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Fingerprints of Polychlorinated Biphenyl (PCB) Congener and Homologous Profile of the Pra River Basin Sediment

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Abstract

Polychlorinated biphenyls are toxic, man-made, organic chemicals that have hazardous effects on the environment and our health, yet their occurrence in sediments and water from drinking water sources in Ghana has not been extensively studied. This study focused on assessment of the PCB congeners and homologous profile of sites in the Pra River basin, an important drinking water source in Ghana, considered to be potentially contaminated with polychlorinated biphenyl (PCB). Sediment samples collected from twenty communities along the river Pra basin were analyzed for 12 PCB congeners. Several statistical applications to congener and homologous profile comparisons were used to compare the potential similarity of seasonal and site PCB profiles. The statistical methods included coefficient of determination R^2 metric, principal component analysis and cluster analysis. Generally, sediment PCB concentrations ($\sum_{12}$ PCB congeners) in the dry season 98.78 - 242.16 ng/g (average 167.02 ± 31.39 ng/g) were higher than those of the wet season 60.99 - 158.11 ng/g (average 112.56 ± 27.09 ng/g). Jointly these methods were able to determine the degree of similarity between the wet and dry season PCB profiles, as well as the homologous and PCB congener profiles similarity of the sampling sites.

Subject Areas

Biological Chemistry

Keywords

Polychlorinated Biphenyls (PCBs), Congeners, Homologues, Sediments, Pra River, Coefficient of Determination, R^2, Cluster Analysis, Principal Component Analysis (PCA)

1. Introduction

Polychlorinated biphenyls (PCBs) are one of the groups of persistent organic compounds (POPs) that have contributed significantly to human exposure to hazardous pollutants [1]-[5]. These compounds were banned in 1979 due to their persistence and wide range of toxicity to health and the environment [6]. A PCB congener is a single unique PCB compound that is named based on the total number of chlorine substituents and the position of each chlorine. Whiles PCB Homologs are subcategories of PCB congeners that have equal numbers of chlorine substituents. Polychlorinated biphenyls congeners have varied toxicity. The dioxin-like PCBs (DL-PCBs) are coplanar, such that all the carbons and chlorines lie in the same plane, and are more toxic than other PCBs [7].

The total PCB concentration (∑PCB) is the sum of the individual congener concentrations. The congener profile is viewed as individual congener concentrations or relative congener concentrations, or the percent contribution of each congener to ∑PCB in an environmental sample. The PCB different homologues can then be determined from the congener profile, and their concentrations are calculated as the summation of congeners with the equivalent number of chlorines [8]. Even though PCBs undergo degradation and persist for many years in the environment [9] [10], the congener-specific method is used for environmental investigations of PCB homologous and sources in complex environmental systems [11] [12].

Include references [13] and [14] in the text: Worldwide PCBs have been determined in several environmental media in many regions and countries [15]-[19], and in drinking water sources [13] [20]-[22]. In Ghana it has been detected in electronic waste and air samples [23]; in breast milk at levels that might pose potential health risks for their children [24], soils and coastal lagoon ecosystems [14] [24]-[26].

Even though PCBs have been detected in some environmental samples such as sediments, soils, oysters and lagoon water samples from Ghana, however, the presence of these pollutants in drinking water sources and their potential health and effects have not been investigated extensively. Information on the PCBs in sediments from major drinking water sources, such as the Pra Rivers is lacking. The health, ecological and environmental effects of PCBs have been assessed based on data from this study. The objectives of this paper are to present the spatial and temporal PCB homologous and congener profiles of sediment from the Pra River basin of Ghana, and to compare the homologous and congener profiles for the wet and drying seasons. The method of comparisons included the use of cluster analysis and principal component analysis (PCA) which provides sample groupings due to similar compositional attributes, and coefficient of determination R^2 [27]-[30].

2. Study Area and Methodology

2.1. Study Area

The Pra River basin is located in the southwestern part of Ghana and drains into the Gulf of Guinea [31]. The river and its tributaries provide water for irrigation, domestic, and industrial use for communities within and beyond the basin. The study was conducted in a section of the Pra Rivers basin in the Central and Western regions of Ghana (**Figure 1**). The sampling locations were assigned a location number (P) (**Table 1**).

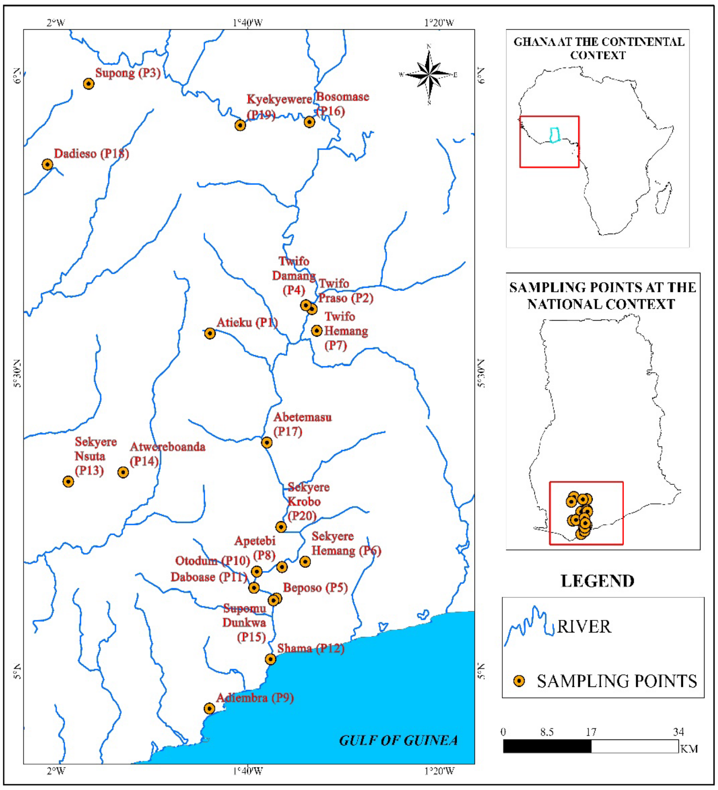


Figure 1. Study area showing sampling sites.

Table 1. Sampling locations with their coordinates.

Sampling Site Name	Site designation	Coordinates (N, W)
Atieku	P1	5.56667, - 1.73240
Twifo Praso	P2	5.36599, - 1.32599
Supong	P3	5.98580, - 1.94344
Twifo Damang	P4	5.61379, - 1.56545
Beposo	P5	5.12163, - 1.61658
Sekyere Hemang	P6	5.183333, - 1.56667

Continued

Twifo Hemang	P7	5.57118, - 1.54650
Apetebi	P8	5.17440, - 1.60710
Adiembra	P9	4.93673, - 1.73319
Otodum	P10	5.16667, - 165100
Daboase	P11	5.13850, - 1.65910
Shama	P12	5.01948, - 1.62690
Sekyere Nsuta	P13	5.31778, - 1.97879
Atwereboanda	P14	5.33333, - 1.88333
Supomu Dunkwa	P15	5.118269, - 1.62188
Bosomase	P16	5.92160, - 1.55930
Abetemasu	P17	5.38333, - 1.63333
Dadieso	P18	5.85025, - 2.01507
Kyekyewere	P19	5.91599, - 1.67971
Sekyere Krobo	P20	5.24156, - 1.60860

2.2. Data Collection and Analysis

The sampling locations were assigned a location number (P) and sampled according to a predetermined, randomized plan. The samples were collected from the Pra Rivers in the 2021 dry and minor wet seasons respectively November to December and August to September in Twenty composite samples and their duplicates were collected from each sampling point. Each composite sample consisted of four 50 g grab samples collected randomly using a corer and inserted into at least 10 cm below the surface of the sediment and then transferred into a clean labelled containers for storage. The samples were placed in an ice chest containing ice and transported to the laboratory and stored at 4˚C in a refrigerator.

2.3. Chemicals and Standards

Standard PCB mixture of "CEN PCB Congener Mix 1" (IUPAC No.'s 18, 28, 31, 44, 52, 101, 118, 138, 149, 153, 180, and 194) (Supelco, USA), which comprises 10µg/L of each component in heptane was employed for the six-point calibration technique. For the analysis, an internal standard solution was added to samples before extraction and it consisted of 50ng/L 4,4'-dibromobiphenyl, Supelco (USA) in n-heptane. First-grade quality solvents namely n-heptane, methanol and acetone were also used for the analytical procedure, respectively. These solvents were procured from the Wako Pure Chemicals, Japan. The laboratory blanks, 18Ω water and washed sand (sediment laboratory blank) were purchased from Sigma-Aldrich.

2.4. Extraction of PCBs

The sediments from the Pra River basin were spiked with 4,4' -dibromobiphenyl as internal standard and extracted following the methods previously described by [31] [32], with n-hexane/acetone (1:1 v/v). All eluates from the Silicagel and Florisil cartridges (Sep-Pac1 Vac 6 cc, Waters) were concentrated in volume to 0.5 ml under nitrogen gas flow. Blank samples were analyzed according to the same procedures as those used for the samples, and no contamination was found. Duplicate extraction was dune.

2.5. Gas Chromatography-Mass Spectrometry Analysis of PCBs

The gas chromatography-mass spectrometry (GC-MS) (Shimadzu QP-2010 Ultra Japan equipped with flame ionization detector) was used for the PCB analysis. The capillary column used was a DB-5MS of length 30 m (0.25-μm internal diameter and 0.25-μm film thickness). The GC conditions were as follows: carrier gas- helium flow rate of 0.72 mL/min and a total flow of 31.8 mL/s and a linear velocity of 32.2 cm/s at purge flow of 3.0 mL/min using splitless injection mode. The injection and detector temperatures were set at 150˚C and 320˚C, respectively. The oven temperature was programmed to increase from 80 to 310˚C at a rate of 40˚C/min and then from 310 to 320˚C at a rate of 2˚C/min. A 1-μL injection volume was used. PCB homologues were determined by single ion monitoring (SIM).

The following quantifying and qualifying ions were monitored simultaneously: m/z values were 256 and 258 for trichlorobiphenyls; 290 and 292 for tetrachlorobiphenyls; 326 and 328 for pentachlorobiphenyls; 360 and 362 for hexachlorobiphenyls; 394 and 396 for heptachlorobiphenyls; and 312 for 4,4'-dibromo biphenyl. All the quality control steps were followed. Laboratory blanks for sediment were subjected to the same analytical procedures as applied to the original samples. However, no significant peaks were observed for the analytes of interest.

The identification of PCB peaks was based on their relative retention times (RRTs) (**Table 2**), and on intensity ratios of the monitored ions for quantification using gas chromatography-mass spectrometry (GC-MS). The concentrations of PCBs were quantified according to the internal standard method. A solution of 4,4'-dibromobiphenyl was used as an internal standard. The calibration technique employed was an internal standard multi-point (six point) calibration using several standard solutions. The PCB concentration in an analyzed sample was calculated as an average value for duplicate.

Table 2. Retention times for the PCB analytes standards replicate samples.

Analyte	Retention time
PCB 18	7.97
PCB 28	8.16
PCB 31	8.27
PCB 44	8.39
PCB 52	10.23
PCB 101	9.89

Continued

PCB 118	10.86
PCB 138	11.12
PCB 149	10.63
PCB 153	10.76
PCB 194	12.14
PCB 180	11.94

For the twenty samples, 3 blank samples were analyzed and the average concentrations obtained in the blanks was used to correct all concentration results [32]. The retention times for the PCB analytes standards are given. The concentrations of the individual PCBs congeners in ng/g were calculated on dry weight basis, and the total PCBs concentration (ΣPCB) calculated by summing up the concentrations of the individual PCB congeners.

2.6. Statistical Analysis

The data was analyzed for the distribution statistics, percent detection and variation (coefficient of variation). Multivariate analysis, principal component analysis (PCA) and cluster analysis (CA) and correlation matrix, and coefficient of determination R^2 were used in the study to provide a better insight and understanding of the groupings due to similar compositional attributes of the sediment PCBs.

The coefficient of variation (CV) is the ratio of the standard deviation to the mean, making it useful for comparing variability across datasets with different units or scales [33]. Principal Component Analysis (PCA) reduces data dimensionality while preserving information by generating uncorrelated principal components from the original variables. It has been applied to environmental data to identify patterns among physical, biological, and chemical factors [34]-[36]. Cluster analysis, an exploratory technique, groups similar data points to identify patterns, such as congeners with shared characteristics.

3. Results and Discussion

3.1. Quality Control

The results of the quality control analysis for the limit of detection (LoD), limit of quantification (LOQ); calibration linearity, recovery and the precision reported as relative percent difference (RPDw and RPD) for duplicate samples for the wet sand dry seasons. The recovery ranged from 75.20 to 105.70%, and were within the acceptable limit of 100 ± 15%. The precision reported as relative percent difference RPD ranged between 2.41 % and 10.19%, lower than the recommended limit of 15%. The calibration linearity R^2 waw high, ranged from 0.990-0.998 and is acceptable.

3.2. Congener Detection Frequency

The detection frequency of various congeners detected in the samples (**Figure 2**), ranged from 45% - 100%. The predominant congeners were PCB 18, 31 153 and 180. PCB 31 recorded 100% detection.

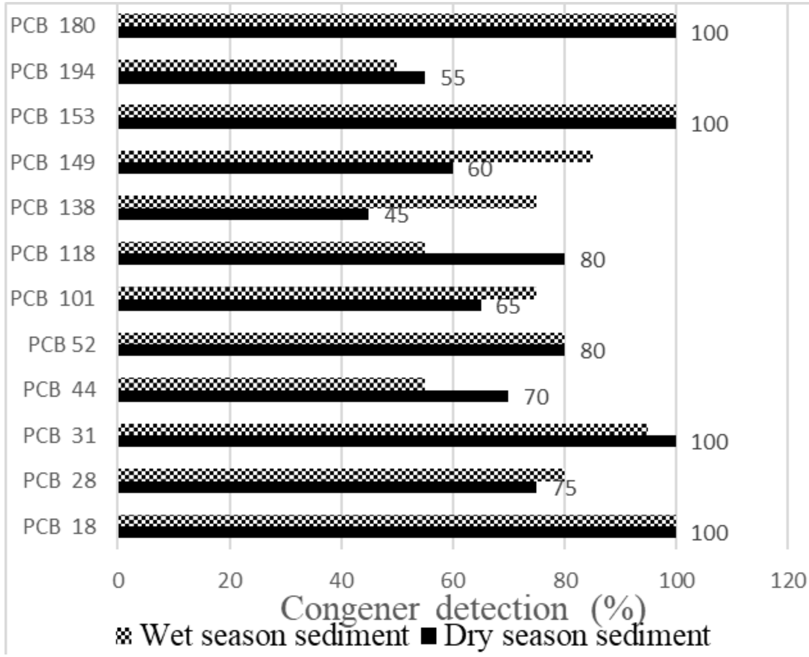
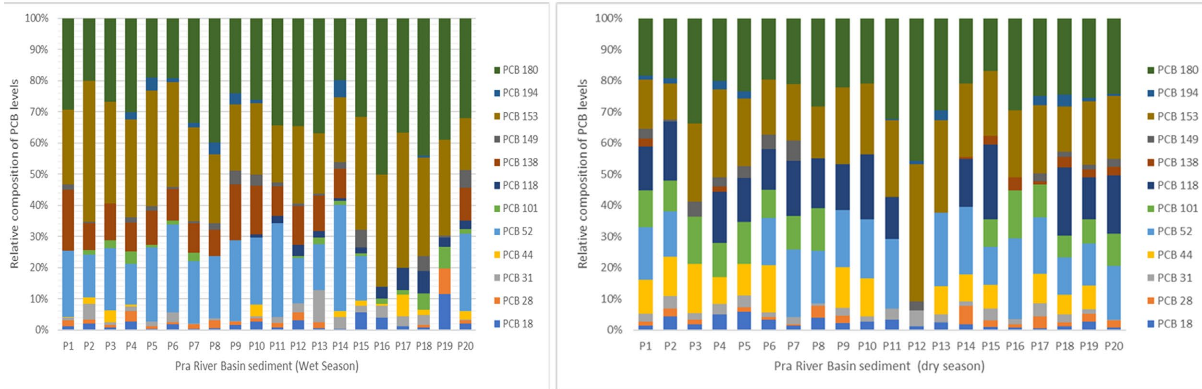


Figure 2. Proportions of PCB congeners detect in soils sediment.

In the drying season only, but PCB 18, 53 and 180 recorded 100% detection in both seasons. PCBs 194 and 138 recorded the least percentage detections in the wet and dry seasons respectively. Except for PCBs 18, 153 and 180, some of the other congeners were not detected in some samples. It is reported that that PCB 18 and PCB 44 were among predominant congeners found in the Udu River water, Nigeria, accounting for 8.90% and 15.6% respectively, of the total PCB burden in the sediments [37]. PCBs 28 (0.018-0.042 μg/L) have been found amongst dominant congeners in Mexico City [38].

3.3. PCB Congener Profile

The PCB congener profile in the sediments dry and minor wet seasons are shown (**Figure 3**). The relative compositions of the PCB congeners show that the more congeners with concentrations above the LOQ in the dry season than in the wet season. For both seasons the dominant congeners were PCBs 180 >153 > 52, and the least were PCB 44 for the wet seasons and PCB 194 for dry seasons (**Table 3**), whiles PCB 138 was rather dominant in the wet season. The compositions of PCBs 52, 138, 149, 153, 180 and 194 were relatively higher in the wet season compared to the dry season. The mean congener concentrations (mean ± sd) based on the dry weight (dw) for the wet season ranged between 1.31 ± 1.16 ng/g (PCB 44) and 35.14 ± 12.85 ng/g (PCB 180); and for the dry season 1.78 ± 2.28 ng/g (PCB194) and 40.08 ± 6.94 ng/g (PCB 180). The overall mean for the period ranged from 1.64 ± 2.1 (PCB 194) to 37.61 ± 6.94 (PCB 180).



(a) (b)

Figure 3. PCB congeners profile of sediments in the (a) wet and (b) dry seasons.

Table 3. Distribution Statistic of PCB Congener Profile of Pra River Sediments.

		Concentration of PCB (ng/g).												
		PCB 18	PCB 28	PCB 31	PCB 44	PCB 52	PCB 101	PCB 118	PCB 138	PCB 149	PCB 153	PCB 194	PCB 180	ΣPCB
Dry & wet Seasons	Mean	3.13	2.30	3.44	6.96	22.35	6.98	12.93	6.21	2.60	33.65	1.64	37.61	139.79
	Std	2.58	2.40	2.64	8.90	13.04	8.51	15.36	6.45	2.74	9.02	2.21	10.49	39.97
	CV	82.30	104.21	76.94	127.83	58.33	121.90	118.86	103.89	105.27	26.80	135.04	27.90	28.59
	Median	2.52	1.65	2.98	2.03	25.45	2.12	3.56	4.25	1.67	33.36	0.30	37.12	143.53
	Min	0.39	nd	nd	nd	nd	nd	nd	nd	nd	18.76	nd	19.35	38.5
	Max	10.38	11.28	12.02	30.45	42.45	24.45	50.78	17.08	9.87	58.89	7.86	63.13	338.64
	PCB%	2.24	1.65	2.46	4.98	17.32	4.99	9.25	4.44	1.86	24.07	1.17	26.90	100.00
Minor wet Seasons	Mean	2.19	1.71	2.61	1.31	20.07	1.89	1.80	10.09	2.23	32.04	1.49	35.14	112.57
	Std	2.01	1.58	2.83	1.61	12.48	1.85	2.22	6.22	2.33	10.91	2.19	12.85	27.09
	CV	91.78	92.40	108.60	123.13	62.16	98.13	123.35	61.63	104.32	34.04	146.74	36.56	24.07
	Median	1.22	1.315	2.21	0.385	19.79	1.46	0.98	12.295	1.315	33.85	0.0039	33.17	113.275
	Min	0.39	nd	nd	nd	nd	nd	nd	nd	nd	19.76	nd	19.35	60.99
	Max	6.97	5.04	12.02	4.67	42.45	5.90	7.82	17.08	7.98	58.89	5.82	63.13	158.11
	PCB%	1.95	1.52	2.32	1.16	17.83	1.68	1.60	8.96	1.98	28.47	1.32	31.22	100.00
Dry- Seasons	Mean	4.08	2.89	4.26	12.61	24.63	12.07	24.05	2.34	2.97	35.25	1.78	40.08	167.02
	Std	2.78	2.93	2.21	9.63	13.50	9.51	14.79	3.90	3.11	6.52	2.28	6.94	31.39
	CV	68.21	101.20	51.80	76.34	54.82	78.83	61.49	167.01	104.80	18.49	127.88	17.31	18.79
	Median	3.455	2.32	3.735	13.73	28.205	15.135	24.78	nd	2.87	32.825	0.785	37.725	161.925
	Min	0.89	nd	0.22	nd	nd	nd	nd	nd	nd	27,75	nd	32.1	98.78
	Max	10.38	11.28	9.46	30.45	41.89	24.45	50.78	15.89	9.87	45.56	7.86	56.56	242.16
	PCB%	2.44	1.73	2.55	7.55	14.75	7.23	14.40	1.40	1.78	21.11	1.07	23.99	100.00

The total concentration of ∑PCB at the various sampling locations is are presented in **Table 4**.

Table 4. Total PCB concentrations in sediments from the Pra river basin.

∑ PCB (ng/g)			
Site	Wet season	Dry season	Average
P1	80.95	183	131.98
P2	130.13	242.16	186.15
P3	106.36	139.63	123.00
P4	145.26	161.04	153.15
P5	109.33	152.97	131.15
P6	101.2	184.69	142.95
P7	150.63	162.81	156.72
P8	158.11	153.74	155.93
P9	89.64	156.34	122.99
P10	90.64	165.13	127.89
P11	115.76	175.45	145.61
P12	137.66	98.78	118.22
P13	115.59	139.38	127.49
P14	124.29	192.89	158.59
P15	120.14	210.97	165.56
P16	93.07	147.42	120.25
P17	67.43	143.9	105.67
P18	110.96	206.86	158.91
P19	60.99	178.24	119.62
P20	143.168	144.98	144.07
Mean	112.565	167.019	139.79
Std	27.0914	31.3879	29.24
CV	24.0673	18.793	21.43
Median	113.275	161.925	137.60

With the exception of P12 at Shama, where the sediment collected in wet season recorded higher ∑PCB 137.66 ng/g compared to the dry season, 98.78 ng/g, at all other locations the dry season samples had higher levels of ∑PCB. The highest ∑PCB levels recorded in the wet seasons (158.11 ng/g) and dry (242.16 ng/g) seasons respectively and were in sediments from P8 (Apetebi) and P2 (Twifo Praso). The least ∑PCB concentrations were recorded at Kyekyewere P19 (60.99 ng/g) and Shama P12 (98.78 ng/g) in the wet and dry seasons respectively. The seasonal variability of PCBs could be due to the joint effect of effluent deposition, dilution of the river in the wet season. fluxes due to illegal mining in the river and transport ability of PCBs as they tend to remain attached to particles of soil or sediment.

The levels recorded in this study are higher than levels reported for sediment samples collected from Bizerte lagoon, Tunisia, 0.8 to 14.6 ng/g dw (average value, 3.9 ng/g [39]; sediments collected from The Ghar El Melh lagoon, Tunisia [40], ranged from not detected to 3.987 ng/g; sediments from the Portugal Sado Estuary and in its coastal area 4.9-114 ng/g [41]; and 0.33–69 ng/g, reported by Cui, in 2020 [42].

3.4. PCB Homologous Profile

In general, the PCB homologous profile of the sediments indicated that Hexa-Cl was the most predominant homologous, and Octa-Cl the lest dominant (**Figure 4**). Elsewhere highly chlorinated PCBs (penta-and hepta-chlorobiphenyls congeners) in superficial sediments collected from the Ghar El Melh lagoon in Tunisia were found to be the predominant homologous [40]. In surface sediment samples collected from Bizerte lagoon, Tunisia the hexa and hepta were the predominant congeners accounting for 60 % of the total PCBs. In Ria Formosa lagoon (Portugal), the tri- and tetra-chlorinated biphenyls were the most abundant congeners in sediment followed by the hexa- (20%), penta- (11%) and hepta and octa-chlorobiphenyls (9%) [43].

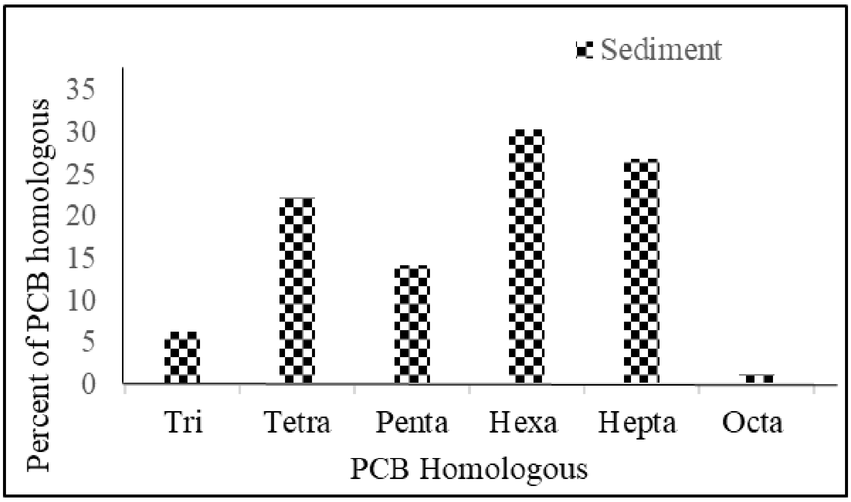


Figure 4. PCB Homologous composition of sediment samples from the Pra river basin.

Other studies have shown that sediment PCB profiles are typically dominated by lightly to moderately chlorinated homologs (2 - 6 Cl) [44]. Except for the Hexa-chloro which recorded higher homologous concentration in the dry season than the wet season, all the others recorded higher concentration the wet season than the dry season. The total homologous concentration (**Figure 5**). follower the order Octa-Cl (1.64 ng/g) < Tri-Cl (6.34 ng/g) <Penta-Cl (19.90 ng/g) < Tetra-Cl (29.31 ng/g) < Hepta-Cl (37.61 ng/g) < Hexa-Cl (42.42 ng/g). The highly chlorinated PCB 194 (Octa-chloro) was found at low concentrations in both seasons, which in agreement with other observation [45].

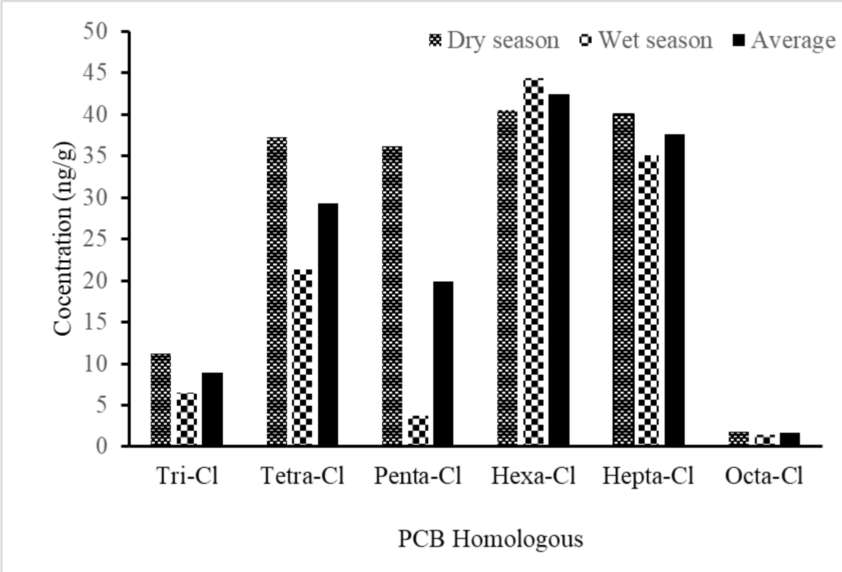


Figure 5. PCB homologous profile of sediments in the wet and dry seasons.

The PCB homologous distribution at the sampling locations is shown for the dry season (**Table 5**), and the minor wet season (**Table 6**). The distribution showed large variations, with the Octa-chloro showing the largest variation in the dry and wet seasons shown by the coefficient of variation; The respective coefficient of variation (CV%) respectively 128% and 147%. The tri, hexa and hepta homologues were present at all the sites, but the tri-chloro PCB showed greater varying concentration levels. The homologue concentration follows the same order for both the dry and wet seasons, where hexa, > hepta,> tetra > penta > tri > octa. The hexa and hepta homologue recorded relatively higher concentrations. The order of variation for the homologous distribution in the dry season was Octa-Cl > Penta-Cl > Tri-Cl > Tetra-Cl> Hepta-Cl > Hexa-Cl; and for the wet season Octa-Cl > Penta-Cl> Tetra-Cl > Tri-Cl > Hepta-Cl > Hexa-Cl.

Table 5. Levels of PCB homologous in sediments from the Pra River basin.

Collected in the dry season						
PCB Homologous concentration in sediments (ng/g)						
Site	Tri-Cl	Tetra-Cl	Penta-Cl	Hexa-Cl	Hepta-Cl	Octa-Cl
P1	9.67	50.66	47.43	39.45	33.45	2.34
P2	26.29	66.01	69.67	29.42	46.43	4.34
P3	6.2	18.34	41.12	34.87	39.1	nd
P4	13.57	13.67	44.39	52.74	32.1	4.57
P5	16.88	15.45	42.32	38.89	35.76	3.67
P6	10.51	55.9	40.52	41.41	36.35	nd
P7	6.27	33.67	52.12	38.21	32.54	nd
P8	15.22	29.9	28.72	29.56	50.34	nd

Continued

P9	10.99	49.3	22.76	38.73	34.56	nd
P10	7.13	51.57	34.54	37.34	34.55	nd
P11	12.1	39.1	23.65	43.45	56.56	0.59
P12	6.16	nd	nd	46.32	45.22	1.08
P13	7.07	45.51	nd	41.24	41.23	4.33
P14	17.58	58.67	29.67	46.65	40.32	nd
P15	14.49	41.78	69.54	49.59	35.57	nd
P16	4.91	35.65	20.98	45.43	40.45	nd
P17	12.33	39.57	15.37	36.74	35.67	4.22
P18	10.45	37.63	59.84	40.32	50.76	7.86
P19	12.08	37.43	37.72	43.78	45.56	1.67
P20	4.85	25.11	42.12	36.93	34.99	0.98
Mean	11.24	37.25	36.12	40.55	40.08	1.78
sd	5.27	16.63	19.30	5.949	6.936	2.28
Median	10.75	38.37	39.12	39.89	37.73	0.79
CV%	69.0	44.6	53.0	14.7	17.3	128.0
Min	4.85	nd	nd	29.42	32.1	nd
Max	26.29	66.01	69.67	52.74	56.56	7.86

Table 6. Levels of PCB homologous in sediments from the Pra River basin sampled in the minor wet season.

	PCB homologous concentration (ng/g)					
Site	Tri-Cl	Tetra-Cl	Penta-Cl	Hexa-Cl	Hepta-Cl	Octa-Cl
P1	3.19	17.43	nd	36.57	23.76	nd
P2	10.81	20.56	1.89	70.9	25.97	nd
P3	2.7	25.3	2.57	47.34	28.45	nd
P4	10.98	19.79	5.9	61.32	43.71	3.56
P5	3.02	25.9	0.98	54.04	20.83	4.56
P6	5.61	28.56	1.34	45	19.35	1.34
P7	3.08	30.02	4.31	60.42	50.35	2.45
P8	5.85	31.74	nd	51.57	63.13	5.82
P9	2.67	23.1	nd	39.08	21.45	3.34
P10	3.98	22.84	0.97	38.21	23.67	0.97
P11	4.9	34.89	2.65	33.61	39.71	nd
P12	11.67	20.13	5.95	52.46	47.45	nd
P13	14.84	17.03	4.76	36.31	42.65	nd
P14	5.26	44.62	2.57	40.3	24.67	6.87
P15	9.39	19.19	3.24	50.43	37.89	nd

Continued

P16	7.18	0.54	5.1	33.6	46.65	nd
P17	2.99	4.67	5.73	29.34	24.7	nd
P18	5.41	1.88	13.6	40.43	48.76	0.88
P19	12.01	dn	6.08	19.1	23.8	nd
P20	4.67	39.46	6.02	47.17	45.84	0.01
Mean	6.51	21.38	3.68	44.36	35.14	1.49
sd	3.75	12.39	3.21	12.21	12.85	2.19
Median	5.345	21.7	2.95	42.72	33.17	0.01
CV%	56.0	58.0	87.0	27.5	36.6	147.0
Min	2.67	nd	nd	19.1	19.35	nd
Max	14.84	44.62	13.6	70.9	63.13	6.87

3.5. Seasonal PCB Profile Comparison

To assess the nature of the similarity between the congener profiled in the two seasons, the PCB concentrations in the samples collected in the dry season were correlated with those recorded in the wet seasons (**Figure 6**). The similarity between any two congener profiles is defined through the use of the coefficient of determination R^2 found by plotting the congener concentration results of one sample or season versus the other sample, and calculating the square of the correlation coefficient (r) [28] [29] [46].

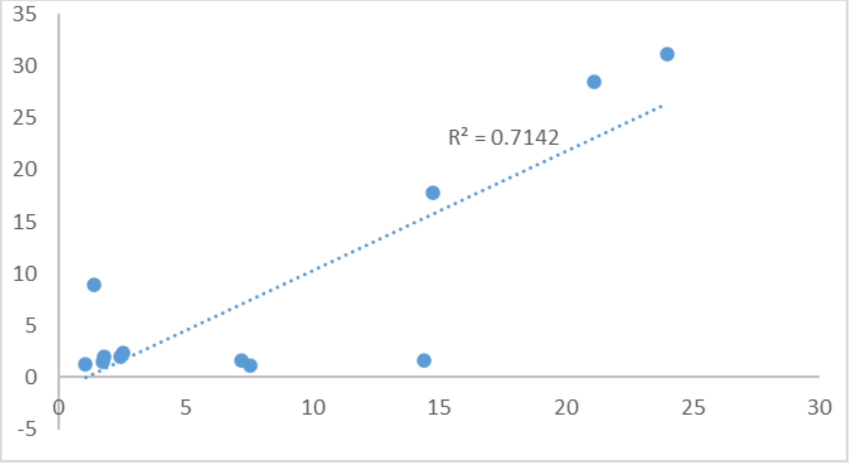


Figure 6. Concentration matrices for dry and wet seasons PCBs.

An R^2 value of 1.0 indicates a perfect match between the profiles, while an R^2 value of 0.0 indicates no relationship between profiles. The following criteria has been applied to qualified the degree of similarity between two sample congener profiles:

A fingerprinting match for R^2 is 0.9 or higher ($r \le 0.949$).

Very similar fingerprints, R^2 is 0.8 to 0.89 ($0.894 \le r \le 0.943$).

Similar fingerprints R^2 is 0.7 to 0.79 ($0.837 \le r \le 0.888$).

Ambiguous relationship R^2 is 0.6 to 0.69 ($0.775 \le r \le 0.831$).

Distinctly different fingerprints R^2 less than 0.6 ($r < 0.775$).

The R^2 obtained for the sediment samples data was $R^2 = 0.714$; $P \le 0.05$, which suggests that the fingerprints of PCB congener profile were similar between the two seasons. Furthermore, there is a strong correlation between the sediment PCB congener profiles of the wet and dry seasons ($R = 0.845$; $P \le 0.05$), which accounts for 71.4% of the variation in the sediment PCB data.

3.6. Sampling Locations PCB Profile Comparison

Since R^2 value does not change for comparison of the PCB congener fingerprints between two sampling points irrespective of whether the profiles are normalized or not., the congener concentrations were not normalized (e.g., divide each congener concentration by the sum of congeners). The correlation coefficients are shown (Table 7 and Table 8). Very few sediment sampling sites showed similarity.

Table 7. Correlation matrices for sampling site PCB profile.

	P1	P2	P3	P4	P5	P6	P7	P8	P9	P10	P11	P12	P13	P14	P15	P16	P17	P18	P19	P20
P1	1.00																			
P2	−0.02	1.00																		
P3	0.22	0.85	1.00																	
P4	0.53	0.60	0.61	1.00																
P5	0.09	0.33	0.21	0.65	1.00															
P6	0.38	0.73	0.88	0.48	0.10	1.00														
P7	0.34	−0.32	−0.19	0.10	−0.07	−0.33	1.00													
P8	0.64	0.53	0.70	0.52	0.09	0.87	−0.01	1.00												
P9	0.33	−0.06	0.04	0.40	0.27	−0.26	0.69	−0.11	1.00											
P10	0.15	0.22	0.29	0.34	0.03	0.36	−0.33	0.16	−0.18	1.00										
P11	−0.02	0.13	0.28	0.29	−0.10	−0.07	0.09	−0.22	0.53	0.20	1.00									
P12	0.46	0.28	0.51	0.57	0.11	0.31	0.12	0.19	0.39	0.26	0.65	1.00								
P13	0.19	0.49	0.42	0.41	0.01	0.30	−0.23	0.04	0.09	0.36	0.48	0.75	1.00							
P14	0.18	0.45	0.73	0.26	−0.19	0.66	−0.30	0.52	−0.10	0.14	0.49	0.48	0.31	1.00						
P15	−0.06	0.66	0.64	0.57	0.19	0.32	−0.15	0.02	0.27	0.45	0.73	0.71	0.80	0.45	1.00					
P16	0.52	0.00	0.05	0.65	0.32	0.01	0.39	0.20	0.32	0.13	0.26	0.46	0.24	0.07	0.24	1.00				
P17	0.54	0.09	0.26	0.71	0.30	0.23	0.23	0.39	0.23	0.46	0.32	0.45	0.14	0.30	0.30	0.86	1.00			
P18	−0.18	0.49	0.66	0.31	0.14	0.54	−0.34	0.31	−0.22	0.52	0.31	0.24	0.12	0.65	0.52	0.03	0.41	1.00		
P19	0.41	0.28	0.34	0.72	0.60	0.20	0.26	0.30	0.26	0.16	0.06	0.57	0.29	0.05	0.37	0.59	0.60	0.28	1.00	
P20	0.28	0.59	0.70	0.64	0.51	0.61	−0.29	0.35	0.05	0.51	0.20	0.68	0.63	0.36	0.66	0.16	0.31	0.53	0.65	1.00

Table 8. Coefficient of determination (R^2) for correlation matrices of sampling site PCBs.

	P1	P2	P3	P4	P5	P6	P7	P8	P9	P10	P11	P12	P13	P14	P15	P16	P17	P18	P19	P20
P1	1.000																			
P2	0.000	1.000																		
P3	0.048	0.728	1.000																	
P4	0.277	0.356	0.373	1.000																
P5	0.009	0.109	0.042	0.429	1.000															
P6	0.142	0.530	0.778	0.228	0.010	1.000														
P7	0.114	0.102	0.037	0.009	0.004	0.106	1.000													
P8	0.406	0.277	0.490	0.274	0.009	0.755	0.000	1.000												
P9	0.110	0.003	0.002	0.164	0.075	0.066	0.475	0.013	1.000											
P10	0.023	0.051	0.085	0.113	0.001	0.126	0.109	0.026	0.032	1.000										
P11	0.000	0.017	0.079	0.083	0.011	0.005	0.008	0.050	0.279	0.032	1.000									
P12	0.212	0.077	0.262	0.330	0.011	0.097	0.013	0.037	0.148	0.279	0.425	1.000								
P13	0.035	0.245	0.179	0.166	0.000	0.091	0.052	0.002	0.008	0.148	0.234	0.181	1.000							
P14	0.034	0.201	0.529	0.067	0.036	0.435	0.088	0.270	0.009	0.008	0.240	0.055	0.098	1.000						
P15	0.003	0.433	0.404	0.321	0.035	0.103	0.022	0.001	0.075	0.009	0.534	0.058	0.634	0.202	1.000					
P16	0.268	0.000	0.002	0.422	0.104	0.000	0.148	0.042	0.105	0.075	0.069	0.285	0.057	0.005	0.055	1.000				
P17	0.293	0.008	0.066	0.501	0.091	0.055	0.051	0.149	0.055	0.105	0.100	0.005	0.020	0.088	0.091	0.743	1.000			
P18	0.033	0.240	0.437	0.095	0.021	0.291	0.117	0.095	0.050	0.055	0.094	0.010	0.015	0.422	0.274	0.001	0.167	1.000		
P19	0.169	0.079	0.116	0.521	0.355	0.040	0.069	0.090	0.066	0.050	0.004	0.009	0.086	0.002	0.136	0.348	0.365	0.077	1.000	
P20	0.078	0.346	0.485	0.415	0.256	0.367	0.083	0.126	0.002	0.066	0.040	0.000	0.398	0.128	0.437	0.026	0.099	0.277	0.055	1.00

Fingerprint: P6 and P3, $R^2 = 0.778$; P8 and P6, $R^2 = 0.755$**;** P2 and P3, $R^2 = 0.727$; whiles P15 and P13, $R^2 = 0.634$ showed an ambiguous relationship. All the other site comparisons indicated distinctly different fingerprints.

3.7. Hierarchical Cluster Analysis

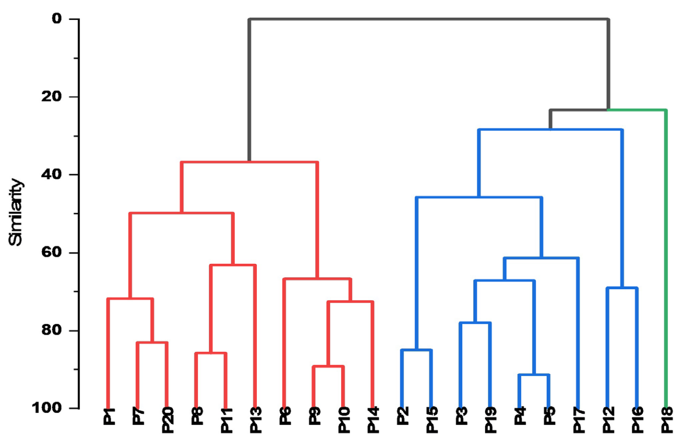


Figure 7. Dendrogram of sediment sites PCB profiles.

The dendrogram shows the hierarchical clustering of the sampling sites. This shows the order of formation and the level of similarities amongst the sampling sites with respect to their PCB congener profiles over the period (**Figure 7**). Clusters with the least Euclidean distance (shortest branch) are most similar, with the extent of similarity decreasing with increasing distance between clusters (increasing distance of branch). The clusters are dissimilar if the distance between them is about half the whole Euclidian distance. The results of cluster analysis for the wet and dry seasons are shown (**Figure 7**).

Sampling sites that were similar are arranged in order to decrease similarity between sites. Points P4 and P5 were the most similar relative to the congener profile followed by P9 and P10 > P8 and P11 > P2 and P15 > P7 and P20. The similarity levels of the sites for any one of the seasons, wet (**Figure 8(a)**) and dry (**Figure 8(b)**) were higher than that of the combine (**Figure 7**).

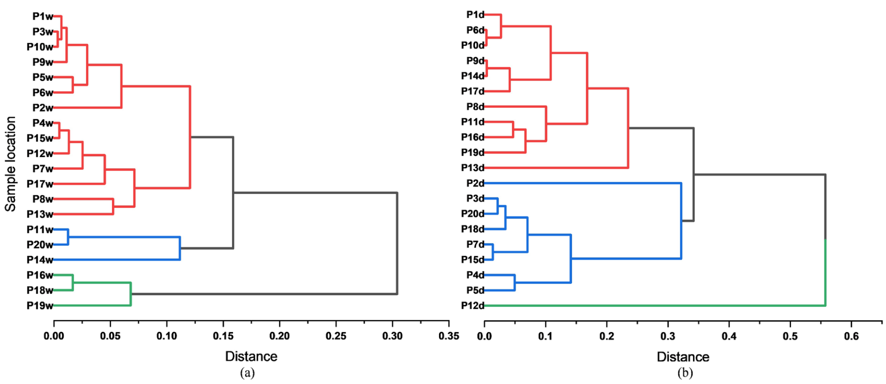


Figure 8. Dendrogram of sediment sites PCB profiles for the (a) wet and (b) dry seasons.

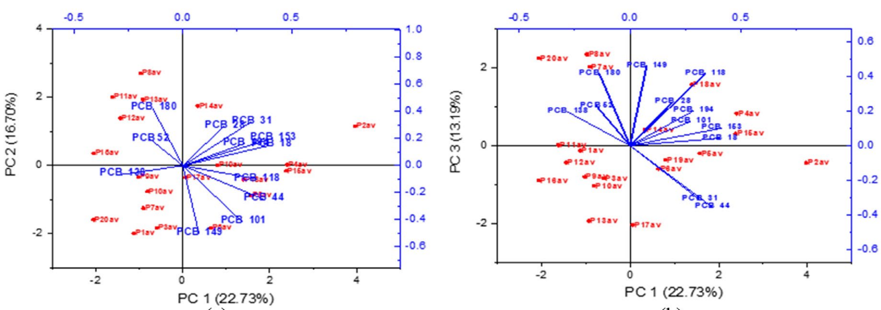
3.8. Principal Component Analysis of PCBs

In order to examine the relationships (similarities and effects) among the PCB congeners, and sampling points; the PCB profiles (mean of PCBs for the two seasons) was subjected to multivariate Principal Component Analysis (PCA). The results are shown (**Figures 8**). The PCA output, a biplot, shows both scores of samples (sampling points P) and loadings of congeners (vectors). It includes scores plot (eigenvalues) on the horizontal and vertical axis, and also describes the major variance orthogonally to the principal components. Each PC has one dimension, and the mid-point has value 0. The sign (positive or negative) indicates the direction that a given variable in that PC is going on a single dimension vector. Variables (Congener) with small score value has small role in explaining the variation due to the PC, whereas those with larger values have substantial roles in explaining the variation due to that PC. Congeners with score value 0, does not account for any of the variation on the PC

If the score values of samples taken from a specific area are similar to those of samples from another area, then these two areas have similar PCB congener and homologue profiles [27]. (The distances among sampling sites **red points** reflect their similarities. Sites (numbers) that are closer together have more similar congener profile, but far apart across a PC are negatively related. Congeners (PCBs) that are closer together occupy more sites in common.

Loading plots (angles between congener vectors) also depicts how congers correlate with one another. The approximated correlation between two variables (congeners) is equal to the cosine of the angle between the corresponding vectors, which also indicates the level of similarity [47]. A small angle less than 90˚ (those pointing in the same direction) have positive correlation between variables (congeners); 0˚ representing a perfect march. A large angle approaching 180° (with vectors or arrows pointing in opposite directions) suggests negative correlation, and Perpendicular vectors (angle 90˚) indicate the lack of correlation (no similarity) between the congeners they represent. The more parallel to a principal component axis is a vector, the more it contributes only to that PC the longer the vector, the more variability of this variable is represented by the two displayed principal components.

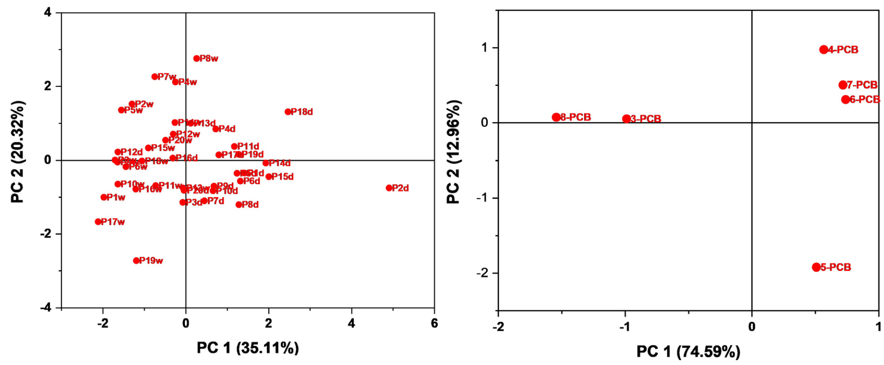
The scores plot is composed of a main principal component (Component 1, plotted on the horizontal axis) that captured most of the variability (22.73% of the variability). Principal Component 2 captured 16.70 % of the variability (**Figure 9(a)**); whiles Component 3 captured 13.19 % of the variability (**Figure 9(b)**). The loadings in PC2 are PCBs 28, 31, 52,101, 149 and 180 (**Figure 8(a)**). However, PCB 149 and 180 with the largest score (longest vector) contributes greatest to the variability in the component. Vectors pointing in the same direction (PCB 52 and 180), and also (101 and 149) correlate positively However, PCBs vectors of 52 and 180 are pointing in opposite directions to PCB 101, 149 and these have negative correlations. The correlations suggest that these congeners have common sources or mutual dependence and/or similar behavior during transport, or are subject to certain factors of control [27] [48].



(a) (b)

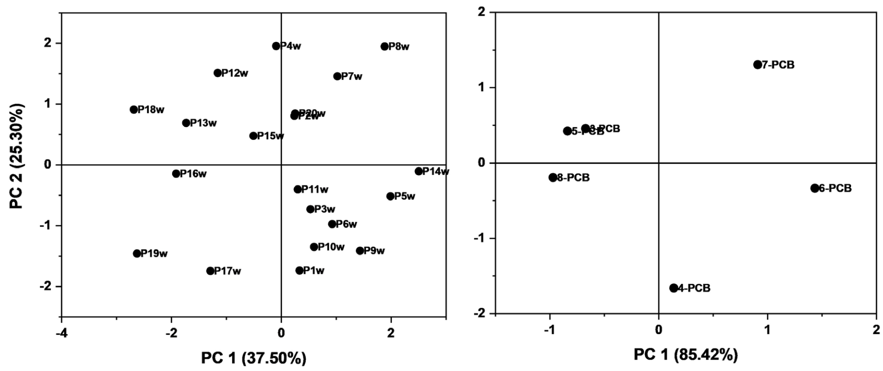
Figure 9. 2-D Principal component analysis of PCBs in sediments.

The PCA loadings plots of sampling sites (**Figure 10(a)**) and homologue groups (**Figure 10(b)**) with regards to the principal components show that the sample sites separation is due to the sites containing different predominant homologue groups (the presence of specific homologues in higher concentrations in some samples compared to others) In the dry season the samples from sites P2, P8, P11, P17, P18 and P19 contain higher tetra, hexa and hepta homologue concentrations, compared with other sites P3, P12, P13 and P16 (Figure 9(b)). In contrast, the P3, P12, P13 and P16 sediment contain tri and octa homologue concentrations, whiles P1, P5. P6. P14 and P15is dominated with the penta homologue concentrations. PCA showed that the majority of the variability in the data (74.59%) for the dry season can be explained by the first principal component (Component 1) Figure 9(b)). The PCA loadings plots of sampling sites and homologue groups in the wet season with regards to the principal components is shown (Figure 11). For the wet season, 85.59% of the variability in the data can be explained first principal component (Component 1).



(a) (b)

Figure 10. PCA loadings plots of (a) sampling sites and (b) homologue groups with regards to the principal components for the dry season.



(a) (b)

Figure 11. PCA loadings plots of (a) sampling sites and (b) homologue groups with regards to the principal components for the wet season.

3.9. Health and Ecological Implication

The presence of pollutants in sediment from this drinking water source raises concerns for both environmental and human health. PCBs are linked to cancer and various non-cancer effects on the immune, reproductive, nervous, and endocrine systems. Exposure in humans, wildlife, and aquatic organisms can lead to severe health risks. Assessing Toxic Equivalent (TEQ) and cancer risks helps determine the significance of PCB contamination. Ongoing monitoring is essential to prevent long-term bioaccumulation and protect the environment and human health.

4. Conclusions

Multiple approaches were used successfully for congener and homologous profile comparisons. The use of the linear correlation coefficient R^2 and PCA and cluster analysis have been applied to qualify the degree of similarity between samples and sampling sites. Except for two sites, P8 and P12., the sediment PCB congener concentration at all sites in the dry seasons 98.78 - 242.16 ng/g (average 67.0256 ± 31.39 ng/g) were higher than those recorded for the wet season 60.99 - 158.11 ng/g (average 112.56 ± 27.09 ng/g). However, the concentration of all the homologous groups were high in the dry season.

The wet season sediment samples have similar congener profiles to the dry season sediment samples collected from the same locations. The PCA loadings plot indicated similarity between the congener profiles for some sites, which was due to the significant presence of all the homologue congeners in sediment samples collected in both seasons. Very few sediment sampling sites showed similarity in fingerprint: P6 and P3, P8 and P6, and P2 and P3 showed similar relationship. P15 and P13 showed an ambiguous relationship, and all the other site comparisons indicated distinctly different fingerprints. Cluster analysis revealed that the similarity levels of the sites for the individual seasons were higher than that of the wet and dry seasons combined. PCA for homologous groups showed that the majority of the variability in the data (74.59%) and 85.59% for the dry and wet seasons respectively can be explained by a main principal component (Component 1). The PCA scores and loading plots provided reasons for the separation of the sediment sampling site. Different PCB congeners were dominant in the various sediments. In general, the statistical methods used proved to be effective in assessing the profile and similarities of the PCB-contaminated sites in the river basin.

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Conflicts of Interest

The authors declare that there are no known conflicts of interest associated with this publication and there has been no financial support from any external organization for this work that could have influenced its outcome. We wish to confirm that the manuscript has been read and approved by all named authors and that no other persons have satisfied the criteria for authorship but are not listed. The authors wish to state that, we owe no other person or a group, any financial obligation.

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