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Concentrations of polycyclic aromatic hydrocarbons in soils of a mangrove forest affected by forest fire

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Surface soils affected by forest fires from Igbanko mangrove forest in Nigeria were analyzed for 16 EPA priority polycyclic aromatic hydrocarbons (PAHs) using gas chromatography–mass spectrometry (GC–MS). The total PAHs concentrations in the soils ranged from 63 to 188 $\mu g\,kg^{-1}$ dry weight (average: 108 $\mu g\,kg^{-1}$). The three predominant PAHs in the soils were naphthalene (Na), fluoranthene (Flu), and benzo(b)fluoranthene (BbF). Compared to the control sample (19 $\mu g\,kg^{-1}$), elevated PAHs concentrations were observed in the soils, an indication of some level of PAHs contamination. PAHs source diagnostic ratios of Flu/(Flu + Pyr) and Ant/(Ant + Phe) indicated that the PAHs have a pyrogenic origin which may have resulted from combustion of grass, wood, or coal. An assessment based on Canadian soil quality guidelines indicated that the studied locations do not pose any serious adverse risk on human health.

Keywords: polycyclic aromatic hydrocarbons; forest fire; mangrove forest; pyrogenic; soil

Introduction

Polycyclic aromatic hydrocarbons (PAHs) containing two or more fused benzene rings belong to one of the most important classes of environmental pollutants. Determination of PAHs in the environment has received much attention for over the past three decades due to their persistent, toxic, mutagenic, and carcinogenic properties (International Agency for Research in Cancer 1983). Sixteen of the PAHs have been listed by the United States Environmental Protection Agency (US EPA) as "priority pollutants". PAHs are ubiquitous environmental contaminants and derived mainly from anthropogenic sources (Gschwend and Hites 1981; Woodhead, Law, and Matthiessen 1999). They are known to accumulate in sediments, especially anerobic sediments (Bauer and Capone 1985; Coates et al. 1997), as well as in mussels and other aquatic invertebrates through which they could enter into food webs and pose a risk to human health (Malins et al. 1988; Baumard et al. 1998; Law and Klungsoyr 2000). They are formed as a result of incomplete combustion of fossil fuels and other organic substances (pyrogenic) and are also constituents of crude oil

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and its refined products (petrogenic) (Zeng and Vista 1997; Yunker et al. 2002; Zakaria et al. 2002).

Forest fires have been identified as potential source of organic pollutants in the environment (Gabos et al. 2001; Simoneit 2002; Kim, Oh, and Chang 2003; Radojevic 2003). Kim, Oh, and Chang (2003) studied the difference in the concentration and distribution of PAHs and polychlorinated dibenzo-*p*-dioxins and dibenzofurans in both burnt and unburnt soils after a forest fire, and reported a higher concentration of these compounds in the burnt soils compared to the unburnt soils. In a review work by Radojevic (2003) on the chemistry of forest fires, close to 100 compounds were identified as resultant effects of wood smoke. The effects of fire on soil and its inherent effects on soil properties depends on several factors such as the severity of the fire which in turn is largely dependent on several other environmental factors which influence the combustion process such as temperature, humidity, nature and amount of dead fuel, wind speed, and topography of the area (Certini 2005).

The objective of this study was to determine the concentrations, distributions, and sources of 16 EPA priority PAHs in soils from Igbanko mangrove forest located in Lagos, Nigeria. It has been suggested that occasional forest burning in the area could have adverse effects on the adjacent Igbanko community and Ologe lagoon.

Materials and method

The study area

Igbanko mangrove forest lies between latitudes 6°8′ and 6°5′N and longitudes 3°3′ and 3°7′E (Figure 1). The forest is situated in Lagos state, one of the most populous metropolitan cities in Africa, with a total population of 18 million. The mangrove is strategically located in the estuary harbor of Lagos state, bordered by a multipurpose Ologe lagoon in the east which opens into the Atlantic Ocean and Igbanko town in the west (Figure 1). The mangrove experiences occasional forest burning yearly, especially during the peak of dry Harmattan period (January–February).

Sample collection

Twenty soil samples were collected from three different locations in April 2008 (Figure 1). Samples were taken from locations representing the beginning, middle, and end of the forest. The soils were sampled at $25 \times 25 \, m^2$ sampling plots with 5–10 sub-samples collected from the top 10 cm layer. The litter layer of the soil samples was removed before sieving with a 200 micron sieve. The sieved soils were mixture of silt and sand. A control sample was collected at the adjacent Igbanko community (L4) where there have not been any burning activities. The soils were collected in pre-cleaned polyethylene bags, frozen at $-20°C$, and immediately shipped within 24 h to Guangzhou Institute of Geochemistry, China, in the frozen state by plane and stored in a freezer until analysis.

Reagents

Deuterated surrogate standards (naphthalene-d_8, Na-d_8; acenaphthene-d_{10}, Ace-d_{10}; phenanthrene-d_{10}, Phen-d_{10}; chrysene-d_{12}, Ch-d_{12}, and benzo [ghi] perylene-d_{12}, BghiP-d_{12}. Sixteen EPA–PAHs standards (Napthalene, Na; acenaphthylene, Acen; acenaphthene, Ace; fluorene, Fl; phenanthrene, Phe; anthracene, Ant; fluoranthene, Flu;

Figure 1. The map of the study area showing the sampling locations, the inset maps show the location of study area (top left) in Nigeria and the location of Nigeria in Africa (top right).

pyrene, Pyr; benzo(a)anthracene, BaA; chrysene, Ch; benzo(b)fluoranthene, BbF; benzo(k)fluoranthene, BkF; benzo(a)pyrene, BaP; ideno(1,2,3-cd) pyrene, IcdP; dibenzo(a,h)anthracene, DahA; benzo(ghi)perylene, (BghiP), and internal standards (2-fluoro-1,1-bipenyl, p-terphenyl-d_{14}, and dibenzo(a,h)anthracene-d_{14}) were purchased from Accustandard Incorporated, New Haven, USA with $98+\%$ purity.

Sample preparation and instrumental analysis

The samples were allowed to thaw at room temperature after removal from the freezer and freeze-dried. The freeze-dried samples were further homogenized, ground with a pestle and mortar, and sieved using a 2 mm sieve. Samples (20–50 g) were Soxhlet-extracted using dichloromethane (DCM) for a minimum of 48 h. Surrogate standards (Na-d_8, Ace-d_{10}, Phe-d_{10}, Ch-d_{12}, BghiP-d_{12}) were added prior to extraction. Sulfur was removed by addition of activated copper to the extracts (Tan, Kong, and Chiu 1993). Subsequently, samples were filtered using Whatman Grade 1 filter paper, extracts were concentrated using a rotary evaporator to approximately 1 mL and the solvent was exchanged for hexane. Concentrated extracts were fractionated on glass column packed with silica-alumina (2:1) into aliphatic, aromatic, and polar fractions by successive elution with 20 mL

of hexane, 70 mL of hexane/DCM (7/3 in v/v), and 25 mL of methanol, respectively (Wang et al. 2007).

Gas chromatography–mass spectrometry (GC–MS) analyses in the selected ion monitoring (SIM) mode (Model 2010 GC–MS, Shimadzu, Japan) were performed with a fused silica column (HP-5MS, 30 m × 0.25 mm i.d., 0.25 μm film thickness). Ultrapure helium gas was used as carrier gas. The mass spectrometer was operated in the electron impact mode at 70 eV. The aromatic fractions were injected with an autosampler in the splitless/split mode with a split time of 1 min after injection and the injector temperature was programmed from 100°C to 280°C at the maximum ramping rate ($\sim$200°C min^{-1}). The column temperature was initiated at 60°C, heated to 200°C at 5°C min^{-1}, then to 250°C at 2°C min^{-1} and further heated to 280°C at 10°C min^{-1} (hold for 20 min), and finally heated to 290°C at 10°C min^{-1} (hold for 5 min). Compound identification was based on matching retention times of target analytes with those of reference standards; Na, Acen, Ace, Fl, P, A, Fluo, Py, BaA, Ch, BbF, BkF, BaP, IP, DahA, and BiP. Quantification was performed with a conventional internal calibration method based on 5-point calibration for individual compounds (0.1, 0.5, 1.0, 2.0, 5.0 ppm) (Wang et al. 2007). Three internal standards were used to quantify compounds with corresponding ring number. The limit of quantification of each target compound is 0.5 μg kg^{-1}.

Determination of total organic carbon

Total organic carbon (TOC) contents were determined on freeze-dried, ground soil samples, following acid treatment with 10% HCl for 24 h, to remove carbonate. The carbonate-free samples were rinsed thrice with distilled water to remove the acid residue and dried at 60°C for 48 h. The organic carbon content was determined using a Perkin Elmer CHN 2400 elemental analyzer (Hedges and Stern 1984; Wang, Zhang, and Chen 2001).

Quality assurance/quality control

Extraction thimbles, silica and alumina used for the cleanup and fractionation were pre-extracted with methanol and DCM before use. All solvents were of analytical grade and redistilled. Field blanks, laboratory blanks, spiked blanks, and two replicate samples were analyzed along with field samples. Each replicate sample was analyzed twice and the relative standard deviations (RSD) of replicate samples were <10%. The average recoveries of the surrogate standards ranged from 65% to 99% in all the blanks and from 75% to 98% in the sediment samples with the exception of BghiP-d_{12} which ranged from 90% to 110%. All the spiked blanks (standards spiked into solvents) had surrogate standard recoveries in the range of 75–98% and the matrix spiked samples (standards spiked into sample) had surrogate recoveries in the range of 68–99% excluding perylene$-d_{12}$ recoveries which ranged from 95% to 115%. The limits of quantification were calculated by dividing the lowest concentrations of the calibration curves by the actual sample weights. It should be noted that the measured concentrations were not corrected by the surrogate recovery data.

Results and discussion

The total concentrations of the 16 PAHs ($\sum_{16}$PAH) in the forest soils ranged from 63 to 188 μg kg^{-1} dry weight, with a mean value of 108 μg kg^{-1} (Table 1). With the exception of

Table 1. Concentrations of PAHs ($\mu g\,kg^{-1}$ dry weight) in surface soils from Igbanko Mangrove forest and Igbanko community.

| | PAHs | | | | |
| | Igbanko mangrove forest | | | | Igbanko community |
Location	L1	L2	L3	Average	L4
Na	28.0 ± 5.25	12.6 ± 1.87	6.6 ± 11.7	22.4 ± 8.51	5.92 ± 0.96
Acen	0.38 ± 0.07	0.26 ± 0.04	0.39 ± 0.16	0.34 ± 0.07	0.14 ± 0.02
Ace	0.38 ± 0.07	0.36 ± 0.05	1.11 ± 0.18	0.62 ± 0.43	0.30 ± 0.05
Fl	1.00 ± 0.19	0.70 ± 0.10	1.11 ± 0.43	0.94 ± 0.21	0.36 ± 0.06
Phe	2.88 ± 0.54	3.28 ± 0.48	12.6 ± 1.49	6.27 ± 5.52	1.05 ± 0.17
Ant	0.40 ± 0.07	0.53 ± 0.08	1.81 ± 0.23	0.91 ± 0.78	0.19 ± 0.03
Flu	2.84 ± 0.53	5.85 ± 0.86	22.3 ± 2.44	10.3 ± 10.50	1.02 ± 0.17
Pyr	2.72 ± 0.51	5.01 ± 0.74	18.4 ± 2.09	8.70 ± 8.45	0.69 ± 0.11
BaA	2.17 ± 0.41	4.79 ± 0.71	14.3 ± 2.00	7.08 ± 6.36	0.80 ± 0.13
Ch	3.58 ± 0.67	5.74 ± 0.85	16.4 ± 2.42	8.57 ± 6.86	1.18 ± 0.19
BbF	7.45 ± 1.39	16.3 ± 5.23	37.0 ± 6.33	20.3 ± 15.17	3.61 ± 0.59
BkF	2.66 ± 0.50	5.31 ± 1.45	12.7 ± 2.08	6.88 ± 5.19	1.65 ± 0.27
BaP	2.01 ± 0.38	3.13 ± 0.46	6.82 ± 1.32	3.99 ± 2.51	0.66 ± 0.11
IcdP	2.80 ± 0.52	4.40 ± 0.65	8.00 ± 1.86	5.07 ± 2.66	0.30 ± 0.05
DahA	0.07 ± 0.01	0.18 ± 0.03	0.20 ± 0.08	0.15 ± 0.07	0.00 ± 0.00
BghiP	3.10 ± 0.58	4.54 ± 0.67	8.30 ± 1.94	5.31 ± 2.68	0.61 ± 0.10
($\sum_{16}$PAHs)[a]	62.5	73.0	188	108	18.5
TOC (%)	0.71	0.74	0.54		0.37
Phe/Ant	7.28	6.19	6.99		5.43
Flu/Pyr	1.05	1.17	1.22		1.48
BaA/(BaA + Ch)	0.38	0.45	0.47		0.40
Flu/(Flu + Pyr)	0.51	0.54	0.55		0.60
(IcdP/(IP + BghiP))		0.47	0.49		0.33

Notes: Phe/Ant, ratio of phenanthrene/anthracene; Flu/Pyr, ratio of fluoranthene/pyrene; BaA/(BaA + Ch), ratio of benzo(a)anthracene/(benzo(a)anthracene + chrysene); (IcdP/(IcdP + BghiP)), ratio of Ideno(12,3-cd)pyrene (IcdP)/(Ideno(123-cd)pyrene (IcdP) + Benzo(ghi)perylene).
[a]The total concentration of 16 PAHs in $\mu g\,kg^{-1}$ dry weight.

L3 sample, the total PAHs concentrations in the other two locations fall within the values reported for uncontaminated forest soil globally (5–100 $\mu g\,kg^{-1}$) (Menzie, Potocki, and Santodonato 1992). However, in comparison to the control samples (19 $\mu g\,kg^{-1}$), elevated level of concentrations of PAHs were observed in the forest soils (average: 108 $\mu g\,kg^{-1}$). The mean concentrations of individual PAHs in Igbanko mangrove forest soils and control sample (L4) are shown in Figure 2. The PAHs distributions in all the samples were dominated by Na, Flu, and BbF. Individual PAHs in Igbanko mangrove forest soils also showed higher values compared to the corresponding values in the control sample (Figure 2). The total concentration of $\sum_{13}$ PAHs in soils from two industrial zones in Korean Peninsula, South Korea, ranged between 110 and 179 $\mu g\,kg^{-1}$ (Hashmi et al. 2005). Urban soils from Beijing, China have a total concentration of 16 PAHs ranging from 467 to 5460 $\mu g\,kg^{-1}$ (Li et al. 2006). A mean value of 187 $\mu g\,kg^{-1}$ of 16 PAHs was reported in rural soils in the United Kingdom by Wild and Jones (1995). Therefore, concentrations of PAHs in the studied samples are in the lower range of values reported for soils from different sources (Table 2), and similar to those reported for forest soils in other countries (Menzie, Potocki, and Santodonato 1992).
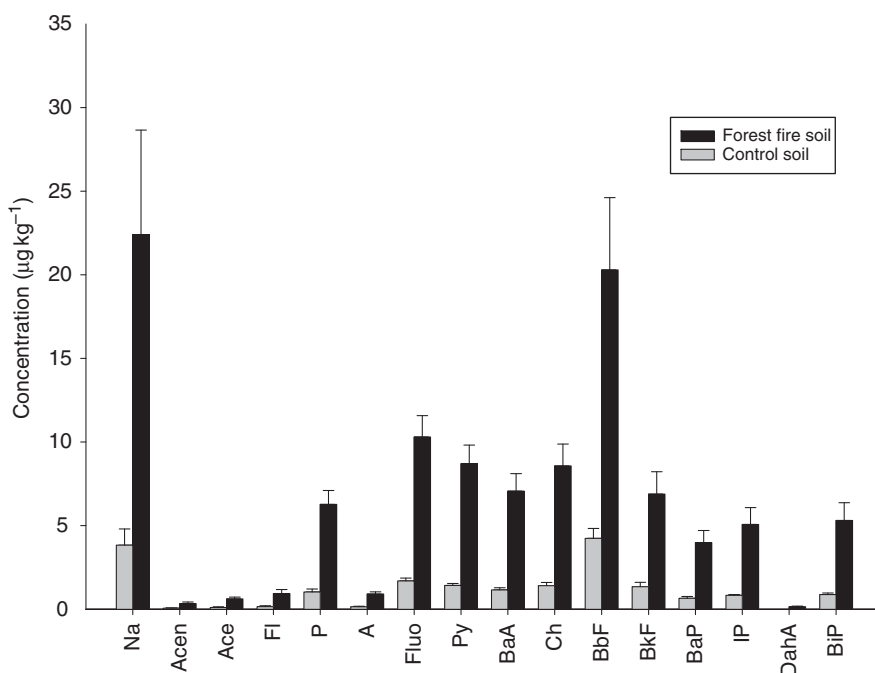
Figure 2. Relative distribution pattern of individual PAHs in Igbanko forest soils (L1, L2, and L3) and control sample (L4). Bars represent average concentration of individual PAHs and vertical lines represent standard deviations, respectively.

The main concern with regard to PAHs in the environment stems out from the fact that the majority of them are carcinogenic, both in humans and other mammals, while some have also been classified as mutagenic e.g., DahA, BaP, and BghiP (Hall and Grover 1990; Colombo et al. 2005). However, since the mangrove forest lies within the metropolitan and residential areas where inhabitants depend mostly on the water from rivers and shallow wells, it is expedient to assess the impact of the PAHs loads in the soils on the environment and human health. The studied soils did not seem to pose any serious adverse effects on human health, because the concentrations of the PAHs were below the minimal concentrations listed under Canadian soil quality guidelines (Table 3).

The mean values of the TOC in the soil samples are listed in Table 1. The TOC values ranged from 0.37% to 0.74% with a mean value of 0.67%. In general, soils taken from areas affected by fire showed relatively higher percentage of TOC compared with the soil not affected by fire (sample L4) collected from Igbanko community (0.37%). It has been reported that burning of biomass could lead to an increase in TOC in the affected soils due to the addition of partially combusted materials or to the accumulation of charcoal (Raisen 1979; Iglesias, Cala, and Gonzalez 1997; Kim, Oh, and Chang 2003). Figure 3 shows the plot of total PAHs concentration against the TOC. It is apparent that no correlation exists between the sum of PAHs concentration and TOC values in the samples ($R^2 = 0.11904$). The nonlinear relationship between the TOC and PAHs concentrations in the soils shows that the PAHs are recent contaminants and are yet to fully partition into the organic matter in the soils. Several workers have reported a linear relationship between PAHs abundance in sediments and TOC values (Neff 1979; Knezovich, Harrison, and

Table 2. Global distribution of PAHs in soils and sediments.

Location	Number of PAHs	$\sum$PAHs (μg kg^{-1})	Source type	Reference
Korea Peninsula	13	109–178	Industrial	(Hashmi et al. 2005)
Beijing, China	16	467–5470	Urban soil	(Li et al. 2006)
Beijing, China	16	16–3884	Surface soil	(Ma et al. 2005)
Canada	17	1400[a]	Highway	(Wang and Meresz 1982)
United Kingdom	7	2000[a]	Motorway	(Butler et al. 1984)
United Kingdom	16	187[a]	Rural soils	(Wild and Jones 1995)
United States	14	3000[a]	Highway	(Yang et al. 1991)
Japan	8	1300 ± 800	Urban soil	(Spitzer and Kuwatsuka 1993)
United Kingdom	16	2700 ± 500	Urban soil	(Meharg et al. 1998)
New Orleans, United States	16	3731[a]	Urban soil	(Mielke et al. 2001)
West Macedonia, Greece	16	55.2–495	Lignite fire Power plant	(Stalikas, Chaidou, and Pilidis 1997)
Five cities (Tallinn, Helsinki, Vilnius, Chicago and London)	16	1092[a]	Urban soil	(Saltiene, Brukstien, and Ruzgyte 2002)
Tokushima, Japan	13	610.6[a]	Urban soil	(Yang, Zhang, and Korenaga 2002)
From several countries	–	5–100	Forest soil	(Menzie, Potocki, and Santodonato 1992)
Igbanko mangrove forest Lagos, Nigeria	16	65.5–188.0	Forest fire soil	This study

Note: [a]Mean values.

Table 3. Comparison of concentrations of PAHs (mg kg^{-1}) in soils of Igbanko Mangrove forest with values listed under Canadian soil quality guidelines.

Compounds	Land use				
	Agriculture	Residential	Commercial	Industrial	This study
Benzo(a)anthracene	0.1	1	10	10	0.007 ± 0.006
Benzo(b)fluoranthene	0.1	1	10	10	0.020 ± 0.015
Benzo(k)fluoranthene	0.1	1	10	10	0.007 ± 0.005
Dibenzo(a,h)anthracene	0.1	1	10	10	<0.001
Ideno(1,2,3-c,d)pyrene	0.1	5	50	50	0.005 ± 0.003
Phenanthrene	0.1	5	50	50	0.006 ± 0.006
Pyrene	0.1	10	100	100	0.009 ± 0.008
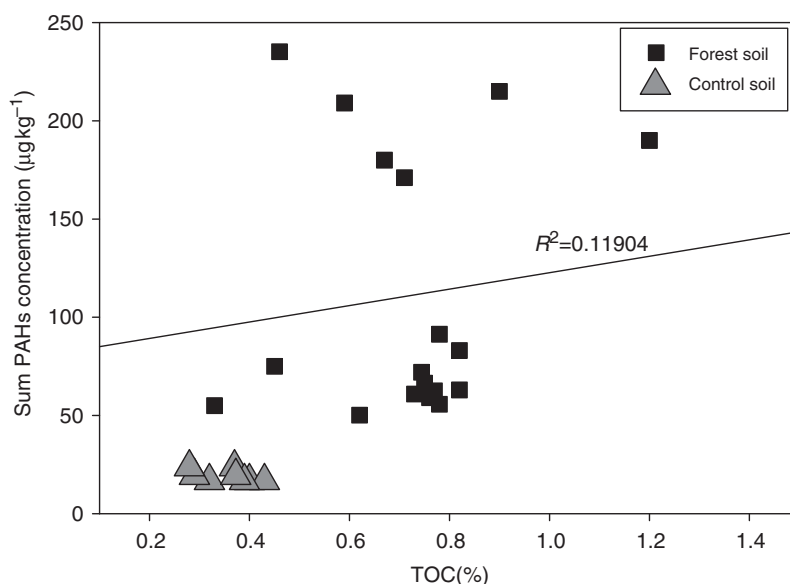
Figure 3. Total concentrations of PAHs plotted against the corresponding TOC values in Igbanko forest soils.

Wilhelm 1987). However, several factors such as organic matter composition and temperature have been suggested to affect the yields and distribution of PAHs formed during incomplete combustion of organic matter or during its thermal maturation (Baumard et al. 1999). Simpson et al. (1996) have shown that the relationship between the total PAHs and organic carbon in sediments was only significant for highly contaminated sites, where total PAHs were in excess of 2000 μg kg^{-1} dw.

The potential source of the PAHs in the soil was assessed in order to ascertain their actual origin. Petrogenic input can occur due to oil spillage or human discharge of petroleum by products while pyrolytic sources include combustion processes involving fossil fuel, forest fire, and shrub and grass fires. A number of studies have demonstrated the use of PAHs isomer ratios for source identification (Neff 1979; Garrigues et al. 1995;
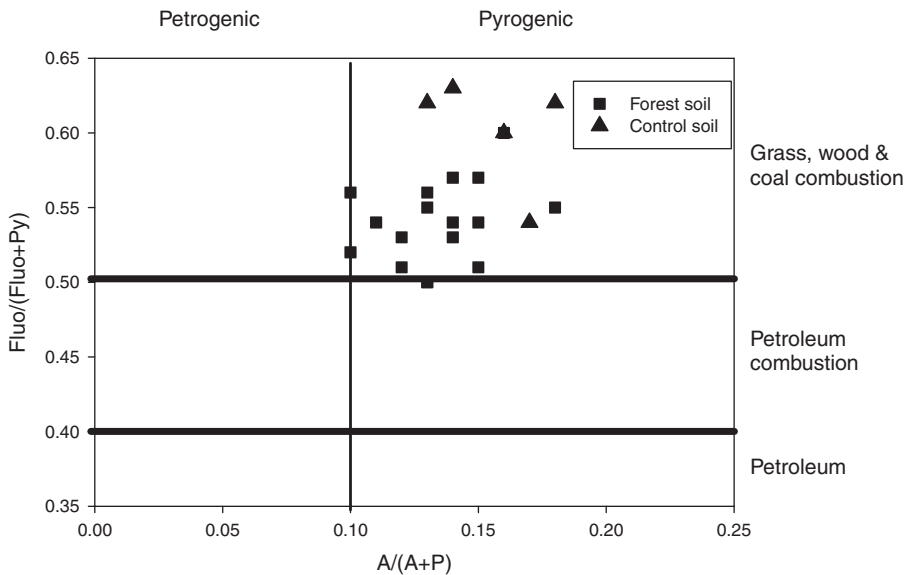
Figure 4. Plots of Flu/(Flu + Pyr) against Ant/(Ant + Phe) in Igbanko forest soils.

Baumard et al. 1998; Ma et al. 2005; Liu et al. 2008). The ratio of Flu/(Flu + Pyr) and Ant/(Ant + Phe) have been used extensively to distinguish between petrogenic and pyrolytic sources (Li et al. 2006; Liu et al. 2008). A Flu/(Flu + Pyr) ratio of <0.4 suggests typical petroleum contamination, while Flu/Flu + Pyr > 0.5 indicates PAHs from combustion of grass, wood, and coal, and 0.4 < (Flu/Flu + Pyr) > 0.5 from combustion of petroleum (Li et al. 2006). Ant/(Ant + Pyr) ratios < 0.1 are typical of PAHs from petroleum contamination, while Ant/Ant + Phe ratios > 0.1 are mainly from combustion sources (Li et al. 2006). In addition, BaA/(BaA + Ch) and IcdP/(IcdP + BiP) ratios < 0.2 indicate petroleum input while values > 0.35 and > 0.5, respectively, indicate pyrolytic source (Yunker et al. 2002). The Flu/Flu + Pyr and Ant/(Ant + Phe) ratios in the studied samples ranged from 0.51 to 0.55 and 0.12 to 0.15, respectively, while the sample from Igbanko community has values of 0.60 and 0.15, respectively. These values indicate PAHs derived from combustion of grass, wood, and coal. The samples also plotted within the zone of PAHs derived from grass, wood, and coal combustion on the plot of Flu/ (Flu + Pyr) against Ant/(Ant + Phe) in Figure 4. The BaA/(BaA + Ch) ratios in the forest fire samples ranged from of 0.38–0.49, indicating pyrolytic sources. The PAHs isomer ratios used in this study indicated that PAHs in the soils have predominantly pyrogenic sources. The wild fire that occasionally engulfed the forest is believed to be responsible for the measured PAHs.

Conclusion

The concentrations of 16 EPA priority PAHs in the surface soils affected by forest fire from Igbanko mangrove forest in Nigeria were analyzed in this study. The total concentration of $\sum_{16}$ PAHs in the soils ranged from 62.5 to 188.0 µg kg^{-1}. These values showed that the soils were not contaminated when compared with values reported for contaminated soils from other parts of the world. The concentrations of individual PAHs

in the soils were below the established minimal levels of the sediment quality guidelines, an indication that they could not pose serious ecotoxicology effect on the environment. PAHs source diagnostic ratios indicate that the PAHs in the soils have pyrogenic sources.

Acknowledgments

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