Polycyclic aromatic hydrocarbons in sediments and soils from oil exploration areas of the Niger Delta, Nigeria

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1. Introduction

The occurrence of polycyclic aromatic hydrocarbons (PAHs) in surface soil and sediments from the Niger Delta area deserves major attention. Successive oil pollution in the area and natural gas flaring have significantly altered the region’s ecology [1,2]. The estuaries located in this area also receive organic pollutants from a number of diverse sources including domestic sewage and wastewater, atmospheric fallouts, and boat engine exhausts [3]. The Niger Delta houses refineries and other petroleum-based industries; therefore, contamination of industrial effluents with oil-related residues is inevitable. At present, protection of streams and rivers from contamination by petroleum and its derivatives in the Niger Delta is inevitable. The Niger Delta is one of the major oil exploring regions in the world, with estimated 36.2 billion barrels of oil and 184 trillion cubic feet of natural gas in reserves [4]. Oil exploration activities started in the region in 1956 when the first oil well was discovered by Shell-British Petroleum at Oloibiri. These activities have led to the release of various pollutants, including trace elements and PAHs into the soil, air, and water in the region. These pollutants can pose a serious threat to the ecosystem except for two locations, Imo river and Ogoni canal where PAH-contaminated sediments were likely to be acutely toxic to certain sediment dwellers.

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derived from crude oil and its refined products. The Niger Delta stands a great chance of having varying input sources; hence there is an urgent need to decipher the sources of PAHs in the region. Finally, the present study was also intended to assess the eco-toxicological risk stemming from the loadings of PAHs in surface soil/sediments, which is of great significance for protecting human health and eco-environmental system.

2. Experimental

2.1. Sample collection

The Niger Delta covers an area of approximately 75,000 km², representing 7.5% of Nigeria’s total land area. The delta is a vast floodplain built up by the accumulation of sedimentary deposits washed down from the Niger and Benue rivers (Figure S1 in the Supporting Information; “S” designates figures and tables in the Supporting Information hereafter). Sampling was conducted in April and July 2008. Forty-four sediment samples were collected from rivers, streams and canals adjoining the exploration sites in some parts of the Niger Delta at depths ranging from 0 to 20 cm and composited from an average of five sub-samples at each location. The rivers sampled include the Great Kwa River (three tidal episodes—low, medium and high tide), Bakassi River, Calabar River, Imo River and Qua Ibo River, which all flux into the Atlantic Ocean. Twenty surface soil samples were collected from some communities very close to oil installations in the Delta (Figure S1).

Sediment samples were collected in pre-cleaned plastic bags using a hand trowel in shallow water and metallic bucket grab samplers in deep water. Representative samples were prepared by mixing five to eight sub-samples from an area of approximately 4 m² and kept in a cooling system prior to analysis. Soils were sampled at 25 m × 25 m sampling plots with five samples from the top 10 cm layer being collected from each location. The samples were transported to the laboratory in pre-extracted thimble was Soxhlet extracted with 200 mL of ethanol at 70°C for 24 h, and concentrated again to approximately 1 mL. They were subsequently shipped within 24 h to Guangzhou Institute of Geochemistry, China, in the frozen state and stored in the freezer upon arrival.

2.2. Sample preparation and instrumental analysis

Frozen samples were allowed to thaw at room temperature for a few hours upon removal from the freezer and freeze-dried. The freeze-dried samples were further homogenized, lyophilized, ground with a pestle and mortar, and sieved using a 2-mm sieve. Each sample of 20–50 g placed in a pre-extracted thimble was Soxhlet extracted with 200 mL of dichloromethane (DCM) for a minimum of 48 h. Surrogate standards (naphthalene-d10,acenaphthene-d10, phenanthrene-d10, chrysene-d12, and benzo[g,h,i]perylene-d12) were added prior to extraction. Sulphur was removed by activated copper added to the extract. The extract was concentrated using a rotary evaporator to approximately 1 mL, solvent exchanged to hexane [10], and concentrated again to approximately 1 mL.

The concentrated extract was fractionated by a glass column packed with silica–alumina (2:1) into two fractions containing aliphatic hydrocarbons and PAHs/OCPs, respectively, by successive elutions with 20 mL of hexane and 70 mL of hexane/dichloromethane (7/3 in v/v). The PAH/OCP fraction was further concentrated with a rotary evaporator at 30°C to approximately 1 mL, transferred to a 2-mL vial, and blown down to 0.5 mL under a gentle stream of nitrogen. Internal standards (2-fluoro-1,1-biphenyl, p-terphenyl-d14, and dibenz(a,h)anthracene-d14) were added to the extracts at this point.

GC–MS analyses in the selected ion monitoring mode were performed on a Shimadzu Model 2010 GC–MS (Shimadzu, Japan) with an HP-5MS fused silica column (30 m × 0.25 mm i.d., 0.25 μm film thickness). Ultrapure helium gas was used as the carrier. The mass spectrometer was operated in the electron impact mode at 70 eV. One microliter of sample extract was 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 to 280°C at the maximum ramping rate (−200°C/min). The column temperature was initiated at 60°C, ramped to 200°C at 5°C/min, 250°C at 2°C/min, and 280°C at 10°C/min (held for 20 min), and finally increased to 290°C at 10°C/min (held for 5 min). Compound identification was based on comparison of the retention times and fragmented ion profiles of the reference standards with the target analytes, whereas quantification was performed with a conventional internal calibration method [10].

2.3. Determination of total organic carbon

Total organic carbon (TOC) contents were determined on freeze-dried, ground sediment and 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 acid residues and dried at 60°C for 48 h. The measurements were conducted using a combustion method with a PerkinElmer CHN 2400 elemental analyzer [11].

2.4. Quality assurance/quality control

Extraction thimbles and silica and alumina used for the cleanup and fractionation were pre-extracted with methanol and DCM before use. All solvents were analytical grade and redistilled. Field blanks, laboratory blanks, spiked blanks, and replicate samples were analyzed along with field samples [10]. Quantification was performed using the internal calibration method based on eight-point calibration curves for individual target compounds. 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 benzo[g,h,i]perylene-d12 which ranged from 90 to 110%. Similarly, the average recoveries of the surrogate standards from the soil samples ranged from 70 to 96%, but for benzo[g,h,i]perylene-d12 they ranged from 80 to 105%. All the spiked blanks (standards spiked into solvents) had surrogate standard recoveries in the range of 75–98% and the matrix spiked samples had surrogate standard recoveries in the range of 68–99% excluding perylene-d12 which ranged from 95 to 115% (Table S1). The reporting limits (Table S1) were calculated from the lowest concentrations of the calibration curves divided by the actual sample weights. It should be noted that the measured concentrations were not corrected by the surrogate recovery data.

2.5. Data analysis

All the concentrations are presented based on the dry weight equivalent of the samples and the source diagnostic indices are calculated from the interpretative PAH concentration ratios (Table 1). The values of phenanthrene/anthracene (Phe/Ant) and fluoranthene/pyrene (Flu/Pyr) have been used extensively to distinguish between petrogenic and pyrogenic source of PAHs. Values of Phe/Ant and Flu/Pyr greater than 10 and less than 1, respectively, suggest strong petrogenic origin of PAHs [12,13] and vice versa for pyrogenic origin [14]. In addition, Ant/(Ant + Phe) < 0.1 is an indication of petroleum contamination while >0.1 indicates a dominance of combustion origin [15]. Likewise, a Flu/(Flu + Pyr) value of 0.5 illustrates a petroleum/combustion transitional point [15]. The ratio of Flu/(Flu + Pyr) > 0.5 is ascribed to the combustion of
3.1. Occurrence and spatial distribution of polycyclic aromatic hydrocarbons

The concentrations of the 28 PAHs, sum of which is defined as $\Sigma_{28}$PAH (Table S2), determined in the sediments ranged from 65 to 331 ng/g with a mean value of 80 ng/g (Table 1). The highest concentrations of $\Sigma_{28}$PAH were recorded in samples from Oginni (331 ng/g) and Olomoro canals (306 ng/g) (Table 1 and Figure S1) which are located in the hinterland hosting several oil wells and oil installations, and obviously the high concentrations recorded at these locations can be attributed to the intense exploration activities undergoing with occasional oil pipeline leakages in the area. The lowest concentration of 65 ng/g was recorded at Great Kwa River during a high tide (Table 1). In general, sediments from the areas close to oil installations, which can be attributed to the constant flux of the rivers into the Atlantic ocean (Figure S2) resulting in reduced PAH concentrations. This clearly explains the lowest concentration recorded during the high tide in the Great Kwa River.

A marked distribution pattern of the PAH concentrations in sediments was apparent (Table S2 and Fig. 1) with predominance of 2-, 3-, and 4-ring PAHs. The 2-ring PAHs accounted for approximately 45% of the total, whereas the 3- and 4-6-ring PAHs contributed approximately 10 and 42%, respectively (Fig. 1). Perylene, likewise, was the dominant constituent in the 4–6-ring PAHs, which may be hypothetized that perylene has biogenic origin and could be generated in situ through biotransformation of some precursors most likely diatom spar [18]. No correlation ($r^2 = -0.18$ and $p = 0.58$ with a two-tailed test) was found between the concentrations of perylene and the 5–6-ring parent PAHs compounds supposed to originate from anthropogenic combustion/pyrolysis processes, which implies that perylene in these sediment samples are of biotic and natural origins. A similar trend was observed in the soils, with the exception of petroleum input, inputs from both sources and combustion input, respectively. Likewise, Icdp/(Icdp + Bghip) < 0.2, between 0.2–0.5 and more than 0.5 infers petroleum input, liquid fossil fuel combustion and grass, wood and coal combustion, respectively [17], Origin 6.1 and SigmaPlot 10.0 were employed in the data analysis.

3. Results and discussion

3.1. Occurrence and spatial distribution of polycyclic aromatic hydrocarbons

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3.2. Comparison with PAH levels from other parts of the globe

Though data on PAHs in oil fields around the world are scanty, the sediments investigated in the present study can be considered to be moderately polluted with PAHs as compared to those reported for similar samples from other parts of the world (Table 2 and Fig. 2) [19–21]. In comparison with estuarine sediments in the
U.K. (Table 2) [22], the PAH concentrations in the present study were relatively low. In the rivers Blythe, Tyne, Wear and Tees, the total concentrations of 15 parent PAHs (Supporting Information) in sediments were in excess of 10,000 ng/g dry wt. [23]. Total PAH (2- to 6-ring parent and branched components) concentrations found in the Firth of Clyde, UK, ranged from 3900 to 27,000 ng/g dry wt. [24]. High PAH concentrations (>2000 ng/g dry wt.) are often found in sediments in estuarine and harbor sites and areas close to industrial or urban activities [23–25]. Total concentrations of PAHs (2- to 6-ring parent and branched) at ∼10,000 ng/g dry wt. were reported in estuarine sediments from the Pearl River Delta of South China [25] though lower values of 31–133 ng/g were obtained in sediment samples from Yellow River, China (Table 2). However, Notar et al. [26] reported lower total PAH (2- to 6-ring parent and branched) concentrations of between 30 and 600 ng/g dry wt. in sediments from the Gulf of Trieste, an area heavily utilized for tourism and industrial and agricultural activities, and classified samples with total PAH concentrations >500 ng/g dry wt. as being relatively contaminated.

Likewise, the levels of ∑28PAH in the soil samples were lower than those reported for soils from some parts of the world. The total concentrations of ∑13PAH (Supporting Information) in soils from two industrial zones in Korean Peninsula, South Korea, ranged from 109 to 79,000 ng/g [27]. Surface soils from the outskirts of Beijing, China, contained total concentrations of 16 PAHs (16 priority pollutants proposed by the United States Environmental Protection Agency; Table S2) ranging from 16 to 3884 ng/g with a mean value of 1347 ng/g [28]. In addition, Wild and Jones [22] reported a mean concentration of 187 ng/g for the 16 PAHs in rural soils of the U.K. Table 2 further compares the global PAH concentrations in soil and sediments.

The concentrations of benzo(a)pyrene (BaP), the most carcinogenic PAH in the present study, ranged from 0.17 to 10.21 ng/g with

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**Table 2**

Global distribution of polycyclic aromatic hydrocarbons in soils and sediments.

<table>
<thead>
<tr>
<th>Location</th>
<th>No. of PAHs</th>
<th>∑PAHs (ng/g)</th>
<th>Source type</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>Soils</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Beijing, China</td>
<td>16 EPA</td>
<td>467–5,470</td>
<td>Urban soils</td>
<td>[44]</td>
</tr>
<tr>
<td>Canada</td>
<td>17</td>
<td>1,400</td>
<td>Highway</td>
<td>[45]</td>
</tr>
<tr>
<td>United Kingdom</td>
<td>7</td>
<td>20,000</td>
<td>Motorway</td>
<td>[46]</td>
</tr>
<tr>
<td>United States</td>
<td>14</td>
<td>3,000</td>
<td>Highway</td>
<td>[47]</td>
</tr>
<tr>
<td>Australia</td>
<td>18</td>
<td>300–79,000</td>
<td>Chemical plant</td>
<td>[48]</td>
</tr>
<tr>
<td>Japan</td>
<td>8</td>
<td>1,300</td>
<td>Urban soils</td>
<td>[49]</td>
</tr>
<tr>
<td>United Kingdom</td>
<td>16</td>
<td>2,700</td>
<td>Urban soils</td>
<td>[50]</td>
</tr>
<tr>
<td>New Orleans (United States)</td>
<td>16</td>
<td>3,730</td>
<td>Urban soils</td>
<td>[51]</td>
</tr>
<tr>
<td>West Macedonia (Greece)</td>
<td>16</td>
<td>55.2–495</td>
<td>Lignite fire power plant</td>
<td>[52]</td>
</tr>
<tr>
<td>Linz (Austria)</td>
<td>18</td>
<td>1450</td>
<td>Industrial area</td>
<td>[48]</td>
</tr>
<tr>
<td>Zelzate (Belgium)</td>
<td>7</td>
<td>3,000–14,000</td>
<td>1.3–4.2 km from an oil refinery</td>
<td>[53]</td>
</tr>
<tr>
<td>Five cities (Tallinn, Helsinki, Vilnius, Chicago and London)</td>
<td>16</td>
<td>1,090</td>
<td>Urban soils</td>
<td>[54]</td>
</tr>
<tr>
<td>Novi Sad (Serbia and Montenegro)</td>
<td>16</td>
<td>47,900</td>
<td>Oil refinery</td>
<td>[55]</td>
</tr>
<tr>
<td>Tokushima (Japan)</td>
<td>13</td>
<td>611</td>
<td>Urban soils</td>
<td>[56]</td>
</tr>
<tr>
<td>Niger Delta, Nigeria</td>
<td>28</td>
<td>23.8–120</td>
<td>Close to oil installations</td>
<td>This study</td>
</tr>
<tr>
<td>Sediments</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>England and Wales</td>
<td>16 EPA</td>
<td>nd–102,000</td>
<td>Surface</td>
<td>[23]</td>
</tr>
<tr>
<td>Thailand</td>
<td>14</td>
<td>6–8,400</td>
<td>Surface</td>
<td>[57]</td>
</tr>
<tr>
<td>Yellow River, China</td>
<td>13</td>
<td>31–133</td>
<td>Surface</td>
<td>[16]</td>
</tr>
<tr>
<td>Niger Delta, Nigeria</td>
<td>28</td>
<td>65–321</td>
<td>Close to oil installations</td>
<td>This study</td>
</tr>
</tbody>
</table>

* Mean values.
a mean value of 1.25 ng/g in sediments and from 0.09 to 2.05 ng/g with a mean value of 0.75 ng/g in soils (Table S1). Again, these values were lower than those reported for the industrial zones in Korean Peninsula with values between 5 and 270 ng/g with a mean concentration of 55 ng/g [27]. Varying values of BaP were reported in commercial areas (90–4190 ng/g) and industrial areas (170–6030 ng/g) in Tokyo [29] and in the city of Chiang-Mai of Thailand (22 ng/g) [30]. The soils studied in this work can be regarded as being moderately polluted with PAHs compared to similar samples from other parts of the world.

3.3. Correlation between polycyclic aromatic hydrocarbons and total organic carbon

The wide disparity in the total PAH concentrations among the sediment samples was also reflected in the TOC contents which ranged from 1.03 to 6.38% with a mean value of 2.95%; TOC contents in the soil samples ranged from 0.33 to 2.33% with a mean value of 1.29% (Table 1). It is apparent from Figure S3 that no significant correlation exists between the total PAH concentrations and TOC contents (r² = 0.082 and p = 0.60 based on a two-tailed test) in the sediment samples. On the other hand, a significant correlation exists between the total PAH concentrations and TOC in soil samples (r² = 0.64 and p = 0.0022 with a two-tailed test). Several researchers have reported a linear relationship between the abundances of PAHs and TOC in sediments [31,32]. However, several factors such as organic matter composition and temperature have been suggested to affect yields and distribution of PAHs formed during incomplete combustion of organic matter or during its thermal maturation [33]. The nonlinear relationship between the TOC and PAH concentrations in the sediments indicates that the PAHs were recently generated and therefore were yet to fully partition into organic matter in the sediments. On the other hand, PAHs in the soils may have fully integrated into the soil matrix; therefore, a good correlation between the concentrations of PAHs and TOC can be expected.

3.4. Source diagnostics

Generally, the prevalence of alkylated PAHs (e.g., 2-MNap and 1-MNap approximately 15%; in sediments and approximately 14% in the soils; Fig. 1) indicates contamination from petrogenic inputs [34]. However, several molecular indices such as Flu/Pyr and Phe/Ant have also been used to ascertain emission sources [17,35]. The Phe/Ant and Flu/Pyr values in the sediments under investigation ranged from 3.2 to 44 and from 0.58 to 1.7, respectively (Table 1). The Phe/Ant and Flu/Pyr values in sediments from Uzere stream, Olomoro, and Ughelli canals ranged from 10 to 13 and 0.58 to 0.88 (Table 1), respectively, suggesting a predominant petrogenic origin for PAHs in these sediments. However, sediments from Calabar River, Imo River, Great Kwa River, and Bakassi River had Phe/Ant and Flu/Pyr ratios ranging from 3.65 to 9.05 and from 1.2 to 1.53, respectively. These values suggest a mixed petrogenic and pyrolytic origin for PAHs in the sediments. Based on the Ant/(Ant + Phe) values (Table 1), the input sources of PAHs in sediments of Uzere stream, Qua Ibo River, Bakassi River, Olinni, Olomoro, and Ughelli canals were mainly petrogenic while those from Calabar River, Imo River, and Great Kwa River were considerably pyrogenic. This observation is consistent with the conclusion drawn from the assessment of Phe/Ant and Flu/Pyr given above. The BaA/(BaA + Chr) values ranged from 0.03 to 0.35 in sediment and 0.18 to 0.45 in soil samples (Table 1), indicating that sediment samples from Oginni, Olomoro and Ughelli are predominantly contaminated with PAHs of petrogenic origin while those from other locations contain PAHs from both petroleum and combustion sources. The PAHs are likewise of petrogenic origin in the soil samples from Oginni, of mixed sources in Irri and Uzere soils and of combustion source in soils from Olomoro. The Icdp/(Icdp + Bghip) values ranged from 0.14 to 0.56 and 0.09 to 0.42 in the sediments and soils, respectively (Table 1), implying that PAHs in sediments from Qua Iboe River (QIRs) are largely petrogenically derived while those in sediments from other locations could be contributed from fossil fuel combustion.

The Phe/Ant and Flu/Pyr ratios in the soils from all the locations ranged from 6.0 to 9.2 and from 1.2 to 1.5, respectively (Table 1). These values strongly affirmed a predominant pyrogenic origin, with minor petrogenic contributions, of PAHs in the soil samples. However, the plot of BaA/(BaA + Chr) versus Flu/(Flu + Pyr) (Fig. 3) suggest that some sediment samples contain PAHs of mixed sources while others have PAHs of pyrogenic origin. On the other hand, PAHs in the soil samples are mainly of pyrogenic origin, consistent with the conclusions drawn based on other diagnostic indices. Our results show that PAHs in sediments of the Niger Delta are predominantly derived from petrogenic sources with some level of pyrogenic inputs in some areas which is evident in Great Kwa River, Imo River and Calabar River and surface soils (Fig. 4). Sediments from the hinterland hosting the majority of oil installations are mainly contaminated by PAHs of petrogenic origin which obviously has resulted from incessant oil pipeline leakages. About 1.5 million tons of crude oil were estimated to have spilled in different parts of the Niger Delta since the inception of oil exploration in 1958 [36]. Gas flaring – where an obsolete “open pipe flare” method...
is being used by oil companies – is also believed to be responsible for the occurrence of PAHs of pyrogenic origin in some of the samples.

3.5. Assessment of potential ecological risk

The main concern about the occurrence of PAHs in the environment stems from the fact that they are mostly carcinogenic, both in humans and other mammals, whereas some PAHs have also been classified as mutagenic, e.g. dibenz[a,h]anthracene, benzo[a]pyrene and benzo[g,h,i]perylene [37,38]. There has been strong concern about the impact of crude oil exploration on the pollutant loads in the Niger Delta, which has caused and is still causing unrest among the citizenry and various interest groups in the area [36]. The dominant view blames oil production and its attendant consequences for the declining productivity of local economies that are mainly based on fisheries and agriculture [39,40], while some others say otherwise [8]. On the other hand, the World Bank in 1997 [41] disagreed with the popular view noted above, but attributed the productivity declines to the consequences of population increase and construction of upstream dams, among others [41]. With these conflicting claims, it should be beneficial to assess the possible impact of the PAH loads in sediments/soils on the ecosystem in the study area.

Based on the sediment quality guidelines (Table S3) which have been extensively tested and validated in several previous studies, such as that of Woodhead et al. [23], the majority of the sediments under investigation did not seem to pose adverse ecotoxicological effects, because the concentrations of all individual PAHs but naphthalene and chrysene were below the effect range low (ERV) and threshold effect level (TEL) (Tables S2 and S3). Conversely, the concentrations of naphthalene and chrysene were high enough to pose potential biological impairments in two main locations, Imo River and Oginini canal (Tables S2 and S3). The concentrations of both compounds were above the TEL values in the respective locations [42,43]. With the daily oil production output of 2.5 million barrels per day compared with the highest recorded value of 331 ng/g for polynuclear aromatic hydrocarbons in the Niger Delta, this has a minimal impact on the PAH load in the area. This, however, does not undermine the need to ascertain other environmental pollution indices in the region.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jhazmat.2009.09.099.

References


