

1 Original Research Article
2 **Levels, Spatial and Seasonal Distribution of**
3 **Polybrominated Diphenyl Ethers (PBDEs) in Water,**
4 **Sediments and *Sarotherodon melanotheron* from Ologe**
5 **Lagoon**
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8

9 **ABSTRACT**

10 Polybrominated diphenyl ethers (PBDEs) are chemical additives of increasing concern in
11 water bodies associated with industrial and urban areas. In this study, they were quantified in
12 sediments, surface water and Black Chin Tilapia, *Sarotherodon melanotheron* from three
13 sampling zones of Ologe Lagoon, Nigeria. The lagoon receives effluents and runoffs from the
14 neighbouring industrial town of Agbara. Quantification of PBDE congeners in the samples
15 was done using a gas chromatograph coupled electron capture detector (GC-ECD). Sediment
16 Σ PBDEs levels of 0.05 ± 0.006 to 0.09 ± 0.006 ng/g were below Environment Canada's (EC's)
17 safe limit (6124 ng/g) while levels in water ranged from 0.01 ± 0.003 to 0.02 ± 0.005 mg/L and
18 were above EC's safe limit (0.0002 mg/L). Σ PBDEs levels in the Tilapia ranged from
19 0.06 ± 0.01 to 0.12 ± 0.01 ng/g and were below EC's safe limit (631 ng/g). There was no
20 significant ($P > 0.05$) seasonal variation of Σ PBDEs concentrations in the assessed fish. The
21 low levels of PBDEs in *Sarotherodon melanotheron* suggest that the fish are safe for
22 consumption. However, there is need for continuous efforts to improve industrial effluents
23 treatment and manage urban surface run-offs into water bodies in order to avoid potentially
24 catastrophic levels of PBDEs in urban water bodies.
25

26 *Keywords: Aquatic toxicology; Polybrominated diphenyl ethers; Urban runoffs; Industrial*
27 *effluents; Waste management.*
28

29 **1. INTRODUCTION**

30 Polybrominated diphenyl ethers (PBDEs) are a group of chemicals that have been used as
31 additive flame retardants in products such as building materials, plastics, and electronic
32 equipment for decades [1,2]. They are hydrophobic and lipophilic [3], and could enter aquatic
33 environments through wet and dry deposition [4,5], surface runoff, and municipal and
34 industrial wastes [5]. Despite the importance of PBDEs in consumer products, the
35 characteristics of these chemicals make them persistent in aquatic environments [6]. They
36 tend to stick to sediments which usually act as a sink for hydrophobic contaminants [7]. Their

37 presence has been detected in water [8-10], and aquatic organisms such as molluscs [11] and
38 fish [12,13] which are able to bioaccumulate them, and most importantly, traces of PBDEs
39 have been found in humans [14-16]. Their detection in humans has led to the study of their
40 biological effects [17-21]. PBDEs are endocrine disrupting chemicals that can interfere with
41 the production, secretion, transport, binding, and elimination of hormones that are responsible
42 for development, behaviour, fertility, and the maintenance of homeostasis in the body [22-
43 24]. There have also been reports that PBDEs could be carcinogenic [25,3]. These effects
44 make the investigation of the occurrence of PBDEs in aquatic environments essential.

45 Ologe Lagoon has been receiving a variety of wastes from its surroundings and more notably
46 from Agbara Industrial Estate. The ecological and economic services of the lagoon include
47 mining, transportation, waste disposal, and fishery [26]. It is vital to note that effluents from
48 Agbara Industrial Estate, which is close to the lagoon, are discharged into it after treatment in
49 a central effluent treatment plant. There are over 20 factories (food and beverages,
50 pharmaceutical, breweries, metal finishing, chemical, pulp and paper industries) in the estate
51 [27]. Currently, there is no literature on the levels of PBDEs in the lagoon. It is therefore vital
52 to investigate the levels of toxicants such as PBDEs that could end up in abiotic and biotic
53 matrices of the lagoon. The aim of this study was to assess the spatial and seasonal
54 distribution of PBDEs in water and sediments from Ologe Lagoon using *Sarotherodon*
55 *melanotheron* bioindicator.

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58 2. MATERIALS AND METHODS

59 2.1 Study Area

60 The study area, Ologe lagoon, is in the south-western part of Nigeria and an extension of the
61 Badagry creek (Figure 1). The main body of the lagoon borders Badagry in Lagos State as
62 well as Agbara Township in Ogun State, Nigeria (Figure 1). Agbara is an industrial town
63 with industrial estates, and adjoining residential and business areas.

78 used for this study, is commonly exploited as a food resource in its native range and has been
79 utilized as an aquaculture and non-native fishery species [28].

80 **2.3 Extraction and Gas Chromatograph Analysis**

81 The methods used were adapted from [29]. Specifically, 1 L of water was extracted thrice
82 with 300 **ml** mixture of hexane and acetone (3:1v/v). The extract was combined and dried
83 with 20 **g** of anhydrous Na₂SO₄. After 24 hours, a rotary evaporator was used to concentrate
84 the extract to 2 **ml**.

85 Extraction of PBDEs from sediments and fish tissues was done by grinding 5 **g** of each
86 sample and mixing with 5 **g** of anhydrous Na₂SO₄ for homogenization. The homogenate was
87 then extracted with **200 mL** n-Hexane (solvent). The solution was shaken for 1 hour with an
88 electronic shaker and the solution was stored overnight for separation and evaporation of the
89 solvent. The extract was cleaned up using solvent-rinsed chromatographic columns (15 mm -
90 250 mm), packed with a plug of glass wool followed by 3 g deactivated silica gel and topped
91 up with Na₂SO₄. The column was pre-rinsed with **15 mL** hexane after which 2 mL of the
92 extract was added to the column and eluted with 60 mL hexane. The extract was then
93 concentrated to approximately 2 mL using a rotary evaporator and kept in a sample vial for
94 gas chromatographic analysis.

95 Quantification of PBDE congeners was done with a gas chromatograph coupled electron
96 capture detector (GC-ECD) equipped with 30 m×0.25 mm×0.25 µm VF-5ms fused capillary
97 column. The carrier gas was helium. The operating guidelines were as follows: column head
98 pressure was 2 psi for 1.5 min with a constant flow rate of 150 mL min⁻¹ at 4 min, the front
99 injector line was programmed at 250 **°C**. Injection volumes were 2.0 µL in the splitless mode.

100 The column temperature was set at 50 **°C** for 3 minutes initially and increased to 320 **°C** at a
101 rate of 20 °C per minute and then maintained at 320 **°C** for 20 minutes. Quantification of

PBDE by the electron capture detector was based on sample peak areas or peak heights relative to standard peak areas or peak heights.

2.4 Biota-Sediment Accumulation Factor (BSAF)

The biota-sediment accumulation factor (BSAF) was calculated as a factor of the levels of PBDEs in sediment and fish samples [30].

$$\text{BSAF} = \frac{\text{mean concentration of PBDEs in fish}}{\text{mean concentration of PBDEs in sediment}}$$

2.5 Bioaccumulation Factor (BAF)

The bioaccumulation Factor (BAF) was calculated as a ratio of PBDEs in the fish and surface water [31].

$$\text{BAF} = \frac{\text{mean concentration of PBDEs in fish}}{\text{mean concentration of PBDEs in water}}$$

2.6 Statistical Analysis

All results were expressed as Mean $\pm$ Standard Error. Two-way analysis of variance (ANOVA) with Duncan's Multiple Range (DMR) test was used to determine the significance of spatial and seasonal differences in the levels of PBDEs in water, sediments, and fish using SPSS version 20 (IBM).

3. RESULTS AND DISCUSSION

3.1 Congeneric Patterns

PBDEs comprise 209 related molecules, known as congeners [32]. The lower brominated PBDEs are known to exhibit higher toxicity than higher brominated PBDEs [33,34]. Only two congeners (BDE 7 and 28) were detected in this study (Figures 2 to 7). Tri-BDE (BDE 28) was the predominant congener. Higher brominated PBDEs were not detected in the present study. This could be as a result of photodecomposition or photolytic debromination of higher brominated PBDEs into smaller molecules BDEs [35]. [35] assessed the photodegradation of BDE 47, BDE 99, BDE 100, BDE 153, and BDE 183. [35] observed that

128 higher brominated BDE congeners degraded at a faster rate than the lower brominated
129 congeners. It was concluded that the main decomposition mechanism induced by photolysis
130 was reductive debromination to form lower brominated BDE congeners. [36] investigated the
131 photodegradation of BDE 209 in plastics and obtained similar results.

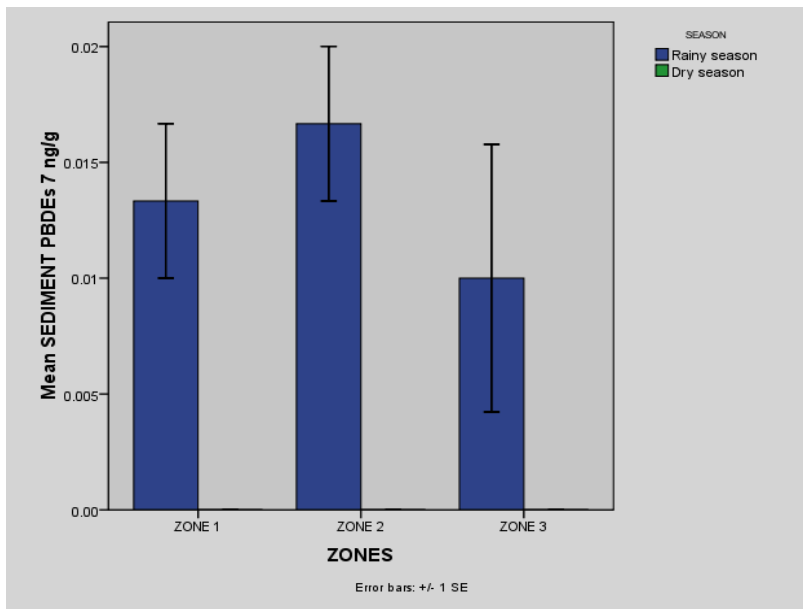
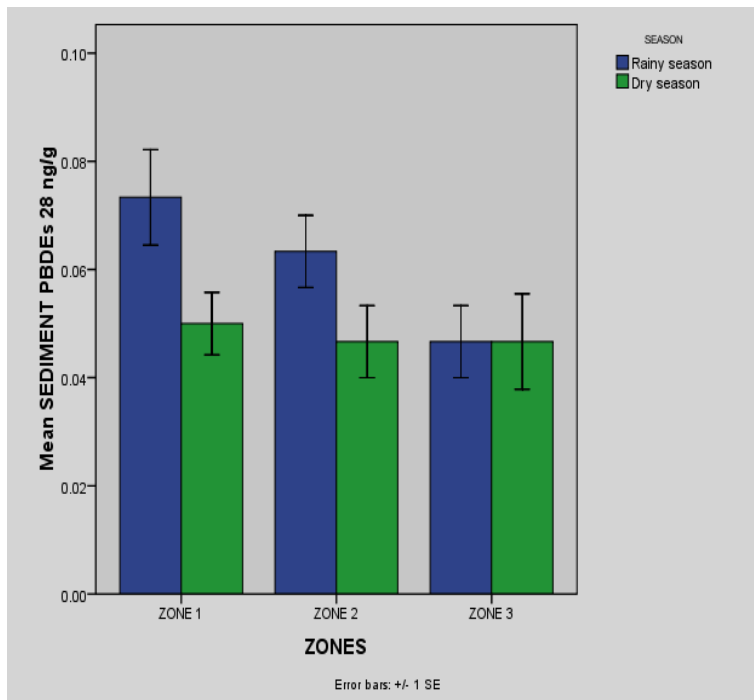
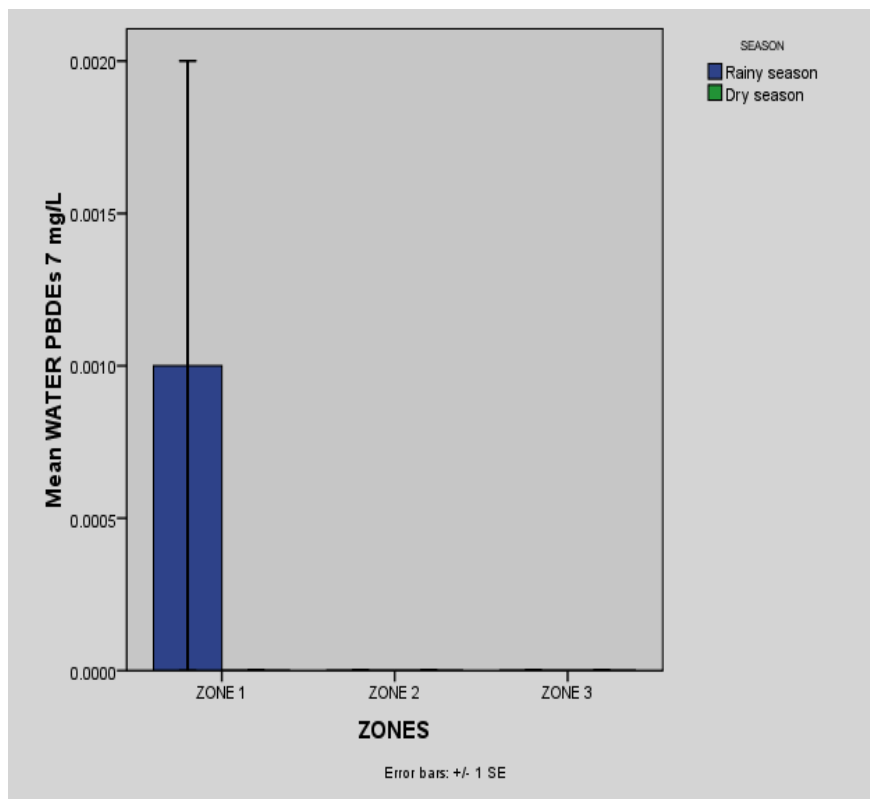


Figure 2: Concentrations of BDE 7 in Sediments samples of Ologe lagoon



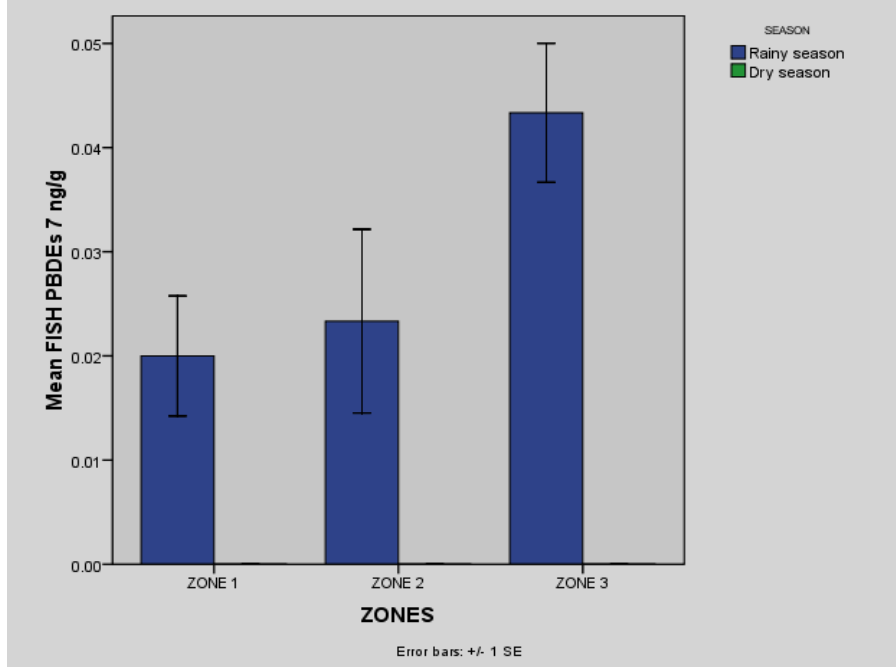
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Figure 3: Concentrations of BDE 28 in Sediments samples of Ologe lagoon



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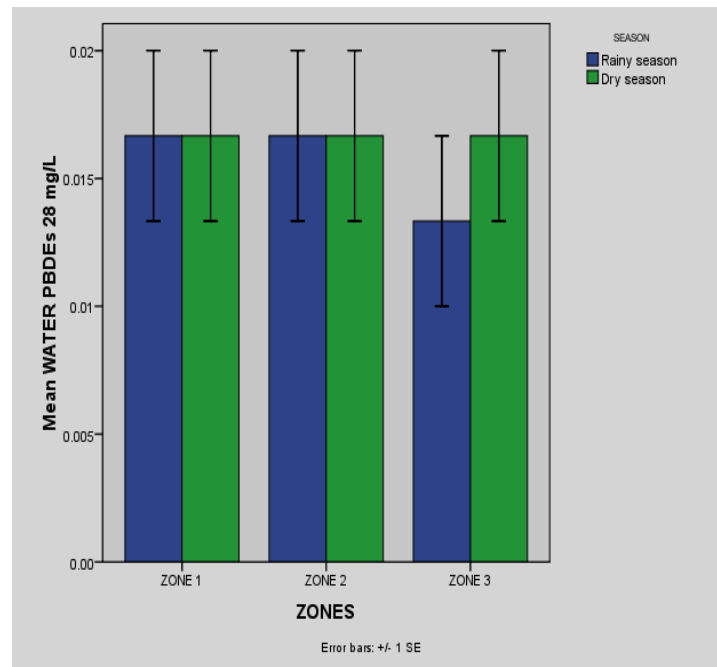
Figure 4: Concentrations of BDE 7 in the surface water samples of Ologe lagoon



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Figure 5: Concentrations of BDE 7 in Fish samples of Ologe lagoon

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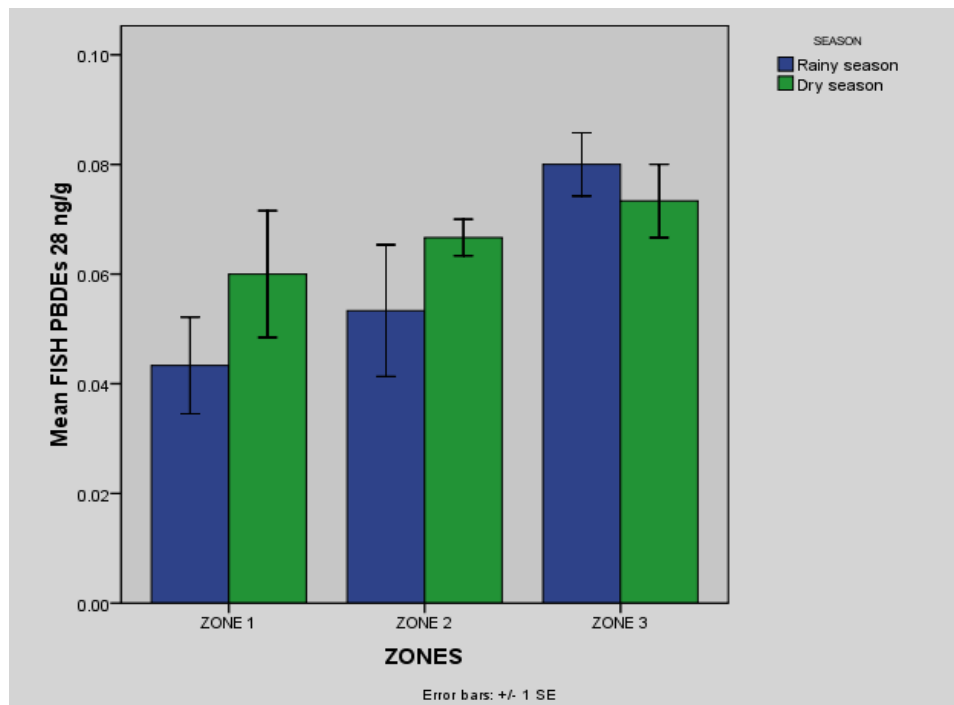
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Figure 6: Concentrations of BDE 28 in the surface water samples of Ologe lagoon



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Figure 7: Concentrations of BDE 28 in Fish samples of Ologe lagoon

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158 3.2 Seasonal and Spatial Distribution of PBDEs in Sediments

159 There was a significant ($P < 0.05$, $F = 4.00$) spatial difference in PBDE levels in sediments.
160 This could be attributed to the varying distances of the zones to the source of effluents.
161 Σ PBDEs levels of 0.05 ± 0.006 to 0.09 ± 0.006 ng/g (Figure 8) in sediments recorded in this
162 study were within the range reported by [37] in China and [38] in Japan and were below
163 Environment Canada's (EC's) safe limit (6124 ng/g). Σ PBDEs levels in sediments were
164 significantly ($P < 0.05$, $F = 25.190$) higher in the rainy season than the levels recorded in the
165 dry season. The variation might have been caused by the seasonal differential influx of
166 industrial wastes into the lagoon from nearby industries (Agbara industrial estate precisely).
167 In the dry season, there was less surface runoff which might cause contaminants to
168 accumulate in the surface soil while in the rainy season, the contaminants previously
169 accumulated in the surface soil are being flushed into the lagoon through surface runoff due
170 to the heavy rains [39-41].

171

172 3.3 The Occurrence of PBDEs in Water

173 The Σ PBDEs concentrations in surface water samples from the lagoon ranged from
174 0.01 ± 0.003 to 0.02 ± 0.005 mg/L (Figure 9). There was no significant spatial ($P > 0.05$, $F =$
175 0.375) and seasonal ($P > 0.05$, $F = 0$) variation in the Σ PBDEs concentrations. [42] assessed
176 the effects of seasonal change on PBDEs concentrations in water but did not report
177 significant seasonal variations as well. The Σ PBDEs levels in water in this study and in Ile
178 Ife [10], another town in Nigeria with much less industries were found to be above EC's safe
179 limit (0.0002 mg/L). This raises concern on the diffuse nature of this pollutant and the
180 potential for environmental and public health risks.

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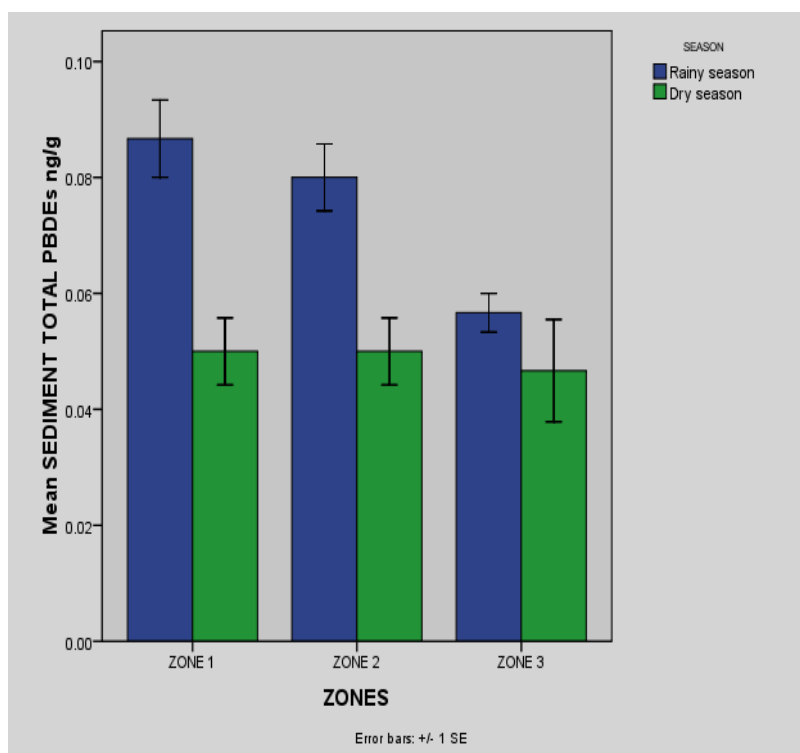


Figure 8: Concentrations of Σ PBDEs in Sediments samples of Ologe lagoon

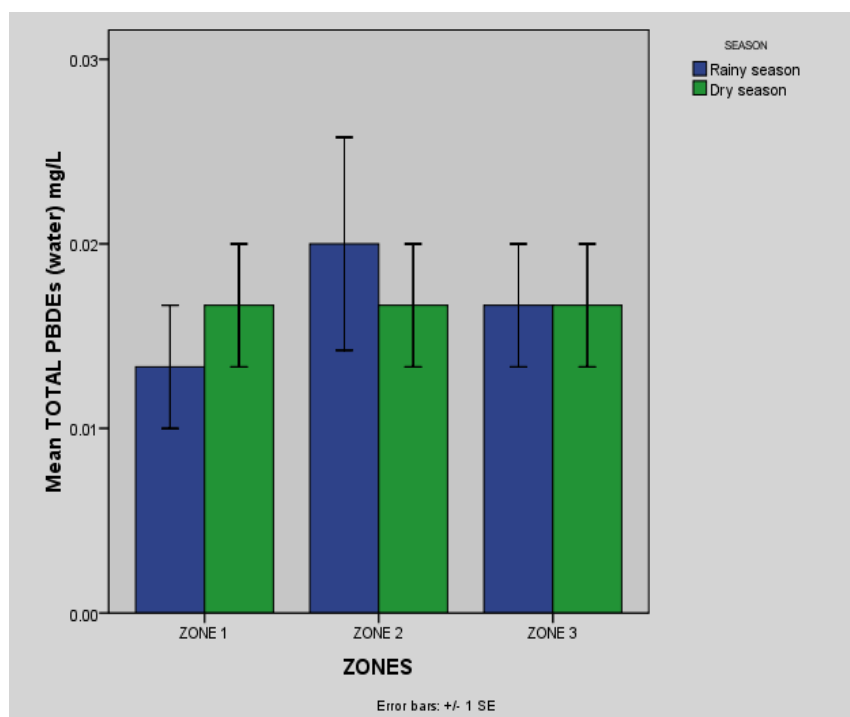


Figure 9: Concentrations of Σ PBDEs in the surface water samples of Ologe lagoon

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193 3.4 Occurrence and Bioaccumulation of PBDEs in *Sarotherodon melanotheron*

194 Σ PBDEs concentrations ranged from 0.06 ± 0.01 to 0.12 ± 0.01 ng/g in the assessed tilapia fish
195 (Figure 10) . There was a significant ($P < 0.05$, $F=4.675$) difference in PBDE concentrations
196 across the sampling zones. The spatial difference could be associated with the difference in
197 the proximity of the zones to the source(s) of effluents. The Σ PBDEs values in *S.*
198 *melanotheron* were below EC's safe limit of 631 ng/g. There was no statistical seasonal
199 variation ($P > 0.05$, $F = 4.349$) in Σ PBDEs levels in fish.

200 The levels of Σ PBDEs in the assessed tilapia were low when compared to data from other
201 countries (Table 1). The low levels of PBDEs in fish found in the present study suggest that
202 the levels of PBDEs bioavailable for aquatic organisms in Ologe lagoon were low. Nigeria is
203 much less industrialized than most of the countries listed in Table 1 while for the case of
204 Ghana the differences might lie on the specific sources of effluents and runoffs entering the
205 water bodies examined.

206 In the rainy season, Tilapia from zone 3 had the highest BSAF and BAF values which
207 suggest that accumulation of PBDEs in the fish from zone 3 was highest (Table 2). In the dry
208 season, BSAF and BAF values were the same except BSAF of PBDEs in fish from zone 1
209 (Table 2). [43] noted that the mobility and reproductive behavior of an aquatic organism
210 could influence the extent of bioaccumulation of contaminants.

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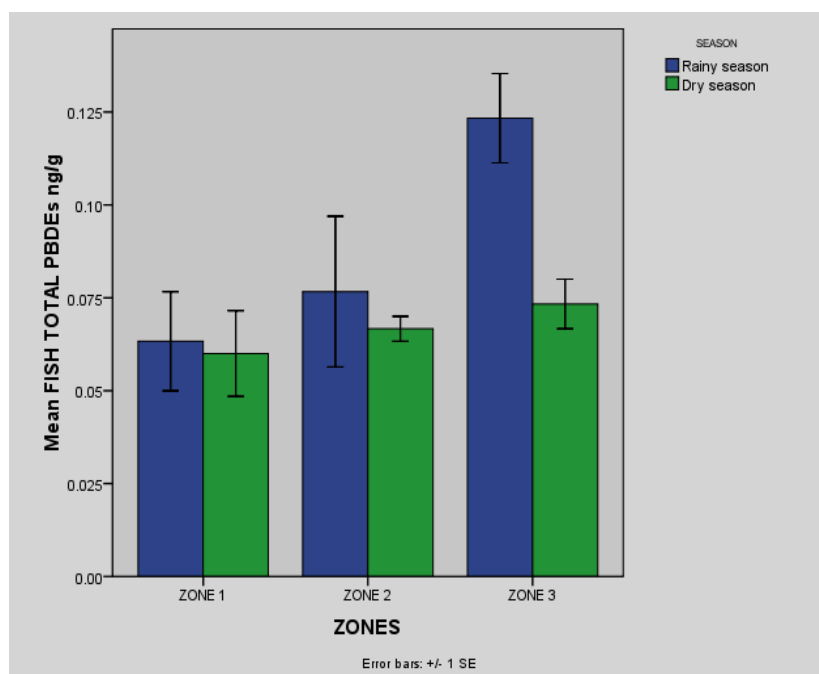


Figure 10: Concentrations of Σ PBDEs in Fish samples of Ologe lagoon

Table 1: PBDEs Levels in Fish from other countries

Country	Levels	References
U.S.A.	1140 ng/g ww	[44]
Sweden	36,900 ng/g lw	[45]
Norway	72–1120 ng/gww	[12]
Ghana	7.3 ng/g lw	[13]
China	18 to 1100 ng/g lw	[46]
Nigeria (Ologe lagoon)	0.06±0.01 to 0.12±0.01 ng/g	Present study

Table 2: Biota-Sediment Accumulation Factor (BSAF) and Bioaccumulation Factor (BAF)

	Sampling Zones					
	Rainy Season			Dry Season		
	1	2	3	1	2	3
BSAF	0.67	1.00	1.33	1.2	1.4	1.4
BAF	0.005	0.004	0.007	0.004	0.004	0.004

4. CONCLUSION

The levels of PBDEs in this study suggest that the release of the PBDEs into the lagoon occurs at a low rate considering the vast array of anthropogenic activities that is carried out in the area. This may be attributed to the fact that industries around the area are more concerned

with the production of pharmaceuticals and beverages rather than electronics and equipment. The present research findings indicate that *S. melanotheron* inhabiting the water body are safe for human consumption with regard to PBDEs level. On the contrary, the Σ PBDEs levels in the water were above EC's safe limit confirming the surface runoffs as possible route of entry into the water bodies. The need for continued environmental monitoring of PBDEs levels is advised in view of their potential to act as potent carcinogens.

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