EVALUATION OF POLYCYCLIC AROMATIC HYDROCARBONS IN SOIL OF SELECTED URBAN AREAS OF DELTA STAT NIGERIA

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Abstract: Sixteen Polycyclic aromatic hydrocarbons (PAHs) grouped as endocrine disruption substances (EDSs) were determined for their concentrations, sources and human health risk. Naphthalene (Nap), acenaphthylene (Acy), acenaphthene (Ace), fluorine (Flu), phenanthrene (Phe), anthracene (Ant), fluoranthene (Flt), pyrene (Pyr), chrysene (Chr), benzo(a)anthracene (B[a]a), benzo(b)fluoranthene (B[b]f), benzo(k)fluoranthene (B[k]f), benzo(a)pyrene (B[a]p), indeno(1,2,3-cd)perylene (I[123-cd]p), benzo(ghi) perylene (B[ghi]p) and dibenzo(a,h) anthracene (D[ah]a) were analyzed in three urban soils of Agbor, Asaba and Issele-Ukwu, at 0-15 and 16-30 cm depth in wet and dry seasons. After extraction using ultrasonication with hexane and dichloromethane and clean-up, PAHs concentration was measured using gas chromatography equipped with mass spectrometer. The concentrations of Σ16 PAHs ranged from 178.0 to 787.0 μg/kg and 105.1 to 437.7 μg/kg in wet season and dry season respectively. PAHs concentrations varied significantly ($p<0.05$) between sites, soil profile and seasons. The Incremental Lifetime Cancer Risk values obtained were relatively above the tolerable target risk levels of 10^{-6} set by the US.EPA, this suggest a potential for human mutagenic and carcinogenic risk in the study area. Source estimates ratios suggest that PAHs were from petroleum, coal, biomass combustion and traffic origin.

Keywords: Concentrations, evaluation, PAHs, risk, sources

Introduction

Polycyclic aromatic hydrocarbons (PAHs) are group of several quotidian aromatic organic compounds having between two and six condensed benzenoid rings braced in angular, cluster or linear forms and possessing carbon and hydrogen atoms only, (Emoyan *et al.,* 2015a). Polycyclic aromatic hydrocarbons comprising two or three fused benzenoid rings are known as low molecular weight PAHs (LPAHS) while those consisting of more than three rings are refer to as high molecular weight PAHs (HPAHs). The HPAHs are more recalcitrant and toxic than the LPAHs (Wenzl *et al.,* 2006). Environmental PAHs originate mainly from natural (volcanic eruption and forest fire) and anthropogenic (oil spillage, incomplete combustion of fossil fuel, coke and other industrial processes) sources (Yang *et al.,* 2012). The persistent and recalcitrant nature of HPAHs is also related to the high resonance energies resulting from the dense clouds of π electrons surrounding the aromatic rings (Johnsen *et al.,* 2005). The solubility of PAHs increases linearly with decrease in molecular weight (Zhang *et al.,* 2006). PAHs are persistent and stable in the environment because of their high soil sorption capabilities and low aqueous solubility (Parrish *et al.,* 2004). Polycyclic aromatic hydrocarbons exhibit trans-boundary transport and carcinogenic and mutagenic characteristics hence their environment and human health concern due to their established pollution capabilities (US.EPA, 2002). They tend to be associated with particles and are widely transported by atmospheric pathways, resulting in elevated concentrations in soils and sediments (Yang *et al.,* 2006).

United States Environmental Protection Agency (US.EPA, 2002) has listed naphthalene (Nap), acenaphthylene (Acy), acenaphthene (Ace), fluorine (Flu), phenanthrene (Phe), anthracene (Ant), fluoranthene (Flt), pyrene (Pyr), chrysene (Chr), benzo(a)anthracene (B[a]a), benzo(b)fluoranthene (B[b]f), benzo(k)fluoranthene (B[k]f), benzo(a)pyrene (B[a]p), indeno(1,2,3-cd)perylene (I[123-cd]p), benzo(ghi) perylene (B[ghi]p) and dibenzo(a,h) anthracene (D[ah]a,) as sixteen PAHs which are most dangerous and occur frequently as 'priority pollutants' that need constant monitoring in the environment (USEPA, 2002; Banger *et. al.,* 2010).

Soil acts as a sink, natural buffer for transport, distribution and fate of PAHs contaminants in the biosphere (Benhaddya and Hadjel, 2014). Polycyclic aromatic hydrocarbons (HPAHs) are readily absorbed by organic matter in soils hence difficult to degrade. Also, PAHs accumulation in soil profile may contaminate the food chain which could increase the potential for human health risk (Yang *et al.,* 2014). Urban areas host about 50% of world population and most industrial and economic activities (Luo *et al.,* 2012; Iwegbue *et al.,* 2016). Relative high concentrations levels of PAHs have been reported in selected urban soils profile and various soil-human interaction may accelerate human vulnerability to the PAHs through ingestion, dermal contact and inhalation, (Abrahams, 2002; Banger *et al.,* 2010; Emoyan *et al.,* 2015a; Iwegbue *et al.,* 2016). Different studies has reported several distributions of PAHs in selected urban soil such as United Kingdom (Heywood *et al*., 2006; Vane *et al*., 2014), United States of America (Banger *et al.,* 2010; Chahal *et al.,* 2010; Yang *et al.,* 2014), Spain (Morillo *et al.,* 2008), China (Li *et al.,* 2006; Peng *et al.,* 2011; Wang *et al.,* 2013; Yu *et al.,* 2014), Nigeria (Okoro and Ikolo, 2007; Olajire *et al.,* 2007; Sojinu *et al*., 2010; Iwegbue *et al.,* 2016). However, literature indicates that there are no published environmental research studies on the concentrations, sources and human health risk of PAHs in the study area. Therefore, the objective of this study is to determine the concentrations and evaluate the sources and human health risk of PAHs. This would provide baseline for future study on local environmental quality, human health risk management and remediation and cleanup.

Materials and Method

Description of study area

The study area consists of the three urban soil of Agbor, Issele-uku and Asaba in Northern part of Delta State. Agbor lies on latitude $6^0 15135'$N and longitude $6^0 1139.12'$ E. Asaba lies on latitude $6^0 1221'$ N and longitude $6^0 4145'$ E while Issele-Uku lies on latitude $6^0 3197'$ N and longitude $6^0 4663'$ E. (Fig. 1). The climate and weather conditions are of the Niger Delta region with annual rainfall of 2673.8 mm and mean annual temperature of 32.8°C, (Uku and Tamunobereton-Ari, 2013). The dry season (October to March) is characterize with dusty haze (harmarttan) of the North-east winds, while in the wet

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season (April to September) is significantly marked with rain fall.
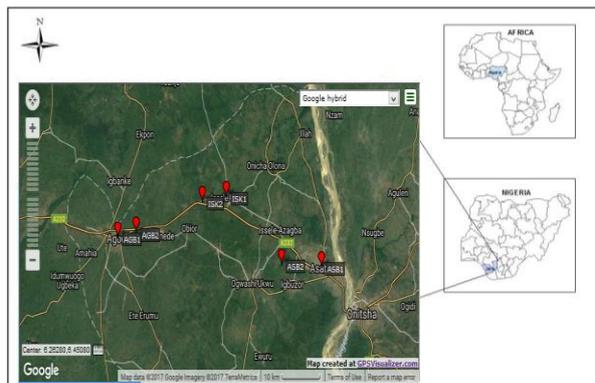
Fig. 1: Map of the study area

Collection of samples
After reconnaissance survey, soil samples were collected from six sites i.e. two sites from each of the three sites at depth 0-15 cm and 16-30 cm representing top and sub soils respectively between the months of August and October (wet season) and November and January (dry season). Soil samples were collected with an auger stainless steel after removal of the uppermost (debris) soil layer. Preservation of samples was carried out in stainless-steel holders and immediately transferred to the laboratory. Prior to analysis, samples were air dried in the dark, twigs and stones removed and sieved over stainless steel sieves (< 2 mm). Samples were stored in sealed stainless steel containers at 4ºC.

PAHs extraction and clean-up
According to US EPA-3550C-Ultrasonic extraction method, 10 g of the soil samples was mixed with 10 g of Na_2SO_4. The resulting mixture was extracted by ultrasonication with 50 mL of hexane/dichloromethane (1:1 v/v) at 30 °C for 30 min and filtered. The extract was evaporated to 1 mL using a rotary evaporator and subsequently purified by solid phase extraction with silica gel and alumina. PAHs were thereafter eluted using 15 mL hexane and dichloromethane (9:1). The eluted fraction was evaporated to 0.5 mL using nitrogen gas.

PAHs determination
Gas chromatograph (HP 6890 Palo Alto, C A, USA) with a flame ionization detector (FID) was used to quantify each PAH in the extracts as described by Tesi *et al.* (2016) and Iwegbue *et al.,* 2016. Separation was carried out using HP5 (cross-linked PHME siloxane) column with dimensions of 0.25 μm × 30 m and 0.25 μm film thickness. Helium was used as a carrier gas with a linear velocity of 30 cm/s. The initial column temperature was 100ºC and it was subsequently increased at 4⁰C/min to 310⁰C as final temperature. The injector temperature and injection volumes was 250⁰C and 2.0 μL in the split-less mode, respectively. The quantification was carried out by the use of external calibrations which was obtained with PAH solutions at five concentration levels.

Human health-risk assessment from PAHs exposure
The human health risks from exposure to PAHs contaminated soil was assessed using the B[a]P toxic equivalent factor [BaP$_{TEF}$], BaP mutagenic equivalent factor (BaP$_{MEF}$) and Incremental Life Cancer Risk models. These models have

been used to determine the risks of PAHs in soils and dust by several researchers (Yang *et al*., 2015; Tesi *et al.,* 2016; Iwegbue *et al.,* 2016; Larsen and Larsen, 1998; Durant, 1996; USEPA, 1993).

B[a]P carcinogenic equivalent [BaP$_{TEQ}$]
The B[a]P carcinogenic equivalent [BaP$_{TEQ}$] for each PAH was calculated using:

$$BaP_{TEQ} = \Sigma\ C_i \times BaP_{TEF} \qquad (1)$$

Where: BaP$_{TEF}$ is the cancer potency relative to B[a]P and C_i is the individual PAH concentration.

B[a]P mutagenic equivalent [BaP$_{MEQ}$]
The B[a]P mutagenic equivalent (BaP$_{MEQ}$) for the individual PAHs was evaluated using:

$$BaP_{MEQ} = \Sigma\ C_i \times BaP_{MEF} \qquad (2)$$

Where: BaP$_{MEF}$ = mutagenic potency relative to BaP and C_i = each PAH concentration.

Incremental lifetime cancer risk (ILCR) from PAHs
Evaluation of the ILCR in terms of dermal contact, inhalation and ingestion was evaluated using equations 3-5 (USEPA 1989; USEPA, 2009).

$$ILCR_{ing} = \frac{Csoil \times IngR \times EF \times ED \times CF \times SFO}{BW \times AT} \qquad (3)$$

$$ILCR_{inh} = \frac{Csoil \times EF \times ED \times IUR}{PEF \times AT*} \qquad (4)$$

$$ILCR_{derm} = \frac{Csoil \times SA \times AF \times ABS \times EF \times ED \times CF \times SFO \times GIABS}{BW \times AT} \qquad (5)$$

Where: ILCR$_{ing}$, ILCR$_{inh}$ and ILCR$_{derm}$ are the incremental lifetime cancer risk through ingestion, inhalation and dermal contact respectively; CF is conversion factor 1×10^{-6}.

The variables and toxicological parameters used in risk assessment are shown in Tables 1 and 2, respectively.

Quality control and assurance
Reagents are of chromatographic grade. Equipments and apparatus were properly cleaned to avoid cross-contamination of samples during sampling, preservation and preparation. Four unit-samples were added to form a unit. To evaluate the PAHs extraction efficiency, a known concentration of a standard PAH mixture was added to already analyzed sample and re-analyzed. Recoveries for the PAH compounds were between 80.8 and 94.2%. The relative standard deviations for replicate analyses (n = 3) were less than 5%.

Approach to data analysis
The statistical evaluations were performed using SPSS 19 version. Relationship between PAHs was established by means of Pearson correlation coefficient. Student's t-test was used to determine the significant variation between the concentrations and profiles of PAHs in depth and between seasons, with p-value less than 0.05 (p<0.05) considered to be statistically significant. One-way Analysis of variance (ANOVA) was applied to evaluate the significant variability of PAHs among various sites.
According to the New York State Department of Health (2007), incremental lifetime cancer value $\leq 10^{-6}$ indicate very low risk; 10^{-6} to 10^{-4} indicate low risk; $>10^{-4}$ to 10^{-3} indicate moderate risk; $>10^{-3}$ to 10^{-1} indicate high risk and $\geq 10^{-1}$ indicate very high risk. The value 10^{-6} is considered the carcinogenic target risk (USEPA, 2011).

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Table 1: Variables for estimation of human-health risk assessment

Parameters	Unit	Description	Values Infant/	Values Adult	Reference
Csoil	µg/kg	PAHs concentration in dust			
ABS	-	Dermal absorption factor for PAH compounds	0.13	0.13	USEPA, 2011
AF	mg/cm^2	Soil to skin adherences factor	0.2	0.07	USEPA, 2011
BW	Kg	Average body weight	15	60	Tesi et al. (2016)
ED	Year	Exposure duration	6	30	USEPA, 2001
EF	d/yr	Exposure frequency	350	350	USEPA, 2001
ET	h/d	Exposure time	24	24	USEPA, 1987
IngR	mg/d	Soil ingestion rate for receptor	200	100	USDOE, 2011
SA	cm^2/event	Skin surface area	2800	5700	USDOE, 2011
AT	D	Averaging time for non-carcinogenic	ED x 365		USDOE, 2011
AT*	d	Averaging time for carcinogenic	LT x 365		USDOE, 2011
LT	Year	Lifetime	54.4yrs (for Nigeria)		Iwegbue et al.(2016)
PEF	m^3/kg	Soil to air particulate emission factor	1.36 x 10^9		USDOE, 2011

Table 2: Toxicological parameters of PAHs used for human health risk assessment

Element/PAHs	SFO$_{ing}$ (mg/kg/d)	IUR (µg/m^3)	ABS$_{GI}$
BaA	7.3 x 10^{-1}	1.1 x 10^{-4}	1
Chry	7.3 x 10^{-3}	1.1 x 10^{-5}	1
BbF	7.3 x 10^{-1}	1.1 x 10^{-4}	1
BkF	7.3 x 10^{-2}	1.1 x 10^{-4}	1
BaP	7.3	1.1 x 10^{-3}	1
IndP	7.3 x 10^{-1}	1.1 x 10^{-4}	1
DahA	7.3	1.2 x 10^{-3}	1
Reference	**USDOE, 2011**	**USEPA, 2010**	**USEPA, 2011**

SFO = Oral Slope Factor; IUR = Inhalation Unit Risk; ABS$_{GI}$ = Gastrointestinal Absorption Factor

Results and Discussion

PAHs concentrations and distribution

As shown in Table 3, the concentrations of 16 PAHs obtained in Agbor ranged between 15.0 and 104.0 µg/kg and 49.1 and 176.0 µg/kg in wet and dry seasons respectively. Also, the concentrations of 16 PAHs obtained in Asaba ranged from 27.0 to 316.0 µg/kg and 4.0 to 44.0 µg/kg in wet and dry seasons respectively. Similarly the concentrations of 16 PAHs obtained in Issele-Uku ranged between 13.9 and 114.0 µg/kg and 14.2 and 75.0 µg/kg in wet and dry seasons respectively. The concentrations of Σ16 PAHs obtained ranged between 178.0 µg/kg and 787.0 µg/kg for wet season and 105.1 µg/kg and 437.7 µg/kg for dry season. The concentrations of PAHs were higher in the subsoil in the wet season than in the dry season while higher concentrations of Σ16 PAHs were observed in topsoil than subsoil during the dry season the concentrations varied significantly (p<0.05) among sites and soil profile. There was significant variation (p<0.05) in the concentrations between the dry and wet season as well as between the top and sub soil. The relatively high concentrations of PAHs recorded in the subsoil in wet season could be related to leaching (Emoyan *et al.*, 2015b). The concentration of PAHs followed the order Asaba > Issele-Uku > Agbor during the wet season and Issele uku > Agbor > Asaba during the dry season. The high concentrations of PAHs observed in Asaba and Isele-Uku in wet and dry seaseons, respectively could be related to high trffic density in these areas. The concentration values of Σ16 PAHs were below the target value of 1000 µg/kg stipulated by Department of Petroleum Resources (Department of Petroleum Resources, 2002).

Comparative concentrations evaluation of Σ16 PAHs obtained in this study were in agreement with others studies in Nigeria, USA, Canada, China, Spain, India, etc (Table 4).

Ring-wise PAHs distribution

The ring-wise concentrations and distribution patterns of PAHs in this study are in the order of 2-ring < 6- ring < 3-ring < 4-ring < 5-ring, Table 3. The concentration of the Σ2-ring PAH (Naphthalene) ranged from 30.5 to 59.0 µg/kg in Issele-Uku and Asaba respectively for wet season and Naphthalene concentration of 16.1 mg/kg was only detected in Asaba during the dry season. The low concentration of Nap observed in this study is due to ease of volatilization and evaporation of Nap as LPAHs, (CCME, 2008). The Σ3-ring PAH concentration ranged between 20.5 and 151.0 µg/kg in Issele-Uku for wet season and 55.0 and 146.0 µg/kg in Issele-Uku and Asaba, respectively for wet season. The 3-ring PAHs are the dominant PAH compounds at top soil in Isseke-Uku during the wet season and sub soil in Asaba during the dry season. Among the 3-ring PAH compounds, Acenapthylene is the dominant compound in terms of occurrence during the wet season while phenanthrene and anthracene were the dominant 3-ringed PAH compounds during the dry season.

The Σ4 ring PAHs are the second dominant PAH compounds in this study. The 4-ringed PAH compounds were detected at all locations, depth and seasons. The concentrations of the Σ4-ringed PAHs ranged between 59.0 and 215.0 µg/kg in Isele-Uku in wet season and 5.0 and 176.0 µg/kg in Asaba and Agbor respectively in dry season. Pyrene is the predominant 4-ring PAH compound in this study. Pyrene was detected in all samples except 0-15 cm depth at Agbor. Also, the concentrations of the Σ5-ring PAHs ranged from 38.0 to 618.0 µg/kg at Agbor and Asaba respectively in wet season and 38.0 to 189.0 µg/kg in Asaba and Agbor respectively in dry season. The Σ5-ring PAH was not detected at sub soil in Asaba. Benzo(a)pyrene is the most dominant 5-ringed PAH compound in this study, also B(a)p was detected in all samples except sub soil in Asaba. Conversely, Dibenzo(a,h)anthracene concentration was detected in all samples. The concentration of the Σ6-ring PAHs ranged between 35.0 and 81.7 µg/kg in Esele-Uku wet season and 24.0 and 110.0 µg/kg in Asaba and Isele-Uku in dry seasons. The Σ5-ring PAHs are the most dominant. The observed concentrations of HPAHs over LPAHs in the study may be due to presence of high density of diesel powered vehicles and biomass combustion which are the sources of PAHs (Khalili, *et al.,* 1995 and Yunker *et al.,* 2002). The distribution of LPAHs and HPAHs shows that the study area soil-TOC enhances HPAHs adsorption on fine-active surfaces of the soil that limit hydrolysis, oxidation, photolysis, biodegradation, volatilization, leaching and mobilization of HPAHs (Valentin *et al.,* 2013; Petruzzelli *et al*., 2013).

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Table 3: PAHs concentrations (µg/kg) in soil samples

PAH	Wet Season						Dry Season					
	Agbor (cm)		Issele-Uku (cm)		Asaba (cm)		Agbor (cm)		Issele-Uku (cm)		Asaba (cm)	
	0-15	16-30	0-15	16-30	0-15	16-30	0-15	16-30	0-15	16-30	0-15	16-30
Nap	BDL	BDL	30.5	57.0	BDL	59.0	BDL	BDL	BDL	BDL	BDL	16.1
Acy	BDL	29.0	30.0	20.0	29.0	BDL	BDL	BDL	BDL	14.2	BDL	32.0
Ace	BDL	33.0	54.0	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	39.0
Flu	BDL	26.0	15.5	BDL	BDL	BDL	BDL	BDL	BDL	47.0	44.0	4.0
Phe	BDL	BDL	35.0	BDL	84.0	BDL	BDL	BDL	15.0	15.7	16.0	34.0
Ant	BDL	BDL	17.0	BDL	BDL	BDL	BDL	BDL	40.0	47.0	23.0	37.0
Flt	BDL	31.3	24.0	40.0	43.0	43.0	176.0	BDL	49.0	36.0	18.0	BDL
Pyr	59.0	58.7	22.0	16.0	83.0	67.0	BDL	56.0	57.0	42.0	17.0	5.0
BaA	BDL	99.0	16.1	45.0	34.0	BDL	BDL	BDL	17.0	15.1	17.0	BDL
Chry	BDL	25.0	19.1	114.0	27.0	BDL	BDL	BDL	21.0	24.0	17.0	BDL
BbF	15.0	22.0	13.9	44.0	27.0	316.0	55.0	BDL	21.0	75.0	18.0	BDL
BkF	BDL	BDL	BDL	BDL	198.0	BDL	BDL	BDL	BDL	BDL	BDL	BDL
BaP	104.0	16.0	82.9	15.0	41.0	302.0	134.0	49.1	44.0	29.4	20.0	BDL
IBDLP	BDL	23.0	64.7	18.0	37.0	BDL	BDL	BDL	70.0	19.3	BDL	BDL
DahA	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
BghiP	BDL	20.0	17.0	17.0	BDL	BDL	BDL	BDL	40.0	73.0	24.0	BDL
TOTAL	178.0	383.0	441.7	386.0	603.0	787.0	365.0	105.1	374.0	437.7	214.0	167.1
2 RINGS	0.0	0.0	30.5	57.0	0.0	59.0	0.0	0.0	0.0	0.0	0.0	16.1
3RINGS	0.0	88.0	151.5	20.0	112.0	0.0	0.0	0.0	55.0	123.9	83.0	146.0
4RINGS	59.0	214.0	59.2	215.0	187.0	110.0	176.0	56.0	144.0	117.1	69.0	5.0
5RINGS	119.0	38.0	96.8	59.0	226.0	618.0	189.0	49.1	65.0	104.4	38.0	0.0
6RINGS	0.0	43.0	81.7	35.0	37.0	0.0	0.0	0.0	110.0	92.3	24.0	0.0

BDL= Below Detection Limit

Table 4: Comparison of PAH in soil in this study with others in literature (Adapted in part from Tesi *et al.*, 2016)

Location	Studied Soil	Distribution Range (µgkg⁻¹)	Reference
Nigeria	**Urban soils**	**105.1-787**	**This study**
America (Miami florida)	Urban soils	251-2364	Banger *et al.* (2010)
Canada	Flood plain	0.016-12.0	Sartori *et al.* (2010)
China (Beijing)	Rural and suburban soils	20-3900	Mai *et al.* (2005)
China (Beijing)	Urban soil	467-5470	Li *et al.* (2006)
China (Guangzhou)	Vegetable soils	42-3077	Chen *et al.* (2005)
China (Hong Kong)	Rural and urban soils	30-170	Zhang *et al.* (2006)
China (Huanghuai plain)	Agricultural soils	15.7-1247.6	Yang *et al.* (2012)
Estonia	Rural, urban and Industrial soils	50-22,200	Trapido (1999)
Germany (Mosel & Saar River)	Floodplain	100-81500	Pies *et al.* (2007)
Germany (Rhine river)	Alluvial soils	20-3600	Gocht *et al.* (2001)
India (Kuruksheta)	Urban Roadside soils	16.1- 2538.0	Kumar *et al.*(2012)
Korea (An-san city)	Industrial soils	109.93-178.92	Imran *et al.* (2006)
Nigeria (Niger Delta)	Soil vicinity oil installation	24-120	Sojinu *et al.* (2010)
Nigeria (Niger Delta)	Urban soils	182-433	Olajire *et al.* (2005)
Nigeria(Lagos)	Mangrove fresh soil	65.5 188.0	Sojinu *et al.*(2012)
Spain (Sevilla)	Agriculture and urban soils	89.5-4004.2	Morello *et al.* (2008)
Switzerland	Pasture grassland and urban soils	50-600	Bucheli *et al.* (2004)
United kingdom	Urban soil	2700 ± 500	Meharg *et al.* (1998)

Table 5: Bap-toxic equivalence (µg/kg)

PAH	Wet Season						Dry Season					
	Agbor (cm)		Issele-Uku (cm)		Asaba (cm)		Agbor (cm)		Issele-Uku (cm)		Asaba (cm)	
	0-15	16-30	0-15	16-30	0-15	16-30	0-15	16-30	0-15	16-30	0-15	16-30
BaA	0.00	9.90	1.61	4.50	3.40	0.00	0.00	0.00	1.70	1.51	1.70	0.00
Chry	0.00	0.03	0.02	0.11	0.03	0.00	0.00	0.00	0.02	0.02	0.02	0.00
BbF	1.50	2.20	1.39	4.40	2.70	31.60	5.50	0.00	2.10	7.50	1.80	0.00
BKF	0.00	0.00	0.00	0.00	1.98	0.00	0.00	0.00	0.00	0.00	0.00	0.00
BaP	104.0	16.00	82.90	15.00	41.00	302.00	134.00	49.10	44.00	29.40	20.00	0.00
IndP	0.00	2.30	6.47	1.80	3.70	0.00	0.00	0.00	7.00	1.93	0.00	0.00
DahA	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
BaPTEQ	105.50	30.43	90.39	25.81	52.81	333.60	139.50	49.10	54.80	40.36	23.52	0.00

Table 6: Bap mutagenic equivalence (µg/kg)

PAH	Wet Season						Dry Season					
	Agbor (cm)		Issele-Uku (cm)		Asaba (cm)		Agbor (cm)		Issele-Uku (cm)		Asaba (cm)	
	0-15	16-30	0-15	16-30	0-15	16-30	0-15	16-30	0-15	16-30	0-15	16-30
B(a)A	0.00	8.11	1.32	3.69	2.79	0.00	0.00	0.00	1.39	1.23	1.39	0.00
Chry	0.00	0.43	0.33	0.00	0.46	0.00	0.00	0.00	0.36	0.41	0.29	0.00
B(b)F	3.75	5.50	3.48	11.00	6.75	79.00	13.85	0.00	5.25	18.75	4.50	0.00
B(k)F	0.00	0.00	0.00	0.00	21.78	0.00	0.00	0.00	0.00	0.00	0.00	0.00
B(a)P	104.00	16.00	82.90	15.00	41.00	302.00	134.00	49.11	44.00	29.40	20.00	0.00
I(nd)P	0.00	7.13	20.05	5.58	11.47	0.00	0.00	0.00	21.17	5.98	0.00	0.00
D(ah)A	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
BaPMEQ	107.75	37.14	108.08	35.27	84.25	381.00	147.85	49.11	72.17	55.77	26.18	0.00

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Human health risks assessment from PAHs exposure
Bap-toxic equivalence and bap mutagenic equivalence of PAHs

The Bap toxic equivalence (BaPTEQ) and BaP mutagenic equivalence (BaPMEQ) obtained in this study are shown in Tables 5 and 6, respectively. The Bap toxic equivalence ranged between 25.81 and 333.60 µg/kg at Isele-Uku and Asaba respectively in wet season, and 23.52 and 139.50 µg/kg in Asaba and Agbor in dry season. There was significant contribution from BaP, BbF and BaA to the BapTEQ in this study. The observed values of BaP mutagenic equivalence in this study ranged between 35.27 and 381.00 µg/kg in Isele-Uku and Asaba respectively in wet season and 26.18 and 147.85 µg/kg in Asaba and Agbor respectively in dry season. Similarly, there was significant contribution from Bap, BbK and BaA to the BapMEQ values. The observed Bap toxic equivalence shows that there high human risk to PAHs exposure in Agbor and Isele-Uku (top soil) and Asaba (sub soil). However, BaP mutagenic equivalence in this study shows that there high human risk to PAHs exposure in Agbor (top soil). The observed trend values could be related to the predominance of 5-ring PAHs in this study. The Bap toxic equivalence and BaP mutagenic equivalence obtained in this study were similar to values reported in other studies, (Olawoyin *et al.,* 2012; Kumar *et. al.,* 2014; Tesi *et al.,* 2016; Iwegbue *et al.,* 2016).

Incremental lifetime cancer risk for human (infant and adult)

The computed values of Incremental Lifetime Cancer Risk for infants and adults are shown in Table 7.

The obtained Incremental Lifetime Cancer Risk values through soil inhalation, ingestion, and dermal contact ranged from 1.84×10^{-11} to 2.59×10^{-10}, 2.19×10^{-3} to 3.11×10^{-2}, and 7.99×10^{-4} to 1.13×10^{-2} for the infant and 1.51×10^{-4} to 2.15×10^{-3}, 1.01×10^{-11} to 1.43×10^{-10} and 7.85×10^{-5} to 1.11×10^{-11} for adult. The observed ILCR values through the three routes of exposure is in the order of Ingestion > Dermal contact > inhalation.

The obtained total cancer risk values in all sites, depth and seasons ranged from $2.99 \times 10{-3}$ to 4.25×10^{-2} with a mean of 1.10×10^{-2} for the infant and 2.30×10^{-4} to 3.26×10^{-3} with a mean of 8.42×10^{-4} for adult. The total cancer risk for children was greater than that of adult. This may be as a result of high frequency of physical contact of infants with soil and dust (Olawoyin *et al.,* 2012; Tesi *et al.,* 2016; Iwegbue *et al.,* 2016). The obtained total cancer risk values were in agreement with those reported in contaminated soils from Hong Kong (Man *et al.,* 2013), metropolitan soils from China (Peng *et al.,* 2011), urban soils from Nigeria, (Iwegbue *et al.,* 2016) and flood plain soils from Nigeria (Tesi *et al.,* 2016).

The lifetime cancer risk values of $\geq 10^{-1}$ = very high, $> 10^{-3}$ to 10^{-1} = high, $\geq 10^{-4}$ to 10^{-3} = moderate, 10^{-6} to 10^{-4} = low and $\leq 10^{-6}$ = very low was classified by New York States Department of Health (2007). The obtained total cancer risk values in this study falls between moderate and high category. Similarly, the risk based action level set by USEPA (2010) for exposure scenarios are based on a potential acceptable value of 1×10^{-6} as target excess risk. The result obtained in this study for total cancer risk for infants and adult are greater than the acceptable value of 1×10^{-6}, indicating that the soil samples in this study has high potential human carcinogenic risk.

Table 7: Incremental Lifetime Cancer Risk for Human (Infant and Adult)

Season	Locations	Depth	INFANT ILCRIng	ILCRInh	ILCRDerm	Total Cancer Risk	ADULT ILCRIng	ILCRInh	ILCRDerm	Total Cancer Risk
WET	Agbor	0-15	9.85E-03	8.18E-11	3.58E-03	1.34E-02	6.79E-04	4.51E-11	3.52E-04	1.03E-03
		16-30	2.84E-03	2.38E-11	1.03E-03	3.87E-03	1.96E-04	1.31E-11	1.02E-04	2.97E-04
	Issele-Uku	0-15	8.62E-03	7.18E-11	3.14E-03	1.18E-02	5.94E-04	3.96E-11	3.08E-04	9.03E-04
		16-30	2.41E-03	2.08E-11	8.77E-04	3.29E-03	1.66E-04	1.15E-11	8.61E-05	2.52E-04
	Asaba	0-15	4.93E-03	5.50E-11	1.79E-03	6.72E-03	3.40E-04	3.03E-11	1.76E-04	5.16E-04
		16-30	3.11E-02	2.59E-10	1.13E-02	4.25E-02	2.15E-03	1.43E-10	1.11E-03	3.26E-03
DRY	Agbor	0-15	1.30E-02	1.08E-10	4.74E-03	1.78E-02	8.98E-04	5.97E-11	4.66E-04	1.36E-03
		16-30	4.58E-03	3.81E-11	1.67E-03	6.25E-03	3.16E-04	2.10E-11	1.64E-04	4.80E-04
	Issele-Uku	0-15	5.12E-03	4.27E-11	1.86E-03	6.98E-03	3.53E-04	2.35E-11	1.83E-04	5.36E-04
		16-30	3.77E-03	3.15E-11	1.37E-03	5.14E-03	2.60E-04	1.74E-11	1.35E-04	3.94E-04
	Asaba	0-15	2.19E-03	1.84E-11	7.99E-04	2.99E-03	1.51E-04	1.01E-11	7.85E-05	2.30E-04
		16-30	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00
		MIN	2.19E-03	1.84E-11	7.99E-04	2.99E-03	1.51E-04	1.01E-11	7.85E-05	2.30E-04
		MAX	3.11E-02	2.59E-10	1.13E-02	4.25E-02	2.15E-03	1.43E-10	1.11E-03	3.26E-03
		MEAN	7.37E-03	6.26E-11	2.68E-03	1.01E-02	5.08E-04	3.45E-11	2.64E-04	7.72E-04
		MEAN	8.04E-03	6.82E-11	2.93E-03	1.10E-02	5.54E-04	3.76E-11	2.88E-04	8.42E-04

Table 8: PAHs source ratios of

Ratio	Wet Season Agbor 0-15	16-30	Issele-Uku 0-15	16-30	Asaba 0-15	16-30	Dry Season Agbor 0-15	16-30	Issele-Uku 0-15	16-30	Asaba 0-15	16-30
Ant/(Ant +Phen)	0.00	0.00	0.32	0.00	0.00	0.00	0.00	0.00	0.73	0.75	0.59	0.52
BaA/(BaP+Chry)	0.00	0.80	0.46	0.38	0.00	0.00	0.00	0.00	0.45	0.39	0.50	0.00
Flt/(Flt+Pyr)	0.00	0.35	0.52	0.71	1.00	0.39	1.00	0.00	0.46	0.46	0.52	0.00
IndP/(IndP+ BghiP)	0.00	0.53	0.79	0.51	0.00	0.00	0.00	0.00	0.62	0.21	0.00	0.00
LMW/HMW	**0.00**	**0.32**	**0.70**	**0.25**	**0.23**	**0.08**	**0.00**	**0.00**	**0.17**	**0.40**	**0.63**	**32.40**

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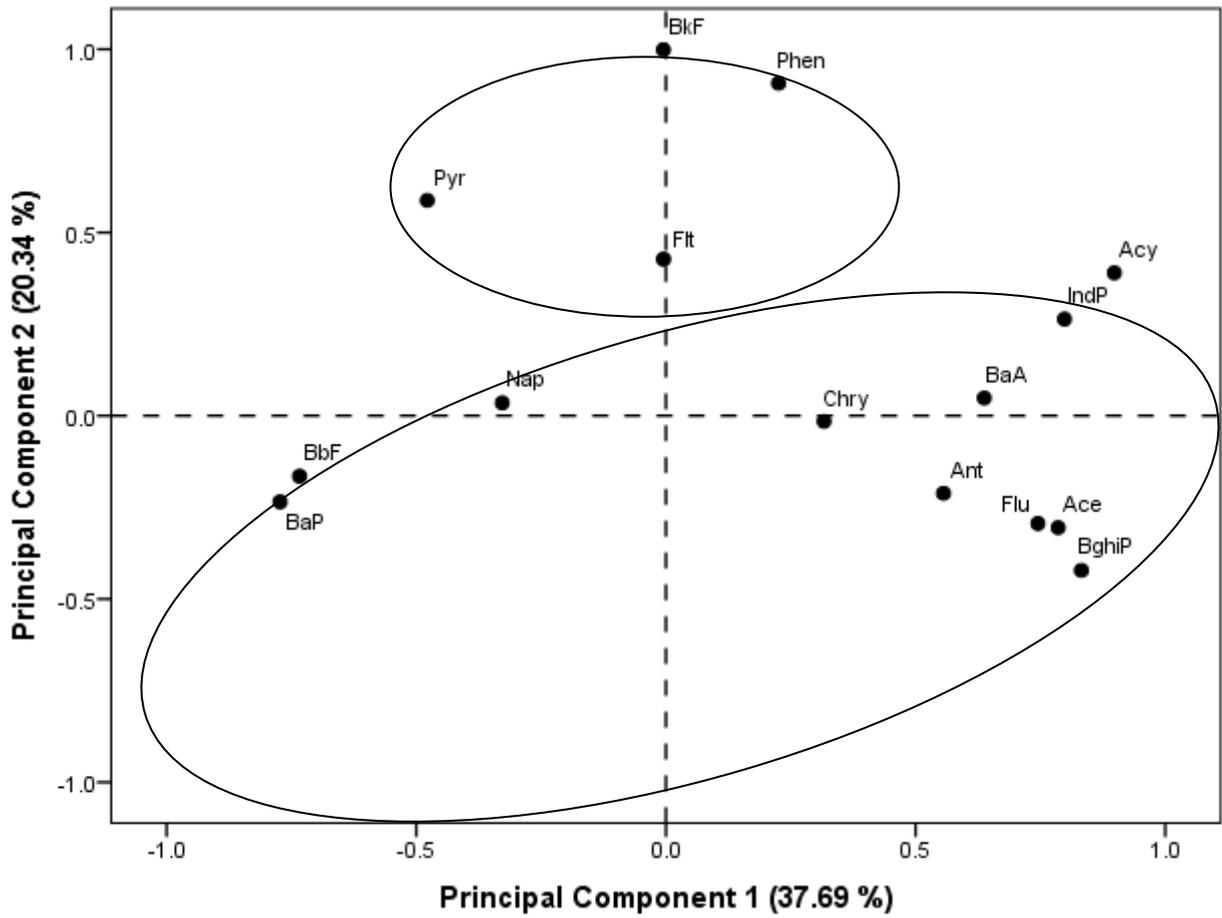
Fig. 2: Plot of PCA in wet season
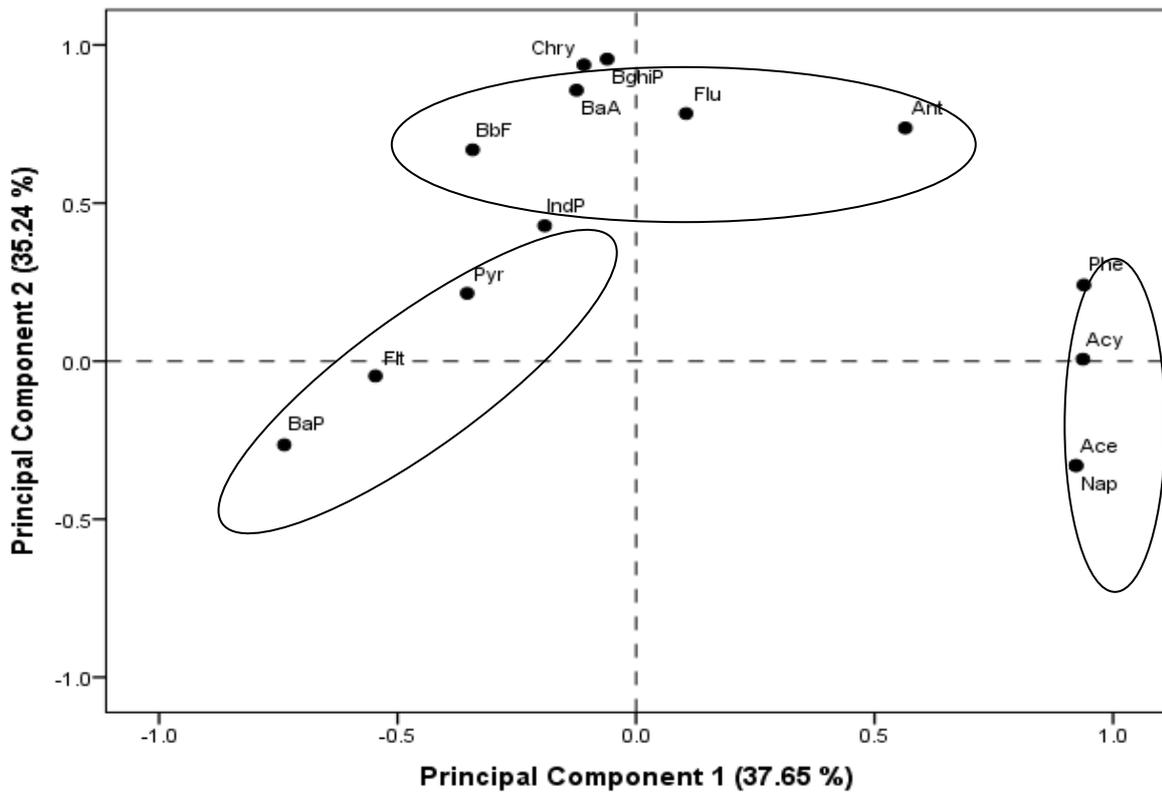
Fig. 3: Plot of PCA in dry season

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Source apportionment

The probable sources of environmental PAHs could be evaluated using ratio of different PAHs compounds (Yunker *et al.,* 2002). As shown in Table 8 in this study, the ratio of Ant/(Ant + Phen) > 0.1 indicates combustion processes as sources of PAHs. A ratio of Flt/(Flt + Pyr) < 0.4, 0.4 – 0.5 and > 0.5 is suggestive of petroleum input, petroleum oxidation and combustion of biomass and coal. The obtained ratio of Flt/(Flt + Pyr) in this study ranged between 0.4 and 1.0 indicating petroleum, coal and biomass combustion as sources of PAHs.

BaA/(BaA + Chry) ratio of < 0.2 suggest petroleum origin, 0.2 – 0.35 indicate petroleum combustion and > 0.35 indicates combustion of coal and biomass. In this study, the values of BaA/BaA + Chry) ratio are > 0.35 indicating combustion of coal and biomass. IndP/(IndP + BghiP) ratio < 0.2 indicates petroleum input, 0.2 – 0.5 indicates petroleum combustion and > 0.5 indicates combustion of coal and biomass. In this study, the obtained ratio of IndP/(IndP + BghiP) ranged between 0.21 and 0.79 indicating that the PAHs are from petroleum, coal and biomass combustion. LMW/HMW ratio > 1 suggests petroleum sources while < 1 indicates pyrogenic sources (combustion from petroleum, coal and biomass). In this study, sub soil in Asaba has LMW/HMW ratio > 1, all the others were < 1 indicating petroleum and coal combustion sources.

Principal component analysis

Yunker *et al*., 2002; Larsen and Baker, 2003 had used principal component analysis (as a tool to classify probable pollution sources of PAHs and results of PCA in this study are shown in Table 9 and Figs. 2 and 3.

Table 9: PCA factor components with Varimax with Kaizer normalization rotation in wet and dry seasons

PAH Compounds	Wet Season		Dry Season		
	Component		Component		
	F. 1	F. 2	F. 1	F. 2	F. 3
Nap	-.328		.922	-.330	
Acy	.898	.390	.937		
Ace	.785	-.305	.922	-.330	
Flu	.745			.783	
Phen		.907	.939		
Ant	.556		.564	.738	
Flt		.427	-.546		-.716
Pyr	-.478	.588	-.354		.821
BaA	.638			.857	.360
Chry	.317			.937	.311
BbF	-.734		-.343	.668	-.577
BkF		.998	-.737		-.551
BaP	-.773				.539
IndP	.798			.428	
BghiP	.832	-.422		.955	
% Variance	37.69	20.34	37.65	35.24	13.92

F = Factor

To further define the possible sources of PAHs in the study area, principal component analysis was applied. In the wet season, two factors were identified which accounts for 58.03% of the variability. Factor 1 account for 37.69% of the total variance is characterized with high loading of Ace, Acy, Flu, IndP and BghiP and moderate loadings of Ant and BaA. Acy, Ace and Flu are combustion products of pyrogenic processes such as wood and coke combustion. IndP and BghiP are indicators of traffic emissions (Khalili *et al.,* 1995; Fraser *et al.,* 1997; Simcik *et al.,* 1999; Yang *et al.,* 2012). Ant is a product of wood and coke combustion, (Yunker *et al*., 2002) while BaA is a marker for diesel oxidation. Thus, factor 1 indicates that combustion of coke, wood, diesel and traffic emissions are the primary sources of PAHs. Factor 2 accounts for 20.34% of the total variance and is characterized by high

components of Phen and BkF and moderate components of Pyr. Phen which are by-products of wood combustion, BkF is a marker for coal and gasoline combustion (Wan *et al*., 2006; Dong and Lee, 2009). Pyr is a marker for coal combustion, (Larsen and Baker, 2003). Therefore, factor 2 suggests that the sources of PAHs are attributed to wood and coal combustion.

Three factors were identified for dry season which accounted for 86.81% of the variability. Factor 1 is characterized by high values of Nap, Acy, Ace and Phe and moderate loading of Ant which constitute 37.65% of the total variance. Factor 1 is Nap, suggest incomplete combustion of wood related sources, (Dong and Lee, 2009). Acy, Ace, and Phen are products of combustion wood (Jenkins, *et. al.,* 1996) while Ant is a by-product of wood and coke combustion (Yunker *et al*., 2002), factor 1 is suggests that wood combustion is the source of PAHs. Factor 2 accounted for 35.24% of the total variance and is characterized by high components of Flu, Ant, BaA, Chry and BghiP with moderate components of BbF, Flu is a product of low temperature pyrogenic processes such as wood combustion, while Ant is a product of coke and wood oxidation (Yunker *et al.,* 2002; Yang *et al.,* 2012). BaA is a marker for diesel oxidation while Chry is a marker for diesel and natural gas oxidation; BghiP is from traffic emissions (Khalili *et al.,* 1995; Simcik *et al.,* 1999), while BbF is a product of fossil fuel combustions (Kavouras *et al.,* 2001; Yang *et al.,* 2012). Thus, from Principal Component Analysis for both wet and dry season, the sources of PAHs in this study are from emission of wood coke, and biomass combustion, and vehicular emission from diesel, fossil fuels engine combustion.

Conclusion

Sixteen PAHs grouped as endocrine disruption substances and priority pollutants were determine for their concentrations and evaluation of sources and human health risk. This study has established that the study area is contaminated with PAHs at different concentrations in top and sub soils as well as wet and dry seasons with 5-ring PAHs being the most predominant. The study also shows that Asaba and Isele-Uku recorded high concentrations of PAHs in wet and dry seasons respectively. Ratio of PAHs and principal component analysis shows that the sources of PAHs are petroleum, biomass and coal, combustion and traffic emissions. The Bap-Toxic Equivalence and Bap Mutagenic Equivalence of PAHs, and Incremental Lifetime Cancer Risk for Human (Infant and Adult) further established that PAHs exposure suggests a potential for high human carcinogenic risk in the study area. Therefore, prolong human exposure to PAHs in the study area may induce some health challenges associated with PAHs exposure.

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