



Ecological Risk Assessment of High Molecular Weight Polycyclic Aromatic Hydrocarbons in Sediments from Qua Iboe River Estuary, South-South, Nigeria

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Abstract: This study sought to assess ecological risk of Polycyclic aromatic hydrocarbons (PAHs) in sediments collected from Qua Iboe River Estuary (QIRE). Sediment samples were collected from five stations and a control station along the estuary in the dry and wet seasons. PAHs in sediments were measured using gas chromatography coupled with a flame ionisation detector (GC/FID), and ecological risk on benthic organisms exposed to PAHs in sediment were evaluated using threshold effect concentration hazard quotient (TEC-HQ) and mean effect range median quotient (m-ERM-q) method prescribed by USEPA. The concentration of high molecular weight PAHs (HMW-PAHs) in the dry season ranged from 1.225×10^{-3} to 1.328×10^{-1} whereas during the wet season it ranged from 2.420×10^{-3} to 7.690×10^{-2} . There were no significant differences ($P < 0.05$) between the PAH levels for the wet and dry seasons. The results for the TEC-HQ and m-ERM-q values of HMW-PAHs for both seasons were less than the threshold values of 1 and 0.1, respectively. Hence, exposure to PAHs in sediments from QIRE may not pose any adverse biological effect on benthic organisms. However, regular monitoring of the level of HMW-PAHs in the sediment within QIRE is advocated to avoid bioaccumulation of organic pollutants.

Keyword: Polycyclic Aromatic Hydrocarbons, Ecological Risks, Qua Iboe River Estuary, Sediments

1. Introduction

Sediments are deposited materials consisting of organic matter in various stages of decomposition, particulate mineral matter and inorganic mineral of biogenic origin. Sediments can be used to establish the assimilative capacity of the environment, since they are pollutants trap. They contain a high level of pollutants that can pass on to the food chain or be mobilized by anthropogenic or natural means [1]. The presence of PAHs in surface sediment is credited to increase in atmospheric particulate matter originating from vehicles and combustion process such as gas flaring. However, sediments are dynamic as their components are subject to changes through several processes such as photo-oxidation, photochemical degradation, microbial degradation, evaporation and solubilisation [2]. PAHs partition preferentially onto the solid phase because of their low aqueous solubility and vapour pressure and the concentration

of PAHs in sediment of coastal estuaries are high [3]. It has been reported that PAHs are not usually present in the dissolved phase in a marine environment but are bound to the sediments or suspended matter and may become available to fish and other marine organisms through food chain. Sediments act as sink for anthropogenic pollutants thus, it has adverse effect on marine wildlife through benthic and bottom feeding organisms or can re-partition to the water column causing adverse effects to pelagic organisms [4]. High concentration of PAHs in sediment has been linked with neoplasm and other abnormalities in bottom-dwelling fish. Ecological risk assessment evaluates the likelihood that adverse ecological effect may occur or are occurring as a result of exposure to one or more pollutants in the environment [5]. Screening level ecological risk assessment performed on sediments evaluates the possibility of adverse ecological effect by PAHs, with the protection of benthic organisms from adverse effect selected as the assessment end

point. Oil related activities such as gas flaring, oil spillage and the use of boats for fishing and transportation are the major sources of polycyclic aromatic hydrocarbons (PAHs) in the marine ecosystem. Qua Iboe River estuary (QIRE) is one of the estuaries in the Niger Delta region of Nigeria with frequent oil spill occurrence and pipeline vandalism. Also, a petrochemical processing plant is located at the proximity of the lower reach of the estuary. The levels of PAHs in environmental samples from Qua Iboe River and other part of the Niger Delta have been reported [6, 7]. However, there is little or no information on the ecological risk induced by the exposure of benthic organisms to PAHs in sediment. This study was conducted to determine the level of high molecular weight PAHs (HMW-PAHs) in sediments from QIRE and assess their potential ecological risk on benthic organisms.

2. Materials and Methods

2.1. The Study Area and Site Description

Qua Iboe River covers about 60% of the local governments in Akwa Ibom state. Five sampling sites located at the lower reach of Qua Iboe River in Ibeno local government area close to a petrochemical effluent treatment and discharge plant were chosen as the examined sites while Ekpenekang in Etinan local government area of Akwa Ibom State, about 27km from the examined sites and is free from oil exploration and production activities was used as the Control. The global positioning system (GPS) coordinates of the different sites are: Okoroutip ($4^{\circ}55'5''N - 7^{\circ}54'47''E$), UkpeneKang ($4^{\circ}27'2''N - 8^{\circ}3'5''E$), Iwoachang ($4^{\circ}36'50''N - 7^{\circ}50'03''E$), Douglas creek ($4^{\circ}30'55''N - 8^{\circ}07''E$), Stubbs creek ($4^{\circ}34'41''N - 7^{\circ}59'47''E$), Ekpenekang ($4^{\circ}47'90''N - 7^{\circ}50'03''E$). Figure 1 is a map of the study area indicating the sample locations.

2.2. Sample Collection and Treatment

Sediment samples were collected from the river/creek banks monthly for 12 months (January to December, 2014) period spanning the dry and wet seasons of the study area using a plastic spatula sampler. Five sub-samples were collected at each sample location and were combined together in a stainless steel bowl to form a composite sample. A total of 360 sub-samples and 72 composite samples were collected. These samples were transported back to the laboratory in a cooler with crushed ice. In the laboratory sediment samples were air dried for one week, after drying, visible remains of organisms and debris were removed. The dried samples were ground into fine particles using pestle and mortar and sieved through a 2mm sieve (mesh) to remove ungrounded matters and separate the coarse fractions from the fine fractions. The fine samples were stored at $4^{\circ}C$ until further analysis.

Extraction and Clean up

The extraction of the analyte was carried out using a Soxhlet extractor according to the method described by Anyakora *et al.* [7] and Olabemiwo *et al.* [8].

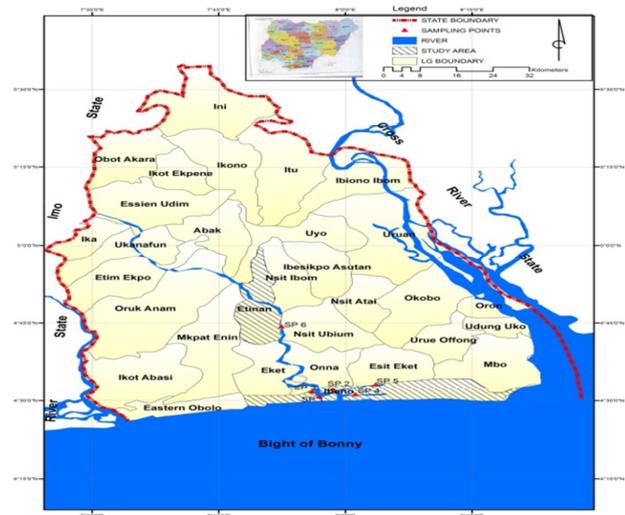


Figure 1. Map of Akwa Ibom State, showing sampling locations.

2.3. Determination of PAHs Concentration

The concentration of PAHs was determined using standard protocol described by Anyakora *et al.* [7]. The sample was automatically detected as it emerged from the column by a flame ionization detector (FID) by measuring the retention time. Usually the identification of the PAH compounds (analyte peaks) was achieved using Chemstation software and was based on matching their retention time with a mixture of PAH standards (16 USEPA priority PAHs) while quantification was obtained from the corresponding areas of the respective chromatograms. The surrogates namely acenaphthene- d_{10} , phenanthrene- d_{10} , chrysene- d_{12} and perylene- d_{12} from Smart solutions, USA were used as internal standard. Procedural blanks and solvent blanks were analysed and quantified with no PAHs found in these blanks. Prior to use, the GC was calibrated using a five point calibration curve established using dichloromethane-based standards (Accustandard PAH mix, $1000\mu g/ml$ in CH_2Cl_2). The coefficient of determination values (R^2) were greater than 0.87.

Chromatographic Conditions

The gas chromatograph was Agilent Hewlett Packard 5890 series II, coupled with flame ionization detector (FID) powered with HP Chemstation Rev. A. 0901 (10206) software. To identify and quantify PAH components, the following GC operating conditions was utilized:

Detector: Hydrogen at 35 ml/min; air at 250ml/min and nitrogen at 30 ml/min.

Oven: Initial temperature $65^{\circ}C$; final temperature; $325^{\circ}C$, Run time: 30 minute.

Inlet: Splitless injection was adopted using a rubber septum and the volume injected was $1\mu l$. The inlet temperature was $275^{\circ}C$, with a pressure of 14.8 Psi and total flow of $65.4ml/min$.

Column: The column was lined with 1,3-dimethyl polysiloxane with capillary HPS type of 30m length, 0.32mm wide bore diameter, 0.25 μm film diameter.

2.4. Ecological Risk Assessment

Ecological risk of PAHs in surface sediments to benthic organisms was assessed using the threshold effect concentration-hazard quotient (TEC-HQ) and the mean effect range median quotient (m-ERM-q) developed by (Khairy *et al.* [9] and Long *et al.* [10]. This was obtained from the equation below:

$$m - ERM - q = \frac{\sum(\frac{Ci}{ERMi})}{n} \tag{1}$$

Where Ci=Concentration of target PAH in sediment
ERMi =ERM value for the same target PAH
n=Number of PAHs

$$TEC - HQ = \frac{Ci}{TEC} \tag{2}$$

Where Ci=Concentration of target PAH in sediment
TEC=Threshold effect concentration (sediment quality guideline)

2.5. Statistical Analysis

All values were expressed as mean of three determinations ± standard deviation. Student’s t-test was used to compare between the mean of total PAH values in both the dry and wet seasons and a p< 0.05 was considered statistically significant. Pearson correlation coefficient was used to examine the relationship between PAH pairs in sediments. Statistical analyses were performed using SPSS statistics 17.0 windows.

3. Results and Discussion

3.1. Levels of PAHs in Sediments

Tables 1 and 2 show the level of PAHs in sediment from QIRE in both the dry and wet season. The least value during the dry season was recorded at the Control site (2.095E-06) while the highest value was recorded for B(b)Fat Douglas creek. During the wet season the result ranged from 1.615E-06 for chrysene at EkpeneUkpa to 1.259E-02 for B(b)F at Okoroutip. The concentrations of high molecular weight PAHs (HMW-PAHs) ranged from 7.4784E-05±6.7357E-06 to 5.87E-02±5.6862E-03 at Okoroutip, 4.9782E-05±1.5044E-06 to 4.8236E-02±2.814E-03 at Upenekang, 6.0910E-06±5.0332E-08 to 4.654E-03±4.5825E-04 at Iwoachang, 1.0435E-04±2.8431E-06 to 5.858E-02±4.6032E-03 at Douglas Creek, 3.0578E-06±9.0185E-08 to 1.3221E-02±9.0423E-03 at Stubbs creek and 2.959E-06 to 2.5166E-08 to 1.7927E-05±7.97517E-03 at EkpeneUkpa (control). The concentration of the total C-PAHs in the dry season was higher in some locations than the value for wet season. However, when compared with student’s t-test there was no significant difference (p< 0.05 between the two seasons. The interrelationships between individual PAHs in sediments during the dry and wet season are presented in Tables 3 and 4. The matrix for the wet season revealed that the correlation coefficients were mostly positive values although few negative values were recorded in the wet season.

Table 1. Mean concentration of high molecular weight polycyclic aromatic hydrocarbon in sediment from QIRE in the dry season (mg/kg).

PAH mixture	Statistics	Okoroutip	Upenekang	Iwoachang	Douglas Creek	Stubbs Creek	EkpeneUkpa
B(a)A	Mean± S.D.	7.4784E-05±6.7357E-06	4.9782E-05±1.5044E-06	6.5325E-06±6.5064E-08	1.6075E-06±1.9857E-06	2.115E-07±5.9561E-07	2.9986E-07±3.2419E-07
Chrysene	Mean± S.D.	9.0657E-06±4.414E-06	8.1836E-06±2.0033E-05	7.3011E-06±6.0928E-06	3.3653E-06±8.7842E-06	5.7555E-05±1.5874E-05	1.7927E-05±7.97517E-03
B(b)F	Mean± S.D.	5.87E-02±5.6862E-03	4.8236E-02±2.814E-03	4.654E-03±4.5825E-04	5.858E-03±4.6032E-03	4.9196E-04±7.5055E-04	2.3074E-04 to 5.5973E-05
B(k)F	Mean± S.D.	1.1334E-03±6.245E-05	1.8703E-03±8.888E-05	8.477E-04±4.9328E-06	1.7039E-03±9.0211E-04	3.0578E-06±9.0185E-08	2.959E-06 to 2.5166E-08
B(a)P	Mean± S.D.	4.0366E-04±2.1224E-05	4.8614E-04±1.3076E-05	2.3067E-04±7.5055E-07	1.0435E-04±2.8431E-06	6.408E-06±2.1830E-07	2.991E-06±3.5921E-07
Ind(1,2,3-cd)P	Mean± S.D.	0.7567E-02±6.937E-03	4.172E-02±5.428E-03	1.487E-02±7.0237E-05	9.379E-03±8.3865E-05	1.3221E-02±9.0423E-03	3.8377E-02±1.40E-05
D(ah)A	Mean± S.D.	9.1898E-05±4.459E-05	7.570E-05±6.8432E-05	9.4689E-05±3.6055E-07	2.6066E-05±6.6883E-06	2.6263E-05±2.1126E-05	5.911E-05±7.000E-08
B(ghl)P	Mean± S.D.	1.89630E-03±8.3865E-05	1.8604E-03±4.882E-05	6.0910E-03±5.0332E-08	1.2151E-03±4.441E-05	6.7871E-03±5.0964E-07	3.106E-03±6.03517E-07

Results are presented as Mean±SD

Table 2. Mean Concentration of high molecular weight polycyclic aromatic hydrocarbon in sediment from QIRE in the wet season(mg/kg).

PAH mixture	Statistics	Okoroutip	Upenekang	Iwoachang	Douglas	Stubb	EkpeneUkpa
B(a)A	Mean	8.033E-06±5.7743E-07	7.126E-06±4.297E-07	7.0915E-06±5.5293E-07	7.9155E-06±1.2220E-08	7.452E-06±8.6127E-07	3.153E-06±5.3702E-07
Chrysene	Mean	4.5345E-06±6.1329E-07	1.3544E-06±1.4730E-06	1.1250E-06±5.6888E-06	1.99030E-06±8.3865E-08	2.5853E-06±3.2140E-06	1.6156E-06±4.5092E-08
B(b)F	Mean	1.2599E-02±8.544E-04	1.5841E-02±5.644E-04	1.374E-02±8.962E-03	7.347E-03±4.267E-04	1.226E-02±6.1538E-03	2.0157E-02±4.735E-04
B(k)F	Mean	8.681E-06±8.681E-06	5.4650E-06±5.4650E-06	5.1397E-06±5.1397E-06	1.8781E-06±1.8781E-06	7.7433E-06±7.7433E-06	1.9143E-06±1.9143E-06

PAH mixture	Statistics	Okoroutip	Ukpenekang	Iwoachang	Douglas	Stubb	EkpeneUkpa
B(a)P	Mean	06±6.2939E-07	06±5.7873E-07	06±5.4884E-07	06±8.0208E-08	06±5.923E-07	06±1.2503E-07
		1.1733E-	7.9435E-06±8E-	1.0310E-	2.2915E-	1.0988E-	1.6715E-
Ind(1,2,3-cd)P	Mean	05±5.416E-06	08	05±5.5824E-06	06±4.4560E-07	05±5.5157E-06	06±3.2145E-08
		2.8949E-	8.480E-	1.406E-	2.563E-	5.671E-	2.635E-
D(ah)A	Mean	03±2.498E-04	03±6.506E-04	03±5.1316E-05	03±6.658E-05	03±7.085E-04	04±9.839E-06
		4.403E-	6.1924E-	4.4393E-	7.8003E-	2.2783E-	4.833E-
B(ghi)P	Mean	05±1.0008E-07	05±5.4580E-06	06±6.0044E-07	04±8.1445E-05	02±7.094E-03	05±6.550E-06
		6.4932E-	3.8633E-	1.459E-	1.0297E-	1.1943E-	9.066E-
		06±1.0214E-07	06±4.000E-07	05±5.0332E-07	05±2.00E-07	05±3.0550E-07	05±5.000E-07

Results are presented as Mean±SD

Note: B(a)A = Benzo(a)anthracene, B(b)F = Benzo(b)Fluoranthene, B(k)F = Benzo(k)Fluoranthene, B(a)P = Benzo (a) Pyrene, Ind (1,2,3-cd)P = Indeno(1,2,3-cd)Pyrene, D(ah)A = Dibenzo(a,h) Anthracene, B(ghi)P = Benzo(ghi)Perylene

Table 3. Correlation matrix for high molecular weight PAHs in sediments from QIRE during the dry season.

	B(a)A	Chrysene	B(b)F	B(k)F	B(a)P	Ind(1,2,3-cd)P	D(ah)A
B(a)A	1						
Chrysene	0.357	1					
B(b)F	0.376	-0.057	1				
B(k)F	0.892*	0.335	0.246	1			
B(a)P	0.932**	0.165	0.443	0.918**	1		
Ind(1,2,3-cd)P	0.421	0.885*	-0.183	0.577	0.351	1	
D(ah)A	0.283	-0.025	-0.166	0.643	0.368	0.349	1

**correlation is significant at 0.01 level (2-tailed), *correlation is significant at 0.05 level (2-tailed)

Table 4. Correlation matrix for high molecular weight PAHs in sediments from QIRE during the wet season.

	B(a)A	Chrysene	B(b)F	B(k)F	B(a)P	Ind(1,2,3-cd)P	D(ah)A
B(a)A	1						
Chrysene	0.825*	1					
B(b)F	0.769	0.666	1				
B(k)F	0.645	0.494	0.883*	1			
B(a)P	0.926**	0.831*	0.751	0.741	1		
Ind(1,2,3-cd)P	0.857*	0.826*	0.722	0.744	0.987**	1	
D(ah)A	0.962**	0.944*	0.758	0.633	0.940**	0.904*	1

**correlation is significant at 0.01 level (2-tailed), *correlation is significant at 0.05 level (2-tailed)

3.2. Ecological Risk Assessment of PAHs in Sediment

Tables 5 and 6 show the result of the TEC-HQ values for HMW-PAHs in sediment from QIRE during the dry and wet seasons. The TEC-HQ values for HMW-PAHs in the dry season ranged from 8.726E-06 for B(a)A at EkpeneUkpa to 2.086E-01 for Ind (1, 2, 3-cd) P atUkpenekang. The TEC-HQ

values for HMW-PAHs in the wet season ranged from 4.516E-06 for B(a) P atEkpeneUkpa to 2.295E-01 for B(b)F at Douglas creek. The values for the mean ERM quotient for HMW – PAHs in sediments from all locations for both dry and wet seasons are presented in Figure 2.

Table 5. TEL-THQ values of HMW-PAHs in sediment during the dry season.

HMW-PAHs	Okoroutip	Ukpenekang	Iwoachang	Douglas Creek	Stubbs Creeek	EkpeneUkpa
B(a)A	2.336E-03	1.555E-04	2.042E-05	5.021E-05	6.609E-06	9.368E-06
Chrysene	2.666E-03	2.406E-03	2.150E-04	9.897E-04	1.692E-03	5.278E-05
B(b)F	1.830E-01	1.507E-01	1.454E-02	1.830E-01	1.537E-02	7.209E-04
B(k)F	4.720E-03	7.791E-03	3.535E-03	7.095E-03	1.271E-05	8.729E-06
B(a)P	1.090E-03	1.313E-03	6.232E-05	2.818E-04	6.610E-02	8.053E-06
Ind(123-cd)P	1.378E-01	2.086E-01	7.435E-03	4.684E-02	4.376E-03	1.918E-03
D(ah)A	1.534E-02	1.261E-02	1.578E-03	4.343E-03	4.376E-03	9.851E-06
B(ghi)P	1.115E-02	1.094E-02	3.582E-05	7.147E-03	3.993E-05	1.827E-05

Table 6. TEL-THQ values of HMW-PAHs in sediment during the wet season.

HMW-PAHs	Okoroutip	Ukpenekang	Iwoachang	Douglas Creek	Stubb Creek	EkpeneUkpa
B(a)A	2.510E-05	2.226E-05	2.215E-05	2.473E-06	2.328E-05	9.853E-06
Chrysene	1.333E-05	3.982E-04	3.308E-05	5.852E-06	7.602E-05	4.750E-06
B(b)F	3.934E-02	4.950E-02	4.200E-02	2.295E-01	3.831E-02	6.296E-03
B(k)F	2.367E-05	2.277E-05	2.147E-05	7.825E-06	3.226E-05	7.975E-06
B(a)P	3.170E-05	2.141E-05	2.786E-05	6.191E-06	2.696E-05	4.516E-06
Ind(123-cd)P	1.297E-01	2.740E-02	7.030E-03	1.281E-02	2.835E-02	1.317E-03
D(ah)A	7.338E-04	1.028E-03	7.398E-05	1.305E-02	3.796E-01	7.555E-04
B(ghi)P	3.819E-05	2.272E-05	8.582E-05	6.052E-05	7.023E-05	5.333E-04

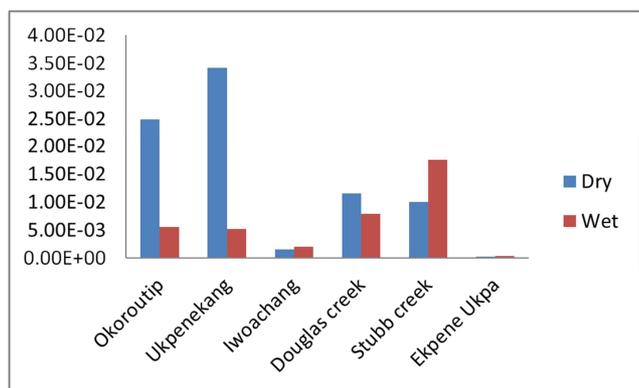


Figure 2. Mean ERM quotient of HMW- PAHs in sediment from QIRE.

The values of HMW- PAHs recorded in this study were higher than values reported elsewhere [4, 11]. When compared with standards for effect range low (ERL) and effect range median (ERM) which is 1.70mg/kg (ERL) and 9.60mg/kg (ERM), respectively, the ΣHMW-PAHs in this study were lower than the stipulated standards in ANZECC 2000 guidelines [12]. This implies that there is no possibility of acute adverse biological effect on the sediment dwelling organisms. The highest level of total HMW-PAHs was recorded at Douglas creek for both seasons and this may be due to the fact that the creek receives direct discharge of effluents from a petrochemical industry located close to it. PAH accumulation in sediment depends on the direct influence of anthropogenic activities such as combustion process of traffic and industrial activities [13]. Witter *et al.* [14] reported that sediments are contaminated by PAHs due to urbanization and other human activities. Seasonal variations in the levels of HMW-PAHs may be due to the effect of temperature. Temperature is significant for the fate of PAHs on soil and sediment. Temperature controls the hydrophobic nature of PAHs, their solubility, volatilization and bioavailability. PAHs are more easily distributed and can diffuse faster at high temperature; bioavailability and photo-oxidation of PAHs are enhanced at elevated temperature [2]. Generally, the variation of PAH content in sediment from different sample locations may be due to physicochemical properties of the sediment such as particulate organic matter and particle size. Studies have shown that the level of PAHs in a sediment is a function of photo-oxidation, chemical oxidation, microbial degradation, adsorption, leaching, bioaccumulation and volatilization [2, 13]. Results for the

interrelationship between PAH pairs in sediment from QIRE revealed that B(k)F, B(a)A, B (a)P, chrysene and Ind (1, 2, 3-cd) P may have originated from the same source during the dry season. The result from the correlation matrix during the wet season indicated similar PAH sources for most of the HMW PAHs. Correlation values give information on PAH sources and identify PAHs that can co-vary or co-exist in the environment [15]. Oil spillage occasioned by equipment failure took place at the examined sites during the wet season and this resulted in the introduction of PAHs into the marine ecosystem. Also, 24-hours gas flaring at the examined sites emits soot into the atmosphere and the soot may be deposited on sediments by dry or wet deposition. Typical PAH compounds such as chrysene, B(a)P, and benzofluoranthene have been identified in soot and the source of PAHs in sediment is atmospheric deposition followed by sedimentation [16]. When the threshold effect concentration hazard quotient (TEC – HQ) is less than 1, rare adverse ecological effects are possible but less frequent. At all the sample locations in this study in both seasons, TEC- HQ was less than 1, indicating that adverse ecological effect on benthic organisms associated with exposure to PAHs in sediment from QIRE may not occur. Mean effect range median quotient (m –ERM-q) can be categorized into 4 levels according to their probability of toxicity; ≤ 0.1 indicates an 11% probability of toxicity, 0.11 to 0.5 indicates 30% probability of toxicity, 0.51 to 1.5 indicates 46% probability of toxicity and >1.5 indicates 75% probability of toxicity [10]. The result obtained in this study revealed that the m- ERM-q was less than 0.1, This findings were similar to the one reported by Naser *et al.* [17], therefore the locations investigated in this study can be classified as having low probability of toxicity. However, regular monitoring of the level of HMW-PAHs in the sediment within QIRE is advocated to avoid bioaccumulation of organic pollutants.

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