

Assessment of Polybrominated Diphenyl Ethers (PBDEs) Level and Physico-Chemical Characteristics of Water and Sediment of Gulf of Guinea

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Abstract

The Lagos lagoon is the largest of the four lagoon systems of the Gulf of Guinea coast. This aquatic resource of multiple usages receives diverse organic and inorganic materials and sources. from domestic, industrial, emission, thermal The levels of Polybrominated diphenyl ethers (PBDEs) were assessed to ascertain the pollution status of surface water and sediment within this lagoon system. Four stations were sampled for PBDEs analysis, and ranged for surface water physicochemical parameters are (27.93 to 28.50 °C), (115.00 to 2152.70 µS/cm), (2.00 to 2.80 mg/L), (6.45 to 7.63), (25.02 to 84.60 NTU), (226.30 to 3999.00 mg/L), for temperature, electrical conductivity, dissolved oxygen, pH, turbidity, and total dissolved solids respectively. While the Total Organic Carbon (TOC) in sampled sediment ranged from 0.5 to 5.71 mg/L., the total concentrations of PBDEs in sediments ranged from 31.6 to 47.5 μ g/Kg. Whereas the BDEs concentration in water ranged from 3.7 to 10.5 pg/L, total PBDE concentration of individual congener for water ranged from approximately 0.8 to 12.5 pg/L. The BDE 28 predominates across the locations followed by BDE 99. Impact of PBDEs concentration in the marine environment was evident within the study area.

Received: March 23, 2022; Accepted: April 17, 2022

Keywords and phrases: PBDEs; contamination; sediment; Lagos lagoon; Gulf of Guinea.

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Introduction

Human health and environmental quality are undergoing continuous degradation by the increasing amount of wastes being produced [16]. Lagos lagoon, considered to be the largest of the four lagoon systems of the Gulf of Guinea coast, receives diverse organic and inorganic materials from domestic, industrial, emission, and thermal sources [2]. The disposal and management of wastes in Lagos poses serious environmental threat to this region's aquatic ecosystem, as the usual methods of waste disposal such as land filling, dumping site and incineration had led to contamination of underground, sediment, and surface water [1]. These intense anthropogenic activities such as direct domestic and industrial waste discharges, oil spillage, and sand mining among others have been reported in the lagoon system [18].

Polybrominateddiphenyl ethers (PBDEs) are one of the classes of recalcitrant and bio accumulative halogenated compounds that have emerged as a major environmental pollutant. PBDEs constitute a group of brominated flame retardants (BRFs) known as additive flame retardants, which have been used in a variety of consumer products since the 1960's [3].

Emerging pollutant are usually applied in care products, pesticides, domestic and industrial chemicals and these product are mostly found in plastics, used in a varieties of consumer products ([7], [10]). Despite the restriction placed on their production under the Stockholm Convention due to their toxicity and persistence ([19], [23]), they have been found in water bodies, sediment, soil and air samples from all over the globe [14].

PBDEs have been found to be readily accumulated by marine animals via ingestion from diet and gill for oxygenated exchange [27]. Being lipophilic and persistent organic compounds, PBDEs can bioaccumulate in lipid rich tissues and biomagnify along the food chain [13]. Consequently, food items like fish from high trophic levels or lipid-rich oils have been found to contain relatively high concentrations of PBDEs [9], thus presenting an important exposure pathway to humans. While there are empirical evidences suggesting global increase in the levels of PBDEs in human bodies, there is still paucity of information on the health implications of this group of compounds. This study was therefore carried out to assess the levels of PBDEs in water and sediment of Lagos Lagoon – an eastern arm of the Gulf of Guinea, and also make recommendations based on their distribution.

Materials and Methods

The Study Area

Lagos Lagoon, an eastern arm of the Gulf of Guinea is a brackish coastal lagoon which forms part of the continuous system of lagoons and creeks that are found along the coast of Nigeria from the border of the Republic of Benin to the Niger Delta. The major outlet of freshwater is at Lagos, Nigeria, where it forms an extensive harbor [17]. Being an expanse of shallow water with depth ranging from 0.5 m to 5 m, dredged parts of the Lagos harbor can attain a depth of up to 25 m. The tidal range is small and it is only about 0.6 m to 1 m. Figure 1 shows the bathymetric chart of Lagos lagoon. Economic

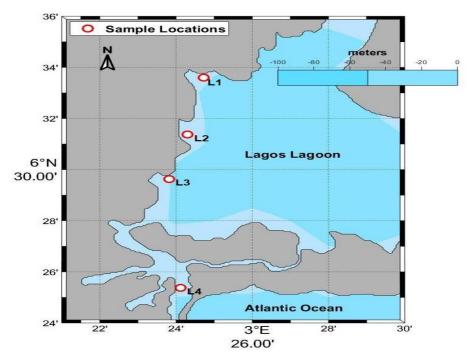


Figure 1. Map showing sampling locations in Lagos lagoon.

activities that surround Lagos Lagoon are shipping, fishing, aquaculture, sand mining, dredging activities and sawmilling. The communities that make up the Lagos lagoon waterfront include Makoko, University of Lagos, Ilaje, Oworonshoki, Ogudu, Bayekun, Agboyin, Moba, Ofin, Ikorodu, Ibeshe, Aja, Lekki peninsula, Banana Island and Ikoyi. Four sampling stations were established based on ecological factor, pollution level, and human activities within the area.

Sample Collection / Field Data Acquisition

Physico-chemical parameters including pH, water temperature, turbidity, salinity, total dissolved solids (TDS), conductivity, dissolved oxygen, organic matter and organic nitrogen, were measured and recorded in situ with the aid of Hanna Automatic water analyzer. Meteorological parameters including atmospheric temperature and pressure, heat index, wind speed, altitude, dew point temperature, density altitude were measured and recorded using weather tracker.

Glass bottles were used for water samples collection while an Ekman grab sampler was used for sediment sample. Thirty six samples (sixteen for water and sixteen for sediment) were collected at 2 to 4 different spots in a grid pattern, and composites (samples from more than one area that were combined in a single sample container) of each were taken to give full representative of the sample in the station. Sediment samples were wrapped in Aluminum foil (for PBDEs, Chloride and Total organic carbon (TOC) analysis), kept in ice chests and taken to the laboratory. Global Positioning System (GPS) was used to determine the geographic and rectangular coordinate of each station and a map of the study area was generated. The sampling activities cover a complete transverse of the Lagos Lagoon as shown in Figure 1.

Laboratory Analysis

Total Organic Carbon (TOC) Determination

0.5g dried sediment sample was weighed into a beaker/conical flask. 10ml prepared Potassium Dichromate ($K_2Cr_2O_7$) was added and the mixture was swirled gently. 20ml concentrated Sulphuric acid (H_2SO_4) was rapidly directed into the suspension and the mixture was immediately swirled gently until the reagents were mixed. The flask was allowed to stand for 30mins, then 100ml distilled water and 10ml H_3PO_4 was added. 3-4 drops ferroin indicator was added giving blue-green color formation. It was titrated against ferrous ammonium sulphate FAS (wine red coloration gives the end point). TOC is calculated using the formula below;

$$\% OM = [(V_b - V_d) \times 6.971/V_b] / Weight of sample (g)$$
(1)

where; V_b = volume (ml) (FAS) blank, V_a = volume (ml) sample

$$\% OC = [6.719/1.724x] [(V_b - V_a)/V_b]$$
(2)

where 'x' is the Weight of sample (g)

For % Organic Nitrogen (%ON);

$$\% ON = \% OC/20$$
 (3)

Chloride Determination

10ml sample is measured, and distilled water is added to make it up to 50ml. $K_2Cr_2O_7$ indicator is added to solution, color changes to yellow. Titrate with AgNO₃ titrant to a brick red.

Calculation

 $Cl(mg/L) = [((V_s - V_b) \times N \times 35.45 * 1000) \div Volume of sample (ml)] \quad (4)$

where;

 V_s = Titre value of sample (ml)

 V_b = Titre value (ml) for blank

 $N = Normality of AgNO_3 (0.0257N)$

Extraction and Clean-up of Samples

5g of air dried sediment sample was homogenized with 5g of anhydrous sodium sulphate in a mortar. The mixture was then transferred into an amber bottle and 25ml n-hexane was added to the mixture to extract the analyte matrix from the sample. It was shaken for 30minutes on the mechanical shaker at about 1000 rpm/min. The extract was filtered and collected into a well labeled beaker. The extract was cleaned-up using a multi-layer glass column plugged with glass wool followed by silica gel and then activated anhydrous sodium sulphate. N-hexane was used to condition the column and PBDEs were eluted with n-hexane. The eluate was concentrated to near dryness and the extract was re-solubilized in 1 ml iso-octane.

Water Samples

Liquid-liquid Extraction Technique (LLE)

25ml of water sample was measured into a Separating Funnel and 25ml of Dichloromethane (DCM) was added. The mixture was shaken rigorously for 30mins and the organic layer was allowed to separate clearly from the aqueous phase for a minimum of 5 minutes. The organic layer was collected inside a well labeled beaker. The extraction was repeated for each sample and the final extract was concentrated.

Sample Cleanup / Purification and Separation

Packing column was set-up by inserting glass-wool inside glass syringe using glassrod. Five grams (5g) pre-heated silica gel was put into the column, and then 2g preheated sodium sulphate anhydrous salt was again added. The column was activated by running about 30ml n-Hexane through the packed column until it was ensured that it runs well. The sample was run through the activated column. It was eluted by running 50ml mixture of n-Hexane and Petroleum Ether (1:1) through the column. The extract was collected and concentrated to about 2ml. 1ml iso-octane was added to the extract and then ready for injection into the GC.

Gas Chromatography Analysis

The column was calibrated with standard mixtures for the recovery of all compounds of interest.

Analysis of PBDEs

- Agilent GC-7890A (ECD) Column Type: (DB-17) (30m x 0.25mm x 0.25 micron)
- Detector type: FID Carrier Gas: Helium, Make-up: Nitrogen
- Injection Temperature: 250 °C Inlet Mode: Splitless Injection Volume: 1uL
- Flow rate: 2ml/min

$$Formula = \frac{GCresults from chromatogram x dilution factor}{Volume of sample taken for extraction}$$
(5)

where

$$Dilution \ factor \ = \ \frac{Final \ volume \ of \ extract}{Weight \ of \ extract \ or \ cleaned-up \ extract}$$

Quality Assurance and Quality Control

The quality assurance and quality control (QA/QC) samples included solvent blank, procedural blank and matrix spiked samples, all of which were analyzed together with the collected sediment samples. Solvents injected before and after the injection of standards showed negligible contamination or carryover, the blanks did not contain quantifiable amounts of any target compounds. The percent recoveries of was $78.6 \pm 7.2\%$ in matrix

spiked samples. A procedural blank and spiked sample consisting of all reagents was run to check for interference and cross-contamination. Instrumental QC was performed by injecting solvent blanks and standard solutions. The relative percent difference were less than 20% for all targets analyses. The limit of detection range from 5 to 9.7 ng/g. Relative errors and standard deviations obtained from the analyses of four spiked samples were used to evaluate the accuracy and precision of the analytical method.

Result and Discussion

Altitude and Wind Speed

The Altitude of the study area ranges from -15 to -8 m, with highest altitude below the sea around Ijora and lowest around Agbonyi (Table 1). This indicates that sample was taken from areas having high elevation. Wind speed ranges between 0.8 m/s and 2.27 m/s and becomes maximum around Agbonyi (due to closeness to the sea), then reduces around Apapa with minimum value around the entrance of Makoko station.

Atmospheric and Water Temperature

Temperature has a pronounced effect on the rate of chemical and biological processes in water and largely affects development and growth of some biological species like fish [4]. Water temperature also has the ability to cause mortality as well influence the solubility of Dissolved Oxygen needed by aquatic lives [20].

The Atmospheric temperature ranges from 28.03 °C to 30.10 °C with lowest value at Apapa and highest value at Makoko. The water temperature ranges between 27.93 °C and 28.5°C, with minimum value at Agbonyi and maximum at Ijora (Table 1). The time of sampling may influence the readings at each station. In the course of sampling, atmospheric temperature gives a higher value than the water temperature. Higher value of atmospheric temperature than water temperature could be due to higher heat capacity of the air. The optimal water temperature of the coastal waters ranged from 28 °C – 30 °C, within which maximal growth rate, efficient food conversion, best condition of fish, resistance to disease and tolerance of toxins (metabolites and pollutants) are enhanced [21]. The water temperature of the study area is within 20 – 32°C permissible limit given by WHO [28].

PARAMETERS	STATIONS			
	APAPA	IJORA	МАКОКО	AGBONYI
ALTITUDE (M)	-13.50	-15.00	-14.00	-8.00
WIND SPEED (M/S)	2.18	1.63	0.80	2.27
ATM. TEMP. (°C)	28.03	28.80	30.10	28.17
HUMIDITY (%)	89.03	86.10	82.95	88.70
HEAT INDEX	35.15	36.27	56.31	34.67
DEWPOINT (°C)	26.40	26.33	26.90	26.27
BAROMETER	29.96	29.97	29.96	29.94
WETBULB	26.70	26.80	27.30	26.70
SALINITY (ppt)	5.76	7.80	2.45	1.92
РН	7.63	7.49	6.81	6.45
TDS (mg/L)	3549.30	3999.00	1020.50	226.30
WATER TEMP. (°C)	28.33	28.50	28.45	27.93
CONDUCTIVITY (µS/cm)	2000.00	2152.70	509.50	115.00
OM%	3.79	0.87	4.31	8.45
OC%	2.19	0.51	2.50	4.90
DO (mg/L)	2.80	2.00	2.00	2.80
CHLORIDE (PPM)	3188.30	4316.00	1360.40	1063.50
TURBIDITY	25.02	73.97	84.60	49.40

 Table 1. Some measured physico-chemical and Meteorological Parameters.

Humidity and Heat Index

The humidity values range between 82.95 and 89.03% and heat index between 34.67 and 56.31. The humidity decreases gradually from the starting point at Apapato Makoko, before falling back, then finally increases particularly 100% during the end of sampling showing that the atmosphere was very humid then (Table 1). Though this value range is high as heat index from 32 to 41 could lead to heat cramps and heat exhaustion, heat stroke is also possible [29], but, because of the tropical region Nigeria is situated and extreme effects of climate change could be a possible reason behind this high values.

Dew Point and Wet-bulb Temperature

Dew point is the temperature to which air must be cooled to become saturated with vapour. It values ranges from 26.27°C to 26.90°C in the study area and it shows similar variation like the heat index having its maximum around Makoko although minimum at Agbonyi (Table 1). This high range of values is due to reason mentioned above but generally, Dew point greater than 26.00°C is severely high and could be deadly [29].

Wet bulb is the temperature an air parcel would have if it were cooled to saturation by evaporation of water into it, with the latent heat being supplied to the parcel. The values range from 26.70 to 27.30 °C with maximum value at Apapa and Agbonyi while the minimum value at Apapa. The wet bulb of the study area below the 35.00 °C, the threshold at which human body will no longer be able to cool it and begins to overheat.

Turbidity

The Turbidity range from 25.02 to 84.60 NTU with the most turbid water recorded at Makoko and least at Apapa (Table 1). The entire water samples in the study area are higher than the maximum permissible limit of 8 NTU for surface water as set by WHO [28]. The general high value in this region could be due to the precipitation, whereby particles were washed down to stream and rivers. Waste discharge, urban run-off, bottom feeders that stir up sediments, wave, current actions especially in less deep areas [25] and upwelling may also be a factor [4].

pН

pH is closely linked with biological productivity [22] and also have numerous effects on fishes; for example, < 4.0 for acid death point, 4.0 - 5.0 for no production, 6.5 - 9.0for desirable range for fish, 9.0 - 11.0 for slow growth and > 11.0 for alkaline death point [9]. The largest varieties of fresh water aquatic organisms prefer a pH range from 6.5 to 8.0 [5, 28]. pH ranges from 6.45 at Agbonyi to 7.63 at Apapa (Table 1). Water samples from Apapa and Ijora show that these stations are alkaline (pH > 7) while Agbonyi and Makoko are slightly acidic (pH < 7). Hence, aquatic organisms including fish will survive in all the sample stations.

Dissolved Oxygen, Organic Carbon and Matter

Dissolved oxygen ranges from 2.0mg/L (Ijora and Makoko) to 2.8 mg/L (Apapa and Agbonyi) (Table 1). WHO limit for DO is > 4.0 indicating that the level of dissolved

oxygen in this region is low. Aquatic organisms are put to stress as dissolved oxygen drop below 4.0 mg/L, and it is very dangerous for aquatic organisms below 2.0 mg/L. The dissolved oxygen (DO) concentration average in this study is 2.4 mg/L, which is almost half of that found by Hossain et al. [12] in mangrove ecosystem of Bangladesh, and indicates that the waters are polluted by organic enrichment. Moreover, addition of oxygen consuming waste such as sewage, nutrients, rising of water temperature and addition of chemicals are strictly responsible for low dissolved oxygen in this study area.

The Organic matter and Carbon range from 0.87 to 8.45 % and from 0.51 to 4.90 % respectively (Table 1). The diverse nature of the relationship between Organic matter and DO may be as a result of local factors, but location like Agbonyi indicates that high organic matter can lowers the amount of DO in a water body as bacteria will make use of available oxygen to degrade the excess organic materials.

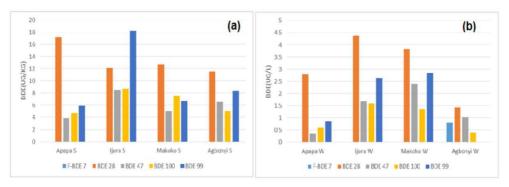
Chloride and Salinity, Conductivity and Total Dissolved Solids (TDS)

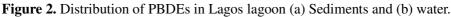
Chloride and Salinity range from 1063.50 to 3188.30mg/L and from 1.92 to 7.80ppt respectively. High values were recorded at stations closer to the ocean and decreases as we moved inland, so does Salinity. The values for chloride all exceed WHO limit which is 600 mg/L showing that this cation is high in the fresh water region.

The Total Dissolve solids (TDS) range from 226.30 to 3999.00 mg/L and conductivity from 115.00to 2152.70 μ S/cm. The conductivity and TDS showed strong positive correlation. This shows that conductivity depends on the amount of dissolved solids in water. The conductivity of water is affected by the suspended impurities and also depends up on the amount of ions in the water. The conductivity of fresh water should ideally be between 150 to 500 μ S/cm to support diverse life.

PBDEs

The study revealed five PBDE congeners which includes; F-BDE 7, BDE 28, BDE 47, BDE 99 and BDE 100. BDE 153, 154, 183 and 209 were not detected at all in both surface water and sediment samples. BDE 7 was also not detected in the sediment samples at all. For individual PBDEs concentration, the highest concentration detected throughout the whole locations was BDE 99 in Ijora sediment, followed by BDE 28 in Apapa sediment (Figure 2). BDE 28 has the highest concentration at each station except at Ijora where BDE 99 was the highest. After BDE 28, BDE 99 also showed some dominance through the sediment samples. BDE 47 and 100 have their highest values in the sediment and water samples of Ijora (Figure 2a and 2b). BDE 28 has the highest





concentration at each station throughout the water samples, and far higher than the levels of the next congener at Apapa and Ijora (Figure 2b). BDE 99 was detected in the entire water sample except in Agbonyi conversely BDE 7 was detected only in Agbonyi water sample. BDE 47 has its highest value at Makoko and lowest at Apapa, while BDE 100 has highest value at Ijora and lowest at Agbonyi. The BDE values in sediment are normally higher than the water, and they showed similar variation pattern except in Ijora where BDE 99 has the highest value in sediment sample followed by BDE 28. Ijora sediment has the highest total PBDE and others have nearly close values following the order, Ijora (33%) > Makoko (22.4%) > Apapa (22.2%) > Agbonyi (22.1%) (Figure 3). For water, Makoko has the highest total BDE (36%) followed by Ijora (35.5%), then Apapa (15.9%), with Agbonyi having the lowest (12.6%) (Figure 3).

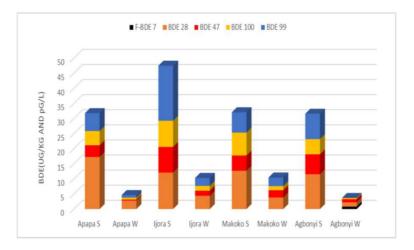


Figure 3. Distribution of Total PBDE in the study area.

Ijora is surrounded by a number of industries and among them are Nigerian Breweries Igunmu, Apex paper mill, Nigeria flour mills Apapa, Thermal power plant at Egbin, and some of the oil marketers have their oil depots very close to the creek. Furthermore, battery chargers, motor mechanics and Panel beaters are all situated there. Makoko comprises many houses built on and beside the creek and human feces are dumped into the lagoon, coupled with waste water, including plastics, and dirts, leachates from electronics components etc. Generally, the intensive domestic, industrial, commercial and marketing activities in Apapa, Makoko and Ijora may be responsible for these high PBDE levels.

For total congeners in sediment, BDE 28 (37.5%) > BDE 99 (27.5%) > BDE 100 (18.2%) > BDE 47 (16.8%) > BDE 7 (0%) (Figure 4), while the trend in water BDE 28 (42.9%) > BDE 99 (27.5%) > BDE 47 (18.8%) > BDE 100 (13.6%) > BDE 7 (2.8%) (Figure 4).

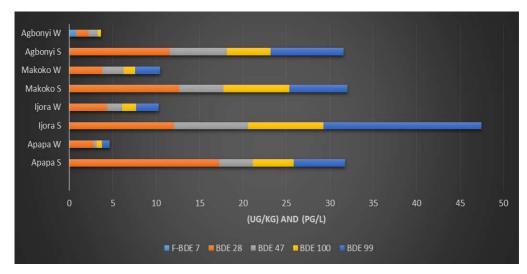
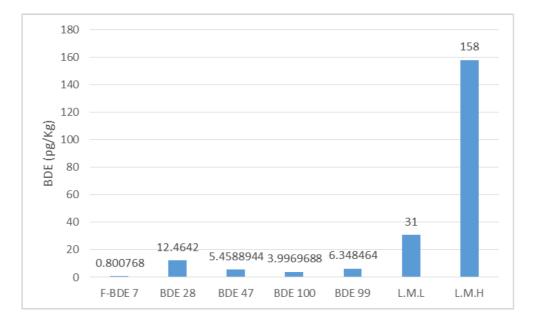


Figure 4. Distribution of Total PBDE at each location.

Luckey et al. [15] measured total (dissolved and particulate phases) PBDE (mono- to heptaBDE congeners), more than 60% of the total was composed of BDE47 (tetraBDE) and BDE99 (pentaBDE), with BDE100 (pentaBDE) approximately 5 to 8% of the total. Also, Stapleton and Baker [26] analyzed water samples from Lake Michigan in 1997, 1998 and 1999 and found that total PBDE concentrations (BDEs 47, 99, 100, 153, 154 and 183) ranged from 31 to 158 pg/L. The total PBDE concentration in Lagos Lagoon as determined in this work for water samples ranged from 0.8 to 12.55 pg/L, showing that

the result is much lower in comparison to the Lake Michigan as also shown in Figure 5. When compared with the work of Luckey et al. [15] BDE 47, 99 and 100 also composed



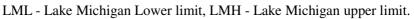
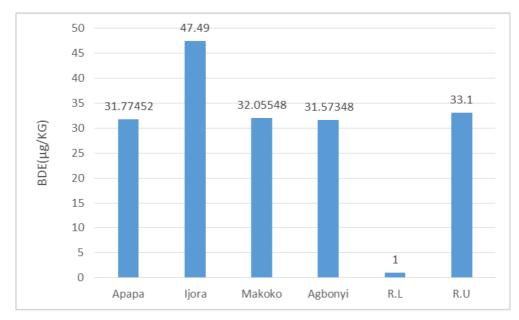


Figure 5. Comparison of PBDE concentration in Lagos lagoon with Lake Michigan, USA.

of about 60% but only BDE 28 dominates in this work. Figure 5 also shows that total individual PBDEs in Lagos lagoon waters were much lower than both the **lower limit** and **upper limit** of the Lake, though the total numbers of stations sampled were not stated which may be one of the reasons for the large difference.

Total PBDE concentration of individual congener for sediment sample of this study range from approximately 0 to 53.6 μ g/Kg. Dodder et al. [8] reported concentrations of total tetra, penta- and hexaBDE ranging from approximately 5 to 38 μ g/kg dw in sediment from the same lake Michigan in the U.S. located near suspected PBDE sources. Total BDE 28 in this study is much higher than both the lower and upper limit value of the Total PBDE concentration in the Lake as shown in Figure 6. Furthermore, total BDE 99 is higher than the upper limit, while the upper limit is higher than Total BDE 47 and 100, and the lower limit greater than BDE 7. This indicates that PBDE has accumulated in sediments of Lagos lagoon.



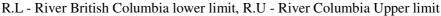


Figure 6. Comparison of PBDEs Sediment Concentration of Lagos Lagoon to British Columbia sediment.

Furthermore, the summation of all PBDEs at each station in the sediment of the study area ranges from 31.6 to 47.5 μ g/Kg. Rayne et al. [24] measured PBDE concentrations (sum of 8 di- to pentaBDE congeners) ranging from 2.7 to 91 μ g/kg in 11 surficial sediments collected in 2001 from several sites along the Columbia River system in southeastern British Columbia. Domestic wastewaters arising from septic field inputs were identified as potentially dominant sources of PBDEs in the region. Comparing both also at the same level yields Figure 5. The location most concentrated with PBDE in Lagos Lagoon (Ijora) has higher concentration than this lake, and others have nearly the same concentration with the Lakes as shown in Figure 6. Hale et al. [11] reported concentrations of total PBDEs (tetra- and pentaBDE) of 76 μ g/kg dw in soil near a polyurethane foam manufacturing facility in the United States, and 13.6 μ g/kg dw in soil downwind from the facility.

Conclusion

The study revealed that the water quality is degrading due to the negative impact of economic activities around this Lagoon. All measured physical and chemical parameters met the acceptable criteria, except dissolved oxygen, turbidity, and anomaly pH level. The air quality is also poor.

Ijora has the highest PBDEs concentration in the Lagos Lagoon and most of the sediments of Lagos Lagoon have high concentration of PBDEs as shown in the study with comparison with various levels worldwide. This result also shows that BDE 28 predominates in all locations followed by BDE 99. Ijora and Makoko and Apapa are highly concentrated with PBDEs, having the highest total PBDE in sediment and closer to the highest total PBDE concentration in water sample. PBDEs in sediment samples likely originated from a combination of point and non-point sources and atmospheric deposition. Larger amounts of industrial activities (such as dismantling electronic products, cables and wires, oils depot and chemical plants), indiscriminate waste disposal such as land filling, open dumps and incineration, plastic debris were likely responsible for the high concentrations of PBDEs in sediment samples of this area and in other sample locations. This could result in various health effects such as endocrine disruption, neurotoxicity, thyroid hormonal issues and brain dysfunction, and other chronic health effects on both human and animals.

PBDEs which have become ubiquitous environmental pollutants in the developed Western countries, is increasing gradually in our environment, accumulating in our water bodies. The extent and adverse health effects of their presence in the food chain, air, water, sediments and consumer products is beginning to emerge.

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