those reported in other water samples [23, 28–30]. The surge in the PBDE levels of the studied river may have emanated from the presence of electrical devices, textiles, and plastics. A direct discharge of wastes containing these products and/or an indirect discharge of wastewater from manufacturing industries may account for the presence of these congeners in the studied river as deca-BDE (BDE-209) products are commonly used in the manufacture of the aforementioned products [5, 31].

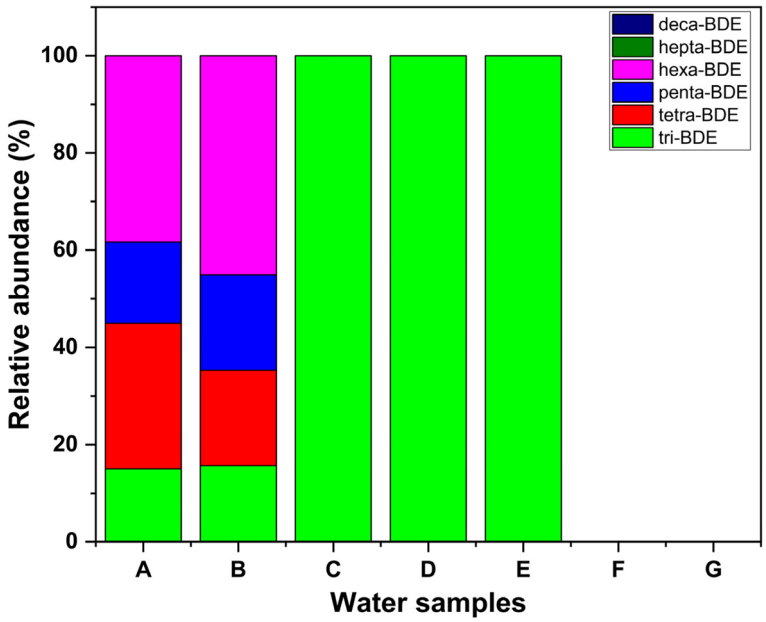

Fig. 2 Compositional profile of PBDE congeners in water samples

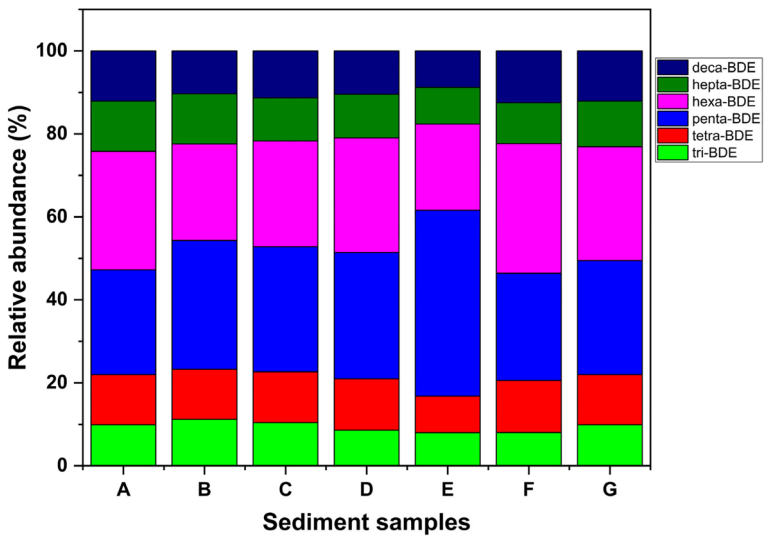

Fig. 3 Compositional profile of PBDE congeners in sediment samples

Although the levels of PBDEs in the sediments were all very high, BDE-100 and BDE-153 were the dominant congeners in the sediment samples. The PBDE levels of sediment samples in the present study were relatively higher than those reported in previous studies [32–35]. One-way analysis of variance (Table S1) indicated that the mean concentrations of PBDEs in the sediment samples were significantly higher than in the water samples ($p < 0.05$) except for BDE-28. The relatively high concentrations of PBDEs in the sediment samples could be attributed to their hydrophobic nature, which explains their predominantly adsorbed nature to suspended particles and ultimately sediments once released into the environment either through accidental discharge during production or refuse dumping/mismanagement [6]. The high levels of PBDEs in the samples are widely associated with an enhanced degree of urbanization which directly translates to increased industrial activities and accumulation of electronic waste [36].

Table 3 Pearson correlation matrix of polybrominated diphenyl ethers in water and sediment samples

	BDE-28	BDE-47	BDE-100	BDE-99	BDE-154	BDE-153	BDE-183	BDE-209
"BDE-28"	1							
"BDE-47"	0.560	1						
	0.037							
"BDE-100"	0.493	0.645	1					
	0.072	0.012						
"BDE-99"	0.474	0.657	0.666	1				
	0.086	0.010	0.009					
"BDE-154"	0.566	0.947	0.703	0.786	1			
	0.034	2.754E-7	0.005	8.454E-4				
"BDE-153"	0.554	0.933	0.682	0.848	0.985	1		
	0.039	1.035E-6	0.007	1.262E-4	1.070E-10			
"BDE-183"	0.525	0.664	0.715	0.967	0.774	0.818	1	
	0.053	0.009	0.004	1.605E-8	0.001	3.398E-4		
"BDE-209"	0.500	0.667	0.670	0.993	0.787	0.843	0.985	1
	0.068	0.009	0.008	9.880E-13	8.217E-4	1.489E-4	1.377E-10	

The predominance of tri-BDE, a lower brominated diphenyl ether, in the water samples might be a result of the transformation of higher brominated diphenyl ether such as BDE-209 [37]. In addition, long-range transport via the atmosphere and hydrodynamic forces might be responsible for the contribution of tri-BDE in the water samples [38]. This observation is consistent with the reports of Ma et al [39] and Liu et al [40].

Despite the proscription of the penta-BDE congener, its relatively high abundance suggests the wide use and disposal of the commercial product in the surrounding areas of the studied river. This observation agrees with a previous study that reported the use and recycling of penta-BDE legacy products despite its prohibition in factories [41].

Comparatively, the compositional profile of the PBDE congeners showed that the percentage abundance of higher BDEs in the sediment samples was higher than that in the water samples. The high concentrations in sediments might be due to the lower reduction rate of higher PBDEs [37]. In addition, higher PBDEs are characterized by higher octanol-water partition coefficient

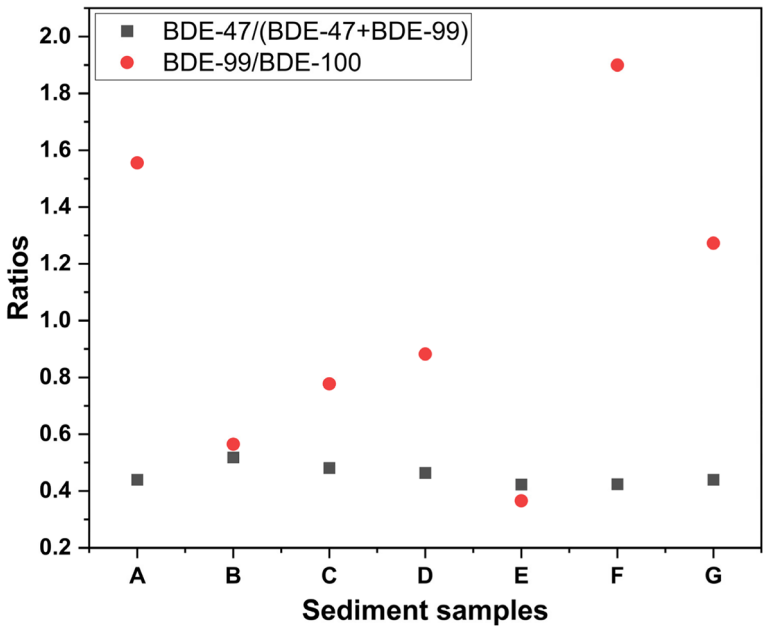

Fig. 4 Ratios of PBDE congeners in sediment samples

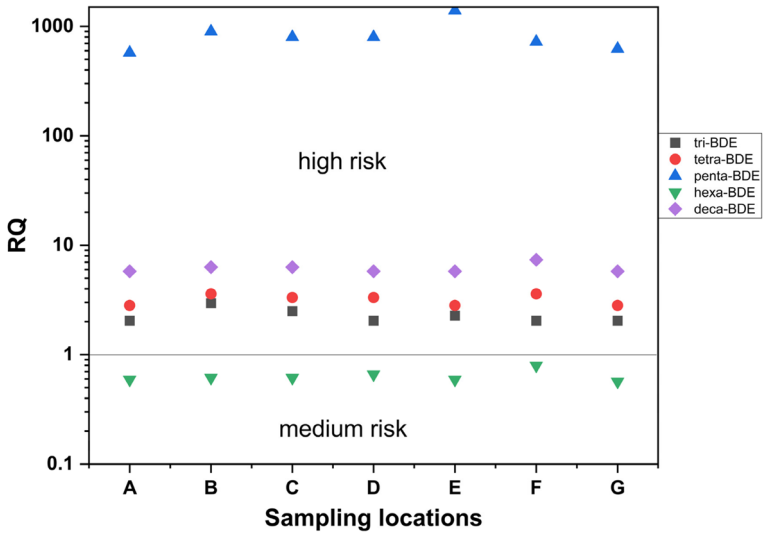

Fig. 5 Risk quotient of PBDE congeners based on FSeQG developed by Environment Canada

(K_{ow}) values and lower water solubility. This explains why they are easily adsorbed to sediments owing to their high affinity to sedimentary organic matter [23, 42]. The contribution of deca-BDE to the total PBDEs in the present study was relatively lower than the reports of previous studies [43–45]. This indicated that commercial deca-BDE mixtures might not be widely used in the study area, relative to other PBDE congeners.

A significant correlation ($p < 0.05$) existed among most of the PBDE congeners. However, penta-, hepta-, and deca-BDE did not show any positive correlation with tri-BDE. This is an indication that the sources of BDE-209, BDE-99, BDE-100, and BDE-183 are distinct from the sources of BDE-28. The relatively lower concentrations of BDE-28 compared to other congeners, particularly in the sediment samples, might as well suggest atmospheric transport as a result of its low vapour pressure [46, 47]. Some studies reported the debromination of BDE-209 into PBDEs with four to nine bromine atoms [48, 49]. The correlation result indicated that commercial deca-BDE mixtures represent the major sources of PBDEs in the studied river. The variation in the mean ratios of PBDE congeners and penta-BDE might be indicative of the debromination of deca-BDE (BDE-209) [45]. The mean concentrations of tri-BDE, tetra-BDE, penta-BDE, and hexa-BDE were all above the acceptable limits, indicating that these congeners might pose serious health risks to the public because of their toxicity. However, hepta-BDE was not detected in all the studied water samples.

Based on the results of the ecological risk assessment, the RQs of hexa-BDE were less than 1, indicating medium risk while the RQs of tri-, tetra-, penta-, and deca-BDE were greater than 1, indicating high risk. The high ecological risk posed by penta-BDE is due to its toxicity. Based on this value, penta- and deca-BDEs in the studied river require effective remediation. It was also observed that the calculated RQs in the present study were higher than those reported in other studies [50].

Conclusion

The present study was focused on the occurrence, source identification, and risk assessment of polybrominated diphenyl ethers in water and sediments of Osun River, Nigeria. Compositional profiles identified six classes of PBDE congeners based on the number of bromine atoms and they followed the order: penta-BDE > hexa-BDE > tetra-BDE > deca-BDE > hepta-BDE > tri-BDE in the sediment samples while tri-BDE was the most frequently detected congener in the water samples. One-way analysis of variance showed that the PBDE concentrations of the sediments were significantly higher than the water samples except for tri-BDE. These PBDE congeners end up in the environment in various forms, producing several toxic by-products upon photodegradation and microbial degradation. Despite the growing urbanization and increased industrial activity of the study area, this study is the first known attempt at quantifying the levels of PBDEs in the surrounding water body. Further studies are therefore required to estimate the bioaccumulation and biomagnification of these contaminants in the food web. Furthermore, monitoring studies should be conducted to continuously assess the pollution status of the river. It is recommended that efficient and cost-effective remediation strategies should be developed and employed in the abatement of these persistent organic pollutants from the environment.

Supplementary Information

The online version contains supplementary material available at https://doi.org/10.1186/s44329-024-00006-2.

Supplementary Material 1

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Authors' contributions
AOA and AST conceived and designed the experiments. AOA and OTO conducted the experiments. FMA provided the methodology and supervised the experiments. OTO wrote the main manuscript draft. All the authors proof read and reviewed the manuscript.

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Availability of data and materials
The authors declare that the data supporting the findings of this study are available within the paper and its Supplementary Information files. Should any raw data files be needed in another format they are available from the corresponding author upon reasonable request.

Declarations

Ethics approval and consent to participate
Not applicable.

Consent for publication
Not applicable.

Competing interests
The authors declare no competing interests.

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