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Occurrence and Source Apportionment of Polycyclic Aromatic Hydrocarbons (PAHs) in Urban Soil of Udu, Near Warri, Delta State

Anthony A. Ogbuta[1], Omotekoro S. Emakunu[2], Juliet N. Tesi[3*] and Bulouebibo Lari[4]

[1]Department of Chemical Sciences, University of Africa, Toru-Orua, Nigeria
[2]Department of Chemistry, College of Education, Mosogar, Nigeria
[3]Department of Environmental Management and Toxicology, Federal University of Petroleum Resources, Effurun, Nigeria
[4]Department of Science Laboratory Technology, Delta State University Abraka, Nigeria

*Corresponding author; Email: juliejane.cj@gmail.com, Tel: +234(0)8036380582

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ABSTRACT: The occurrence and sources of polycyclic aromatic hydrocarbons (PAHs) were assessed in urban soils of Udu, near Warri in Delta State, Nigeria. The PAHs were quantified with gas chromatography equipped with flame ionization detector (GC-FID) after extraction and clean-up of the extracts with n-hexane/dichloromethane. The concentrations of ∑16 PAHs in the urban soils ranged from 269 µg kg^{-1} to 853 µg kg^{-1}. On the average, the occurrence of the individual PAH compounds followed the order: DahA > BghiP > IndP > BbF > BaP > Phen > Ace > BkF > Ant > Flu > Nap > Chry > Flt = Pyr > Acy > BaA while the occurrence pattern of the PAHs with respect to ring size was in the order of 5 rings > 6 rings > 3 rings > 4 rings > 2 rings. The higher molecular weight (4-6 rings) PAHs were the predominant PAHs in these urban soils and accounted for 76% of the total PAHs concentrations. The isomeric ratios used for source apportionment indicate that the PAHs in these urban soils originated majorly from high temperature combustion of petroleum, coal, fossil fuels and biomass such as woods.

Keywords: PAHs, Urban soil, Sources, Udu, Warri

Introduction

Polycyclic aromatic hydrocarbons (PAHs) are a large class of over two hundred persistent semi-volatile organic compounds characterized by high metamorphism and toxicity (Filho *et al*., 2020; Sun *et al*., 2019). Some PAHs have been found to be mutagenic, genetoxic, carcinogenic and have the ability to be transported over long distance. They tend to be associated with particles and are widely transported by atmospheric pathway, resulting in elevated concentrations in soils (Yang *et al.,* 2006). PAHs in the environment come from either natural sources or anthropogenic sources. The former include volcanic eruption and forest fire while the latter involve incomplete complete combustion of petroleum, fossil fuels and other numerous industrial processes is by far the greatest contributor to the global PAHs load (Yang *et al.,* 2012). As a result of their health effects and widespread distribution in the environment, PAHs have gained a lot of attention and sixteen of them has been listed as priority pollutants in the United States Federal Water Pollution Control Act (1972) and the United States Clean Water Act (1997) (Bixian *et al*., 2001). Urban towns are the geographic locations of resource allocations, utilization and chemical release because they are the centre of economic and industrial activities and host to more than 50% of the global population (Emoyan *et al*., 2018; Iwegbue *et al*., 2016). Soils is usually the ultimate repository of PAHs and other pollutants in the environment and are readily absorbed

by organic matter in soils hence difficult to degrade (Benhaddya and Hadjel, 2014). Elevated PAHs concentrations have been reported in urban soils (Morillo *et al*., 2007; Heywood *et al*., 2006). The possibility of human exposure to PAHs through dermal contact, ingestion and inhalation (DII) may increase due to the closeness of soils to humans in urban towns. In addition, bioaccumulation, bioconcentration and biomagnifications of PAHs in the food chain may occur which could increase the potential for human health risk (Hu *et al*., 2017; Yang *et al.,* 2014). Thus it becomes necessary to determine the concentrations of contaminants such as PAHs in urban environments. Some studies have been carried out to assessed the concentrations of PAHs in urban soils in Nigeria (Ehigbor *et al*., 2020; Emoyan *et al*., 2018; Iwegbue *et al*., 2016; Sojinu *et al*., 2010; Okoro and Ikolo, 2007; Olajire *et al*., 2005) but none has been undertaken in the study area. Therefore, the objectives of this study are to determine the concentrations and sources of PAHs in urban soils from Udu, Near Warri.

Materials and Methods

Description of study area: Udu is a suburb of Warri linked from Enerhen by the Udu Bridge over the Warri River. It lies within Longitude 5.45 N to 5.75 N and latitude 5.43 E to 5.72 E. The Warri Airport is just approximately five minutes drive away from the study area. The area is host to some major industries including the Delta Steel Company. The area has an estimated population of about a hundred people. The area is characterized by tropical equatorial climate with moderate wet deposition and humidity, mean annual temperature of 32.8 °C and annual rainfall amount of 2673.8 mm (Tesi *et al*., 2020). The geographical coordinates of the sampling locations in the study area are shown Table 1.

Table 1: Geographical coordinates of the sampling locations in the study area

Sampling Locations	Latitude	Longitude
SU1	5.50592 N	5.80920 E
SU2	5.50676 N	5.80642 E
SU3	5.50655 N	5.80285 E
SU4	5.50829 N	5.80106 E
SU5	5.50994 N	5.80009 E
SU6	5.51181 N	5.80097 E
SU7	5.50456 N	5.80426 E
SU8	5.49180 N	5.80151 E
SU9	5.51073 N	5.84405 E
SU10	5.49716 N	5.82046 E

Sample collection: Top soil samples were collected from ten (10) locations around the study area at depth 0-20 cm. Soil samples were collected with a shovel after removing the debris. Samples were kept in aluminium foil, bagged in black polythene, stored and immediately transferred to the laboratory. In the laboratory, the soil samples were air dried in the dark, sieved over stainless steel sieves (< 2 mm). Thereafter, they were stored at 4^{o}C prior to analysis.

Sample extraction and chemical analysis: 10 g of the soil sample was weighed into a beaker. 50 ml of acetone and DCM in a ratio of 1:1 was added to the sample and sonicated at 70 ^{o}C for 30 minutes. After sonication, 10 g of anhydrous sodium sulphate was added and the solution concentrated using a rotary evaporator. The concentrated solution was cleaned-up by transferring it into a glass column packed with alumina and activated silica gel and eluted with 20 ml of dichloromethane (DCM). The extract was collected and concentrated to 2ml using a rotary evaporator. The concentrated extract was transferred into a vial bottle using a pipette, labelled appropriate and store at -4^{o}C before analysis. All reagents used were of standard analytical grade.

Chemical analysis: Samples were analysed for 16 PAHs by high resolution gas chromatography (Hewlett Packard 6890) equipped with a HP5 (Cross linked PHME Siloxane) (0.025 µm film thickness × 0.25 µm × 30 m) and Flame ionization detector (FID). A 0.5 mL aliquot of the extract was manually injected with a syringe. The injector temperature was 250^{o}C. The temperature program was initially set at 60 ^{o}C, held at 60 ^{o}C for 1min followed by a 20 ^{o}C/min ramping at 300 ^{o}C and then held there for 10min. Helium was used as a carrier gas at a flow rate of 1.36mL/min.

Quality control and statistical analysis: Acetone and DCM were picograde quality. To evaluate the extraction efficiency for the PAH compounds, recovery study was carried out by spiking selected already analysed samples with known concentration of the individual PAHs compound. The percent recovery for the PAH compounds ranged from 90.4 to 96.1%. Analysis of variance (ANOVA) was used to determine whether the concentrations of the PAHs varied significantly at 95% confidence limit using SPSS version 11.5.

Results

The results of the concentrations of PAHs in the urban soils of Udu investigated in this study are shown in Table 2.

Table 2: Concentrations (µg kg^{-1}) of PAHs in the urban soils

PAHs	**SU1**	**SU2**	**SU3**	**SU4**	**SU5**	**SU6**	**SU7**	**SU8**	**SU9**	**SU10**
Nap	7.0	2.0	90.0	17.0	11.0	28.0	4.0	11.0	3.0	4.0
Acy	9.0	15.0	7.0	14.0	7.0	55.0	8.0	16.0	2.0	2.0
Ace	13.0	7.0	5.0	29.0	6.0	26.0	1.0	15.0	154	4.0
Flu	66.0	34.0	3.0	15.0	3.0	14.0	6.0	57.0	10.0	2.0
Ant	54.0	40.0	19.0	17.0	35.0	5.0	29.0	3.0	11.0	6.0
Phen	1.0	ND	8.0	8.0	13.0	5.0	2.0	181	2.0	188
Flt	20.0	48.0	4.0	5.0	1.0	7.0	13.0	17.0	38.0	1.0
Pyr	1.0	12.0	4.0	4.0	4.0	2.0	4.0	22.0	101	ND
BaA	ND	11.0	2.0	1.0	9.0	5.0	5.0	57.0	9.0	6.0
Chry	3.0	14.0	26.0	9.0	2.0	70.0	3.0	39.0	ND	6.0
BbF	ND	12.0	22.0	10.0	15.0	5.0	202	69.0	187	15.0
BkF	21.0	5.0	9.0	5.0	112	42.0	15.0	8.0	3.0	5.0
BaP	21.0	9.0	14.0	152	180	45.0	14.0	6.0	14.0	32.0
DahA	34.0	ND	23.0	66.0	265	102	284	105	112	91.0
IndP	11.0	60.0	90.0	114	126	21.0	25.0	44.0	106	30.0
BghiP	135	ND	49.0	30.0	70.0	77.0	227	112	88.0	176
∑16PAHs	**396**	**269**	**375**	**496**	**859**	**509**	**842**	**762**	**831**	**568**
2 Rings	7.0	2.0	90.0	17.0	11.0	28.0	4.0	11.0	3.0	4.0
3 Rings	143	96.0	42.0	83.0	64.0	105	46.0	272	179	202
4 Rings	24.0	85.0	36.0	19.0	16.0	84.0	25.0	135	148	13.0
5 Rings	76.0	26.0	68.0	233	572	194	515	188	316	143
6 Rings	146	60.0	139	144	196	98.0	252	156	194	206
$\sum PAH_{7C}$	90.0	111	186	357	709	290	548	328	431	185
$\sum PAH_{NC}$	306	158	189	139	150	219	294	434	409	383
LMW-PAHs	150	98.0	132	100	75.0	133	50.0	283	182	206
HMW-PAHs	246	171	243	396	784	376	792	479	658	362

ND = not detected; Naphthalene (NaP), acenaphthalyne (Acy), acenaphthene (Acy), fluorene (Flu), phenanthrene (Phen), anthracene (Ant), fluoranthene (Flt), pyrene (Pyr), benzo(a)anthracene (BaA), chrysene (Chry), benzo(b)fluoranthene (BbF), benzo(k)fluoranthene (BkF), benzo(a)pyrene (BaP), indeno(1,2,3-cd)pyrene (IndP), dibenz(a,h)anthracene (DahA) and benzo(g,h,i)pyrene (BghiP), LMW = lower molecular weight; HMW = higher molecular weight

As shown in Table 2, the concentrations of the Σ16 PAHs ranged from 269 µg kg^{-1} to 853 µg kg^{-1}. The maximum and minimum concentrations of Σ16 PAHs were observed at SU5 and SU2 respectively. BaA and BbF were not detected at SU1 while Phen, DahA and BghiP were not detected at SU2. Chry and Pyr were not detected at SU9 and SU10 respectively. Among the rings, the concentration of the 2-rings PAH compound (Naphthalene) ranged from 2.0 to 90 .0 µg kg^{-1} at SU2 and SU3 respectively. The concentrations of the 3-rings PAHs (sum of Acy, Ace, Flu, Ant and Phen) ranged from 42 µg kg^{-1} at SU3 to 272 µg kg^{-1} at SU8 while the concentrations of the 4-rings PAHs (sum of Flt, Pyr, BaA and Chry) ranged from 13.0 µg kg^{-1} at SU10 to 148 µg kg^{-1} at SU9. Whereas, the concentrations of the 5-rings (sum of BbF, BkF, BaP and DahA) ranged from 26 to 572 µg kg^{-1} while the concentrations of the 6-rings PAHs (sum of IndP and BghiP) ranged from 60 to 252 µg kg^{-1}. The least concentration of 5- and 6-rings PAHs was recorded in location SU2. The maximum concentrations of 5- and 6-rings PAHs were found in locatios SU5 and SU7 respectively. The concentrations of the carcinogenic PAHs ($\sum PAH_{7C}$) ranged from 90 to 709 µg kg^{-1} (with a mean of 324 µg kg^{-1}) while the non-carcinogenic PAHs ($\sum PAH_{NC}$) ranged from 139 to 434 µg kg^{-1} (with a mean of 268 µg kg^{-1}). The concentrations of the lower molecular weight PAHs (LMW) ranged from 50 to 283 µg kg^{-1}at locations SU7 and SU8 respectively whereas the higher molecular weight PAHs (HMW) ranged from 171 to 792 µg kg^{-1}at locations SU2 and SU7 respectively.

Table 3 gives the summary ANOVA result of the concentrations of PAHs among the different sampling locations.

Table 3: ANOVA result of PAHs in soils

Source of Variation	SS	*df*	MS	*F*	*P-value*	*F crit*
Between Groups	26919.15	9	2991.017	0.992162	0.448988	1.942796
Within Groups	452196.8	150	3014.645			
Total	479115.9	159				

As shown in Table 3, the concentrations of PAHs in the soils of the different sampling locations do not varied significantly since p-value is greater than 0.05 and Fcal < Fcrit.

Table 4 gives the comparison of PAHs in the urban soils with others reported around the world.

Table 4: Comparison of PAHs in the urban soils with others reported around the world

Location	No of PAHs analyzed	Concentration (µg kg^{-1})	Reference
Udu, Nigeria	16	269-853	This study
Warri, Nigeria	16	188 – 684	Iwegbue *et al.* (2016)
Asaba, Nigeria	16	105-787	Emoyan *et al*. (2018)
Lagos, Nigeria	16	111–15,577	Ehigbor *et al*. (2020)
Novi Sad, Serbia	16	22-2,247	Skrbic *et al*. (2017)
Miami, Florida-USA	16	251 – 2,364	Banger *et al.* (2010)
Lisbon, Portugal	16	6.3-22,679	Cachada *et al*. (2012)
Kuruksheta, India	16	16.1 – 2,538	Kumar *et al.* (2012)
Shanghai, China	16	83.3-7,220	Wang *et al.* (2013)
Hangzhou, China	16	181-1,981	Yu *et al.* (2014)
Greater London, United Kingdom	16	4,000-6,7000	Vane *et al.* (2014)
Bratislava, Slovakia	16	4,500-12,151	Hiller *et al.* (2015)
Bergen, Norway	16	ND-200,000	Haugland *et al*. (2008)
Minsk, Belarus	16	ND-26,340	Kukharchk *et al*. (2013)
Torino, Italy	15	148-23,500	Morillo *et al*. (2007)
Glasgow, United Kingdom	15	1,487-51,822	Morillo *et al*. (2007)

It is observed from Table 3 that the concentrations of Σ16 PAHs in the urban soils in this study were comparable to those reported for urban soils in Delta State (Emoyan *et al*., 2018; Iwegbue *et al*., 2016) but lower than others reported around the world.

The occurrence pattern of PAHs rings in the urban soils is displayed in Figure 1.

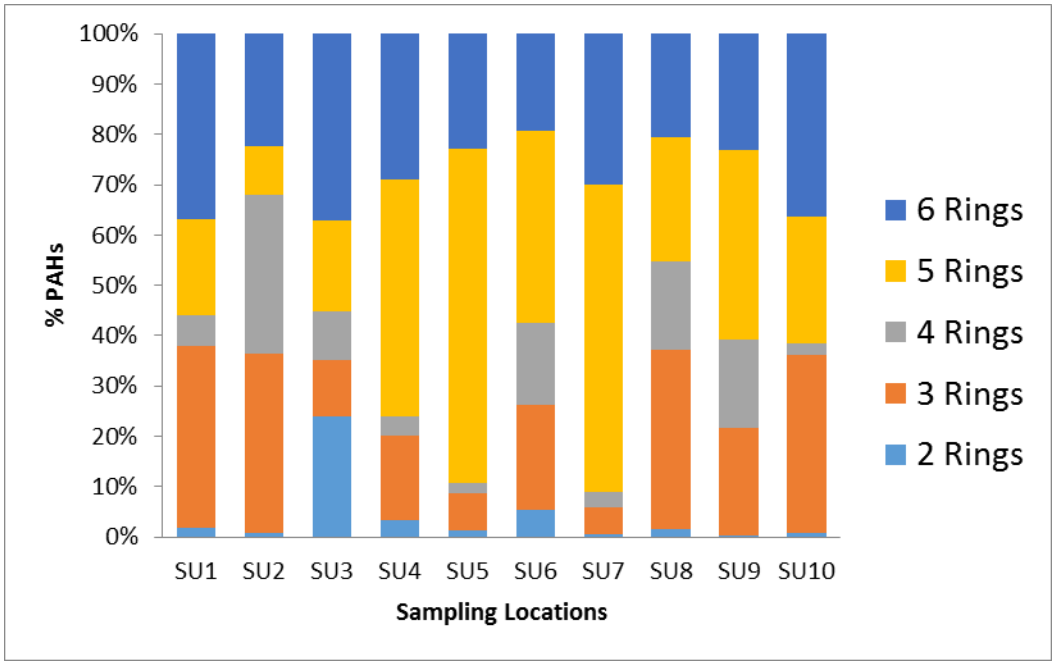

Figure 1: Occurrence pattern of PAHs rings in the urban soils

On the average, the occurrence pattern of the PAHs with respect to ring size was in the order of 5 rings > 6 rings > 3 rings > 4 rings > 2 rings (Figure 1).

The isomeric ratios of PAHs use for source apportionment in the urban soils in this study are shown in Table 5.

Table 5: Isomeric ratios of PAHs in the urban soils

Location	BaA/ (BaA+Chry)	IndP/ (IndP+BghiP)	Ant/ (Ant+Phen)	Flt/ (Flt+Pyr)	LMW/ HMW	COMB PAHs/ TPAHs	Total Index
SU1	0.00	0.08	0.98	0.95	0.61	0.54	12.3
SU2	0.44	1.00	1.00	0.80	0.57	0.64	16.2
SU3	0.07	0.65	0.70	0.50	0.54	0.59	9.94
SU4	0.10	0.79	0.68	0.56	0.25	0.67	10.3
SU5	0.82	0.64	0.73	0.20	0.10	0.60	13.2
SU6	0.07	0.21	0.50	0.78	0.35	0.54	7.71
SU7	0.63	0.10	0.94	0.76	0.06	0.60	14.6
SU8	0.59	0.28	0.02	0.44	0.59	0.49	4.79
SU9	1.00	0.55	0.85	0.27	0.28	0.66	15.2
SU10	0.50	0.20	0.03	1.00	0.57	0.48	5.60

As shown in Table 5, the ratio of BaA/(BaA+Chy) in these urban soils from Udu varied between 0.0 and 1.0. The IndP/(IndP+BghiP), Ant/(Ant+Phen) and Flt/(Flt+Pyr) ratios varied from 0.08 to 1.0, 0.02 to 1.0 and 0.20 to 1.0 respectively. The ΣLMW/ΣHMW and ∑CPAHs/∑16PAHs ratios of the soil samples varied from 0.06 to 0.61 and 0.48 to 0.67 respectively. The total index values for the urban soil samples in this study varied between 4.79 and 15.2.

Discussion

PAHs concentration in soils: The concentrations of the Σ16 PAHs ranged from 269 µg kg^{-1} at SU3 to 853 µg kg^{-1} at SU5. The concentrations of PAHs in the soils of the different sampling locations do not varied significantly ($p > 0.05$ and Fcal < Fcrit) (Table 2). The concentrations of PAHs in these soils were higher than the background value of 1-10 µg kg^{-1}, for typical endogenous PAHs in soils (Edwards, 1983). The concentrations of ∑16 PAHs in 60 % and 40 % of these soils falls into weakly contaminated (200-600 µg kg^{-1}) and contaminated (600-1000 µg kg^{-1}) categories according to Malizewkwa-Kordybach (1996) classification. The

concentrations of Σ16 PAHs in these soils were below the target value (1000 µg kg^{-1}) set for soil by the Department of Petroleum Resources and the Dutch Government (DPR, 2002; Netherlands Ministry of Housing and Environment, 1994). The concentrations of Σ16 PAHs in the urban soils in this study were comparable to those reported for urban soils in Delta State (Emoyan *et al*., 2018; Iwegbue *et al*., 2016) but lower than others reported around the world (Table 4).

On the average, the concentrations of the individual PAH compounds followed the order: DahA > BghiP > IndP > BbF > BaP > Phen > Ace > BkF > Ant > Flu > Nap > Chry > Flt = Pyr > Acy > BaA. The highest concentration of Nap was found in SU3 while that of Acy and Chry were observed at SU6. Location SU9 has the highest Ace and Pyr concentrations while SU1 has the highest Flu and Ant concentrations. Furthermore, the highest concentration of Phen and BaA were recorded in SU8 and that of Flt was recorded in SU2. Location SU7 has the highest BbF, DahA and BghiP concentrations while SU5 recorded the highest BkF, BaP and IndP concentrations. The occurrence pattern of the PAHs with respect to ring size was in the order of 5 rings > 6 rings > 3 rings > 4 rings > 2 rings (Figure 1). The higher molecular weight (4-6 rings) PAHs were the predominant PAHs in these urban soils accounting for 76% of the total PAHs concentrations compared to the 24 % accounted for by the lower molecular weight (2-3 rings) PAHs. This may be attributed to the faster rate at which soil bacteria use 2-3 rings PAHs for energy in biodegradation processes (Li *et al*., 2010) as well as the association of 4-6 rings PAHs with airborne particulates that undertake a single-hop transport pathway making them to accumulate in soils nearer to the PAHs sources. In this study, the carcinogenic PAHs were the dominant PAHs in these urban soils. The concentrations of the carcinogenic PAHs ($\sum PAH_{7C}$) ranged from 90 to 709 µg kg^{-1} (with a mean of 324 µg kg^{-1}) while the non-carcinogenic PAHs ($\sum PAH_{NC}$) ranged from 139 to 434 µg kg^{-1} (with a mean of 268 µg kg^{-1}).

Source apportionment of PAHs using isomeric ratios: PAHs in soils originates from both pyrogenic and petrogenic sources. The isomeric ratios of PAH have been used to distinguished between pyrogenic and petrogenic sources by several researchers (e.g. Yunker *et al.*, 2002; Wang *et al.*, 2011; Semlali *et al.*, 2012; Tesi *et al*., 2016; Iwegbue *et al*., 2020). Based on the PAH isomeric ratios measurement compiled by previous researchers, BaA/(BaA + Chry) ratio < 0.2 = petroleum origin; 0.2-0.35 = petroleum combustion and > 0.35 = coal and biomass combustion. IndP/(IndP+ BghiP) ratio < 0.2 = petroleum origin; 0.2-0.5 = petroleum combustion and > 0.5 = combustion of coal, wood and grass. Ant/(Ant + Phen) ratio < 0.10 = petroleum origin and > 0.1 = combustion processes while Flt/(Flt + Pyr) ratio < 0.14 = petroleum origin; 0.4-0.5 = petroleum combustion and > 0.5 = coal and biomass combustion. The ratios of ∑LMW/∑HMW and ΣCOMB PAHs/Σ16PAHs < 1 = combustion of fossil fuels or wood and > 1 = petroleum origin. Total index is the summation of the indices standardized by their individual limits between low and high temperature processes (Iwegbue *et al*., 2016). The total index = (BaA/(BaA+ Chry))/0.2 + (IndP/(IndP + BghiP))/0.5 + (Ant/(Ant +Phen))/0.1 + (Flt/(Flt + Pyr))/0.4. Total index value < 4 = low temperature combustion processes and >4 = high temperature combustion processes. The isomeric ratios of PAHs in the urban soils in this study are shown in Table 5. The ratio of BaA/(BaA+Chy) in these urban soils from Udu varied between 0.0 and 1.0. The BaA/(BaA+Chy) ratio indicates that PAHs in 60 % of the soils originates from combustion of petroleum, coal and biomass while PAHs in 40 % of the soils originates from petroleum inputs. The IndP/(IndP+BghiP), Ant/(Ant+Phen) and Flt/(Flt+Pyr) ratios varied from 0.08 to 1.0, 0.02 to 1.0 and 0.20 to 1.0 respectively. The IndP/(IndP+BghiP), Ant/(Ant+Phen) and Flt/(Flt+Pyr) ratios indicates that the PAHs in 80 % of these urban soils from Udu originates from combustion of petroleum, coal and biomass while PAHs in 20 % of the soils originates from petroleum inputs. The ΣLMW/ΣHMW and ∑CPAHs/∑16PAHs ratios of the soil samples varied from 0.06 to 0.61 and 0.48 to 0.67 respectively. The ΣLMW/ΣHMW and ∑CPAHs/∑16PAHs ratios of all the soils were < 1.0 which suggests that the PAHs in these urban soils of Udu originate from combustion processes including fossil fuels and wood combustion. The total index values for the urban soil samples in this study varied between 4.79 and 15.2. The total index values were >4.0 implying that PAHs in these urban soils originates from high temperature combustion processes.

Conclusion

The result of this study has provided information on the occurrence and sources of PAHs in soil of the study area. The concentrations of the total PAHs in these urban soils falls into the range of weakly contaminated to contaminated category. The higher molecular weight and carcinogenic PAHs were the predominant PAHs in these urban soils. The isomeric ratios used for source apportionment indicate that the PAHs in these urban soils originated majorly from high temperature combustion of petroleum, coal, fossil fuels and biomass such as woods.

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