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Levels of Polycyclic Aromatic Hydrocarbons in Soils of Ibeno, Nigeria; Distribution, Sources and Potential Risk

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Abstract:
The contamination of PAHs in soils may result in accumulation in the food chain, leading to potential health risks. This study was designed to determine the levels of PAHs in soils of Ibeno, Nigeria and evaluate their potential ecological and health risks. Soil samples were collected at Ukpenekang, Mkpanak, and Iwuchang districts of Ibeno and analysed for eighteen PAHs using gas chromatography- mass spectrometry. Carcinogenic and ecological risk assessments were performed using USEPA models. The total concentration of the 18 PAHs ranged from 10.3 to 276.6 µg/kg with a mean value of 71.9 µg/kg in Ukpenekang; 9.3 to 176.5µg/kg with a mean value of 58.7 µg/kg in Mkpanak and 11.4 to 179µg/kg with a mean value of 71.4 µg/kg in Iwuchang. The total PAHs in the soils were higher than guideline values for agricultural soils. The higher molecular weight (HMW) compound which constitute the 4 to 6-ring, were the most abundant components of the soil with percentage composition of 58.2 – 65.5%. The diagnostic ratios show fuel combustion as the source of PAH in the area, predominantly from gas flaring and venting in the area. The soils are potentially carcinogenic with total carcinogenic toxic equivalency factors (BaPeq) values ranging from 103.05 to 148.8µg/kg-Bapeq dry weight. Ecotoxicity studies revealed minimal effect of PAH on soil functions, with mean effects range median quotient (m-ERM-q) values ranging from 0.06 to 0.08. Continuous environmental monitoring, total elimination or improvement in the efficiency of gas flaring system used by oil prospecting companies can reduce the risks associated with these hazardous compounds.

Keywords: *Polycyclic aromatic hydrocarbons, soils, risk assessment, Ibeno*

1. Introduction

Polycyclic aromatic hydrocarbons (PAHs) are a group of organic compounds with fused benzene rings (Wei and Jen, 2007). They are divided into two categories: Low molecular weight (LMW) compounds composed of fewer than four rings, and high molecular weight (HMW) compounds of four or more rings. PAHs are widely distributed in air, soil and water and there are several hundred PAH compounds of which benzo[*a*]pyrene (BaP) is the best known. The major sources of PAH are petroleum and petroleum products or products of incomplete combustion of biomass and fossil fuels (Lima *et al*., 2005; Yu *et al*., 2014).

Once in the atmosphere, PAHs are attached to particles and are transported to the soil through wet and dry deposition. They adsorb readily to soil particle and are persistent in the soil as a result of their low solubility (Jiao *et al*., 2013). Human exposure to PAHs is by direct inhalation and dietary exposure and they may accumulate in organs as a result of their low solubility. PAHs are routinely used in various products such as pharmaceuticals photographic products, agricultural products thermosetting plastics and as lubricating materials and these products are known environmental contaminants (Nasher *et al*., 2013).

The accumulation of PAHs in soils may result in the contamination of food chains, leading to potential health risks. PAHs are known carcinogens and are reported to have genotoxic and mutagenic properties (Tam *et al*., 2001). The Toxic Equivalency Factors (TEFs) were models developed to facilitate risk assessment and regulatory control of exposure to PAHs (Zhang *et al*., 2009). TEFs increase the reliability of evaluation of the toxicity of a sample containing PAHs (Sandro *et al.*, 2010). Since BaP is well characterized toxicologically (Lodovici *et al* 2003), the risks of PAHs are calculated on the basis of BaP concentration.

Ibeno occupies the largest Atlantic coastline of more than 129 km in Akwa Ibom State, Nigeria.

Located in the mangrove swamp forest, the area has rain throughout the year with the peak between May and September. The climatic condition in Ibeno is favorable all year round for fishing and farming activities. The prime occupation of the people is fishing. The presence of oil exploration activities by oil prospecting and other service companies influence activities in the area.

Levels of PAHS in soils have been widely investigated along with their health risks in Nigeria and elsewhere (Maliszewska-Kordybach, 1999; Fagbote and Olanipekan, 2003; Oviasogie *et al*., 2006 and Song *et al*., 2008). Result of such studies show low to very high levels of soil PAHs in the studied areas. To date, no investigation has been conducted on the levels of PAH in soils of Ibeno and their associated health risks. Therefore, studies on PAH concentration and distribution in soils and their health risk is needful. The

aim of this study was to estimate the level of PAHs in soils of Ibeno and to determine the ecological and health risks associated with them. The results obtained will be useful for understanding the potential risks to the ecosystem and people of Ibeno.

2. Methodology

2.1. Description of the Study Area

The study areas were three villages of Mkpanak, Iwuoachang and Ukpenekang all in Ibeno Local Government Area. Mkpanak is located between latitude $4^0$33' 09.50"N and longitude $7^0$59' 50.94" 33E. Iwuoachang is located between latitude $4^0$34' 12.78"N and longitude $7^0$58' 03.14"E. Ukpenekang is located between latitude $4^0$34' 27.94"N and longitude 7^0 58' 32.32"E. Ibeno is characterized by distinct dry and wet seasons. The wet season which begins in April and ends in October is always characterized by heavy storms of short duration. Map of the study area is shown in Figure 1.

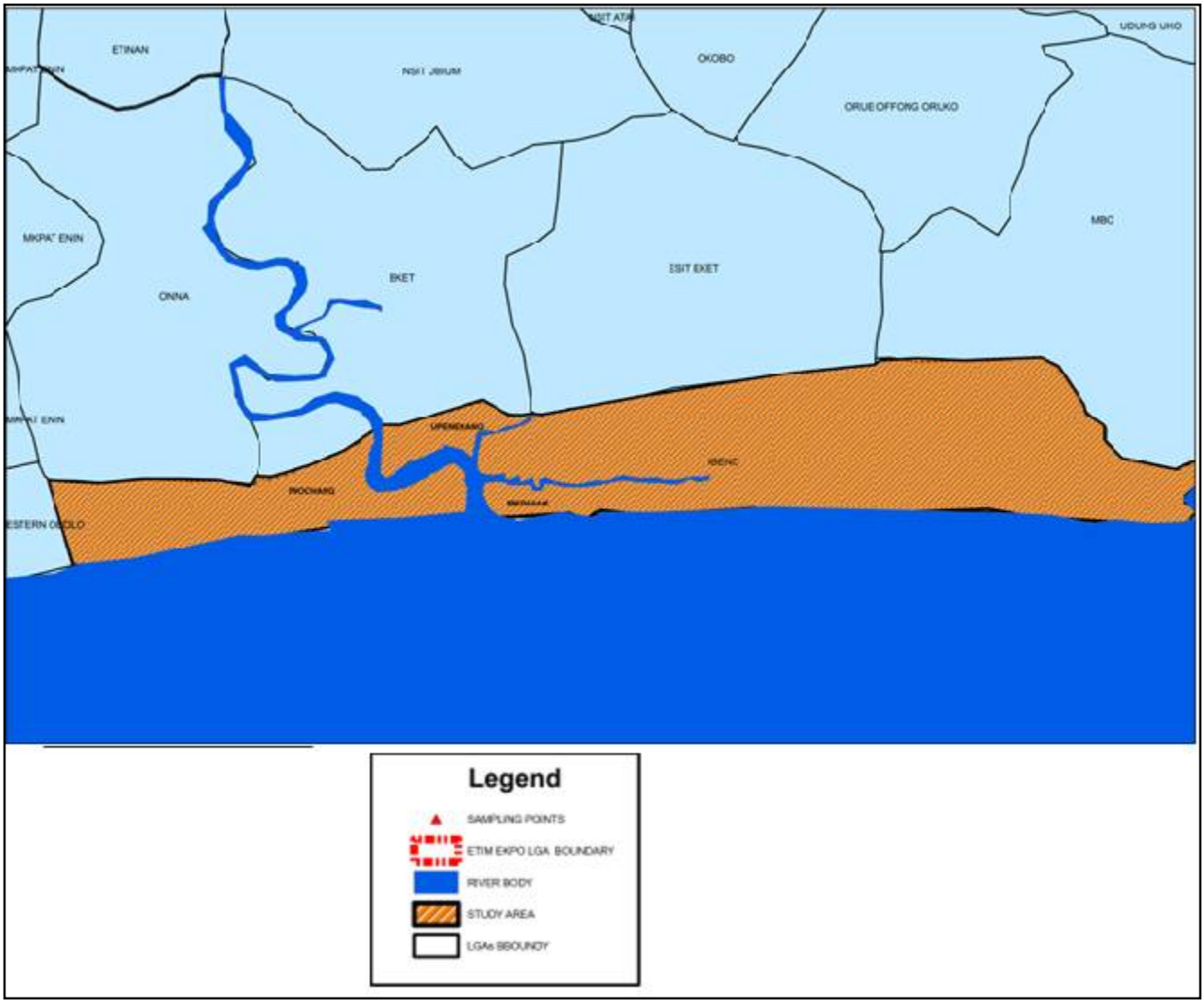

Figure 1: Map of Ibeno showing Iwuchang, Mkpanak and Ukpenekang

2.2. Collection of Soil Samples

Soils were sampled as described by Jiao *et al*. (2013). About 50 g each of fresh soil samples were collected at a depth of 0-20cm from several locations at the three villages. Each sample was a composite of subsamples taken from four locations within an area of about 100 × 100 m^2. Samples were dried at room temperature (Nadal *et al.*,2004) sieved through 100-mesh sieve and stored in glass bottles prior to analysis.

2.3. Sample Extraction Procedure

Exactly 30.0g of the soil sample was weighed into a clean extraction bottle. The sample was dried to free flowing texture with anhydrous sodium sulphate. 1ml of 30µg/ml pyrene d_{10} surrogate standard was added and a 100 ml mixture of 1:1 methylene chloride: acetone was also added immediately. The sample was extracted ultrasonically for 3 minutes. The extract was allowed to settle for

some minutes. The sample was carefully filtered through with a funnel fitted with phase separator filter paper into a clean beaker or amber colored extraction bottles washed with methylene chloride. The extraction was repeated for two or more times. The residue was washed with some volume of extracting solvent and filtered through the funnel. The sample extract was concentrated and then separated.

Calibration of the gas chromatography- Mass spectrometry system was then carried out using Agilent 6890N GC prior to calibration. A sequence table was selected from a sequence menus and the sequence table was created. The external standard mode for calibration mode was clicked in sequence parameter through edit/sequence parameters. The data for the standard mixture was acquired by injecting 3mg/l of the PAH standard mix of 5, 10, 15, 20 and 30mg/l in CH_2Cl_2 for a five level calibration. The names of the components of PAH mixture in the calibration was filled in. Subsequently, the other levels of the calibration were added through the calibration/update compound menu. The concentration of each calibration level was inserted through the update dialogue box. The correlation factor of the calibration was checked. Minimum acceptable correlation factor was 0.90.

2.4. Analysis of Sample Extracts on Agilent 6890N

A sequence table was created by filling the sample name, sample type, multiplier and dilution. 3µl of methylene chloride was injected as a blank sample via auto-samples mode. 3ml of extract was also injected after level into the injector via auto-sampler mode. If the extract peaks were generated above scale, the extract were diluted with methylene chloride and reanalyzed.

Data Processing: Calculation of level of PAH in the extract was as follows:

concentration (mg/kg) = $A \times B/C$

where;

A = data generated by equipment in mg/L

B = Total volume of extract in ml

C = weight of sample extracted in grammes.

2.5. Quality Control

Samples, blanks and spiked blanks were analysed with no interferences detected. Recovery efficiency was analysed with samples spiked with known amounts of PAH standards. Recovery of 16 PAHs ranged from 74% to 92% in the samples.

2.6. Identification of PAH Sources

Diagnostic ratios were used to distinguish the possible sources of PAH in the soils. The following ratios were used as source indicators: Ant/Ant+Phe, BaA/BaA+Chr and LMW-PAH to HMW-PAH.

2.7. Carcinogenic Risk Assessment

Health risk associated with the PAHs in soils were evaluated using the toxicity equivalency factor (TEF) method described by Nisbet and Lagoy (1992) (TEF for each PAH was an estimate of the relative toxicity of the PAH compounds compared to BaP). The total equivalent concentration was expressed as BaP equivalent (BaPeq).

BaPeq for individual PAH was estimated using the equation

$$BaPeq = \sum Cn \times TEFn$$

2.8. Ecological Risk Assessment

The mean ERM quotient approach was used to evaluate the possible ecotoxicity of PAHs in the soils. The mean ERM quotient values was calculated according to the method formula suggested by Long and Macdonald (1998) as:

$$m - ERM - q = \sum\left(\frac{Ci}{ERMi}\right)/n$$

where Ci is the concentration of PAH, *ERMi* is the ERM value for the same target PAH and *n* is the number of PAH.

2.9 Statistical Analysis

Data collected were subjected to statistical tests using Analysis of variance (ANOVA) at P = 0.05. All data analyses were done using SPSS software for windows version 12.0

3. *Results and Discussions*

3.1. Concentration of PAHs in Soils

The concentrations of PAHs from the three sites are shown in Table 1. All Eighteen PAHs were present in detectable amounts. The PAHs were Naphthalene (Nap), 2-Methylnapthylene (2MNap) 1-Methylnapthylene (1MNap) Acenapthylene (Acy), Acenaphthene (Ace), Fluorene (Flu), Phenanthrene (Phe), Anthracene (Ant) Fluoranthene (Fluo), Pyrene (Pyr), Benzo(a)anthracene (BaA), Chrysene (Chr), Benzo(b)fluoranthene (BbF), Benzo(k)fluoranthene (BkF), Benzo(a)pyrene (BaP), Dibenzo(a,h)anthracene (DBA), Benzo(g,h,i)perylene (B*ghi*P) and Indono(1,2,3-d) pyrene (IND). The total concentration of the 18 PAHs ranged from 10.3 to 276.6 µg/kg with a mean value of 71.9 µg/kg in Ukpenekang, from 9.3 to 176.5µg/kg with a mean value of 58.7 µg/kg in Mkpanak and from 11.4 to 179µg/kg with a mean value of 71.4 µg/kg in Iwuchang. Soils from Ukpenekang recorded the highest total PAHs concentration of 1284 µg/kg, while he lowest concentration of total PAHs in soils was at Mkpanak with a total PAH of 1049 µg/kg.

There were no statistically significant differences in the soil total PAHs (P>0.05) indicating that soils from the three areas had similar sources of anthropogenic input to their PAH levels.

PAHs	No. of Rings	Ukpenekang	Mkpanak	Iwuchang
Nap	2	90.9 ± 19	176.5 ± 29.4	156.4 ± 32.4
1MNap	2	82.6 ± 24	23.4 ± 7.9	102.8 ± 19.3
2MNap	2	65.5 ± 16	18.9 ± 6.2	97.3 ± 12.4
Acy	3	23.0 ±9	34.3 ± 8.8	90.6 ± 16.2
Ace	3	33.5 ± 6.4	42.8 ± 5.4	56.5 ± 11.5
Flu	3	10.3 ± 2.1	9.3 ± 1.4	11.4 ± 3.4
Phe	3	43.1 ± 5.7	51.6 ± 9.5	68.9 ± 9.3
Ant	3	79.7 ± 9.5	23.4 ± 5.2	18.0 ± 7.9
Flua	4	65.5 ± 11.2	39.8 ± 5.3	97.4 ± 10.5
Pyr	4	103.6 ± 18.6	53.7 ± 7.3	89.6 ± 13.2
BaA	4	54.4± 12.3	142.1 ± 18.4	179.3 ± 25.2
ChR	4	276.6 ± 34.5	56.8 ± 8.5	34.7± 8.9
B*b*F	5	41.3 ± 7.4	74.5 ± 10.2	33.5 ± 8.4
B*k*F	5	58.7 ± 9.4	73.6 ± 9.1	57.4 ± 10.2
B*a*P	5	32.1 ± 5.4	65.3 ± 7.4	51.2 ± 8.4
DBA	6	59.6 ± 7.1	53.9 ± 12.3	23.0 ± 6.9
B*ghi*P	6	98.8 ± 13.6	84.0 ± 10.7	88.6 ± 10.2
IND	6	73.2 ± 10.8	34.3 ± 8.6	29.3 ± 9.9
∑18PAHs		1284	1049	1278

Table 1: Concentrations of 18 PAHs in soils of Ibeno (µg/kg (mean ± SD))

The soils were moderately contaminated based on the classification by Baumard *et al.* (1998). Comparing the PAH levels in the study area with values obtained in other places, the concentrations were lower than those of New Orleans (647 - 40,692 µg/kg) (Mielke *et al*., 2001), Poland (80 – 7264 µg/kg) (Barbara *et al.*, 2008) but comparable to the Yellow River Delta (Yuan *et al*., 2008). The total PAHs in the soils were higher than 200 µg/kg, guideline values for agricultural soils (Wcislo, 1998).

3.2. Percentage Distribution of PAHs

The distribution of the PAHs in soils are shown in Figure 2. The results revealed that on average, the four-ring PAHs were the most dominant in the soils, with percentage distribution between 31.2% and 38.7%. The two-ring, three-ring, five and six-ring had percentage compositions of 23%, 14.6%, 14.1% and 15.0% respectively. The higher molecular weight (HMW) compound which constitute the 4 to 6-ring, were the most abundant components of the soil (Figure 3) with percentage composition of 58.2 – 65.5%. HMW (4-6 rings) are carcinogenic (Neff, 1979; Witt, 1995) while LMW compounds have significant toxicity. Dominance of the HMW fractions may be attributed to their lower volatility, solubility and higher persistence in soils compared to LMW compounds (Nasher *et al*., 2013). The source of HMW compounds to the soils might have been anthropogenic activities such as incomplete fuel combustion of vehicles and engine boats as well as spills from oil exploration activities in the area. Overall, the soils at Ibeno were contaminated above background levels and dominance of HMW compounds may have potential carcinogenic risks associated with the soils.

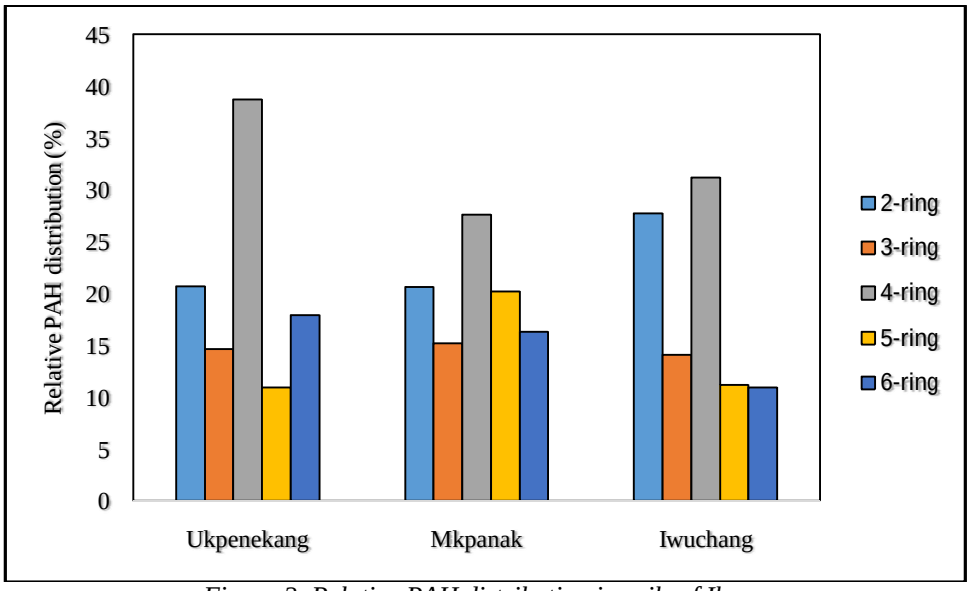

Figure 2: Relative PAH distribution in soils of Ibeno

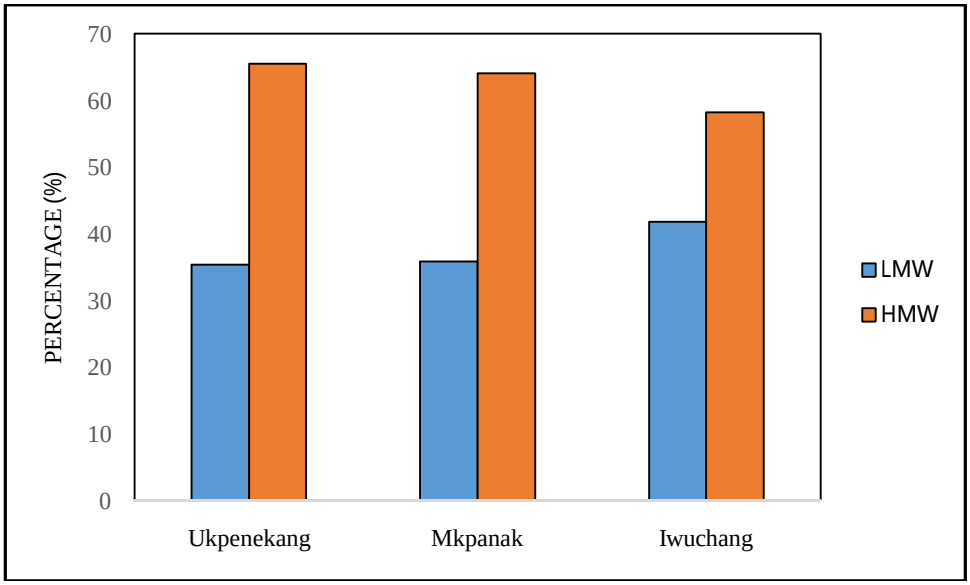

Figure 3: Relative percentages of ∑LMW and ∑HMW PAHs in soils of Ibeno, Nigeria

3.3. PAH Diagnostic Ratio Analysis

PAH diagnostic ratio is used as a tool for identifying and assessing pollution sources (Soclo *et al.*, 2000; Yunker *et al.*, 2002 and Zhang *et al.*, 2004). The diagnostic ratios used in the present study were BaA / BaA + Chr, Ant / Ant + Phe, Flue / Flue + Pyr and LMW/HMW ratio. Ant / Ant + Phe ratio of > 0.1 indicate dominance of heavy fuel composition, while < 0.1 indicate petroleum source, BaA / BaA + Chr ratio of 0.2 to 0.35 indicate mixed petrogenic and pyrogenic origin and > 0.35 indicate pyrogenic origin (Zhang, 2004). Table 2 shows the diagnostic PAH ratios for soils in Ibeno. The ratio of BaA / BaA + Chr were between 0.16 and 0.84 with mean of 0.57 suggestive pyrogenic origin. The Ant/ Ant + Phe ratios were between 0.21 and 0.63 indicating fuel combustion sources. The LMW/HMW ratios were low for most sites < 1 indicating pyrogenic origin of PAHs to the soils. Values of Flua /Flua + Pyr ratios is used to distinguish between different combustion origins such as burning of liquid fossil fuels or coal wood or grass (Jiao *et al*., 2013). The mean Flua /Flua+Pyr of 0.44 confirm the combustion of fuel as the main PAH source in the area. The diagnostic ratios show fuel combustion as the source of PAH in the area. However, Ibeno does not have heavy vehicular activities, therefore, the combustion source of PAH could very likely be predominantly from gas flaring and venting in the area. Ite *et al.* (2013) had reported high levels of PAH as result of gas flaring in the Niger Delta which corroborates the result of the present study.

		DIGNOSTIC RATIO		
Location	BaA/BaA+Chr	Ant/Ant+Phe	Flua/Flua+Pyr	LMW/HMW
Ukpenekang	0.16	0.63	0.38	0.53
Mkpanak	0.71	0.3	0.42	0.56
Iwuchang	0.84	0.21	0.52	0.72
Mean	0.57	0.38	0.44	0.6

Table 2: Diagnostic PAH ratios in the soils of Ibeno, Nigeria

3.4. Risk Assessment of PAHs

3.4.1. Carcinogenic Risk Assessment

Carcinogenic risk assessment was performed using total toxic BaP equivalent of PAHs. Toxic equivalency factor and total carcinogenic toxic equivalency factors for all soils studied are shown in Table 3. Total carcinogenic toxic equivalency factors ranged from 103.05 to 148.8µg/kg-Bapeq dry weight., with a mean of 120.48 µg/kg dry weight.. The results revealed that DBA, BaP, BaA, and BbF were the most contributors to total carcinogenic potency of the soil samples with values of 5.4 - 17.9 µg/kg for BaA, 3.3 – 4.1 µg/kg for BbF, 5.8 – 7.3 3 µg/kg for BkF, 32 – 51 3 µg/kg for BAP and 23 - 59 3 µg/kg for DBA. These PAHs are highly carcinogenic (Yu *et al*., 2014). BaP is considered the most hazardous of the PAHs (Wang *et al*., 2010). This shows that the soils are potentially carcinogenic. BaPeq values were higher than the target value of 32.96 µg/kg indicating the increased carcinogenic burden of soils in these areas. Comparing the result from the present study with those of other studies, the values obtained were comparable to those of surface soils in Terragona, Spain (124µg/kg BaPeq), higher than those from Liaohe estuarine soils (28.4 µg/kg) but lower than those in surface soils of India (650 µg/kg) and Shanghai, China (892 µg/kg BaPeq). The high carcinogenic potency of PAHs is an indication of high degree of carcinogenic risk.

PAHs	TEF (USEPA)	Ukpenekang	Mkpanak	Iwuchang
Nap	0.001	0.09	0.176	0.156
1Nap	0.001	0.082	0.023	0.102
2MNap	0.001	0.065	0.018	0.097
Acy	0.001	0.023	0.034	0.09
Ace	0.001	0.033	0.042	0.056
Flu	0.001	0.01	0.009	0.011
Phe	0.001	0.043	0.051	0.068
Ant	0.01	0.79	0.23	0.18
Flua	0.001	0.065	0.039	0.097
Pyr	0.001	0.103	0.053	0.089
BaA	0.1	5.4	14.2	17.9
ChR	0.001	0.276	0.056	0.034
B*b*F	0.1	4.1	7.4	3.3
B*k*F	0.1	5.8	7.3	5.7
B*a*P	1	32	65	51
DBA	1	59	53	23
B*ghi*P	0.01	0.98	0.84	0.88
IND	0.01	0.73	0.34	0.29
BaPeq		109.59	148.811	103.05

Table 3: Concentration of carcinogenic PAHs (µg/kg dry weight) BaP equivalent in soils from Ibeno, Nigeria

3.4.2. Ecotoxicity Studies

To assess the potency toxicity of soils in Ibeno, PAH levels in the soils were compared with the soil toxicity screening guidelines values. Screening values are concentrations of a contaminant in soil, which if exceeded may prompt further risk assessment. Mean ERM quotient m- ERM-q were calculated and compared with the screening values. According to Long *et al.* (2000), m-ERM-q are categorised according to their possibility of toxicity. Values ≤ 0.1 indicates an 11 percent probability of toxicity, 0.1 to 0.5 indicates a 30% probability of toxicity and 0.5 to 1.5 indicates a 46% probability of toxicity and > 1.5 indicates a 75% probability of toxicity (Nasher *et al.*, 2013). Calculated ERMs of individual PAHs and the mean ERM quotient of PAHs for this study are shown in Table 4. The m-ERM-q values ranged from 0.06 to 0.08 in the soils with a mean value of 0.07, which were below 0.1 indicating an 11 percent probability of toxicity and are therefore classified as low priority sites. Similar results were reported for sediments of Langkawi Island, Malaysia (Nasher *et al*., 2013). The implication of this result is that there was minimal effect of PAH on soil functions such as the capacity to act as substrate for plants including effect on seed germination and on soil organisms, which are important for proper soil function and nutrient cycling.

PAHs	ERM SSG	Ukpenekang	Mkpanak	Iwuchang
Nap	2100	0.043	0.084	0.07
1Nap	800	0.103	0.029	0.128
2MNap	670	0.097	0.026	0.144
Acy	640	0.035	0.053	0.141
Ace	500	0.066	0.084	0.112
Flu	540	0.019	0.017	0.025
Phe	1500	0.029	0.034	0.045
Ant	1100	0.071	0.021	0.016
Flua	5100	0.013	0.0076	0.019
Pyr	2600	0.039	0.02	0.034
BaA	1600	0.034	0.089	0.112
ChR	2800	0.098	0.02	0.012
B*b*F	-	-	-	-
B*k*F	-	-	-	-
B*a*P	1600	0.02	0.04	0.032
DBA	260	0.226	0.204	0.09
B*ghi*P	330	0.296	0.25	0.27
IND	950	0.076	0.035	0.031
m-ERM-q		0.08	0.06	0.08

Table 4: Mean ERM Quotients of PAHs in Soils of Ibeno

4. Conclusions

Eighteen PAHs were detected in soils of Ibeno, Nigeria. The total PAHs in the soils were higher than the 200 µg/kg guideline values for agricultural soils. The PAH compositions in soils were dominated with HMW PAH of 4 - 6 rings, which raises concern about their associated health risks. Diagnostic ratio analysis revealed input to the soil PAH prodimantly due to ongoing gas flaring activities of oil prospecting companies in Ibeno. The soils high carcinogenic potency of PAHs is an indication of high degree of carcinogenic risk. However, ecotoxicity studies revealed minimal effect of PAH on soil functions which include effect on seed germination and nutrient cycling. Continuous environmental monitoring and improvement in the efficiency of gas flaring system used by oil prospecting companies or stopping of gas flaring can reduce the risks associated with these hazardous compounds.

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