



## Levels and Sources of Polycyclic Aromatic Hydrocarbons in Urban Soils of Akure, Nigeria

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### Authors' contributions

This work was carried out in collaboration between both authors. Both authors read and approved the final manuscript.

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### ABSTRACT

Top soils (0-15 cm) of Shasha market, North Gate motor park, Benin motor park and Ilesha motor park in Akure metropolis, Nigeria was sampled. The aim of the research was to determine the concentration of polycyclic aromatic hydrocarbons (PAHs) in frequently visited urban areas. PAHs were determined by extraction using hexane and dichloromethane in an ultra sonicator with a microprocessor for control of time and temperature. The extract was concentrated and cleaned up using Nitrogen gas. Gas Chromatography-Flame Ionization Detector (GC-FID) was used to analyze for PAHs. The  $\Sigma$ PAHs (summation of PAHs) ranged from 6.184-27.046  $\mu\text{g}/\text{kg}$  and 1.511-13.766  $\mu\text{g}/\text{kg}$ ,  $\Sigma$ Low molecular weight PAHs ranged from 3.237-14.672  $\mu\text{g}/\text{kg}$  and 0.279-8.338  $\mu\text{g}/\text{kg}$ ,  $\Sigma$ High molecular weight PAHs ranged from 2.947-12.374  $\mu\text{g}/\text{kg}$  and 0.627-5.428  $\mu\text{g}/\text{kg}$  in the samples and controls respectively. Shasha market sample and control has the highest  $\Sigma$ PAHs. Source diagnostic ratio inferred that the PAHs originated mainly from combustion sources and vehicular exhaust. The concentration of polycyclic aromatic hydrocarbons in the site samples were all higher than the control samples. Areas with very high activities had higher concentrations.

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## 1. INTRODUCTION

Polycyclic aromatic hydrocarbons (PAHs) are organic compounds/pollutants which contain more than two aromatic rings containing carbon and hydrogen atoms fused together. PAHs are usually formed by incomplete combustion or pyrolysis of organic material including wood, coal, oil, gasoline, and garbage and leaching from coal-tar products such as asphalt and roofing shingles [1,2]. PAHs can also be derived from natural sources such as forest fires and volcanic eruptions. However, the majority of PAHs found in the environment are from anthropogenic sources such as emissions from motor vehicular exhaust; burning of coal, wood and wastes; and industrial sources such as thermoelectric power generation, cooking operations, production and combustion processes. Majority of PAHs emitted to the atmosphere are deposited on soils either by dry or wet deposition [3]. Soil therefore is a good indicator of long-term environmental pollution [4,5]. Humans can be directly exposed to PAHs in soil via inhalation of flying dust, ingestion and dermal contact [6]. In addition, PAHs in soil can be transported with organic matter to water bodies especially in raining seasons [7,8,9]. For these reasons, assessment of PAHs in soils is very important. There are more than 100 PAH compounds [10]. Due to their potential mutagenic, carcinogenic and teratogenic effects on human health, PAHs have been distinguished and 16 PAHs were listed as priority pollutants by the USEPA. Benzo (a) anthracene (BaA), chrysene (Chr), benzo (b) fluoranthene (BbF), benzo (k) fluoranthene (BkF), benzo (a) pyrene (BaP), indeno (1,2,3-cd) pyrene (IND) and dibenz (a,h) anthracene (DBahA) are classified as carcinogenic by the International Agency for Research on Cancer [11]. PAHs have been studied in Nigeria; however the selected study areas have not been researched upon.

Ondo State lies between latitude  $5^{\circ} 45' 1''$  and  $8^{\circ} 15' 1''$  North and longitude  $4^{\circ} 45' 1''$  and  $6^{\circ}$  East, this means that the State lies entirely in the tropics. Ondo state has a land size of  $15,195.177 \text{ km}^2$  and 18 local government areas. Akure south local government area has a land size of  $335.805 \text{ km}^2$  and a population of 360,268 making it the

most inhabited local government area in the state [12].

The study was aimed at assessing the levels of polycyclic aromatic hydrocarbons in top soils of selected motor parks and a market in Akure metropolis. The specific objectives were to extract, clean-up the extract and analyze 16 US EPA PAHs from the soil samples.

## 2. MATERIALS AND METHODS

### 2.1 Study Area and Sampling Method

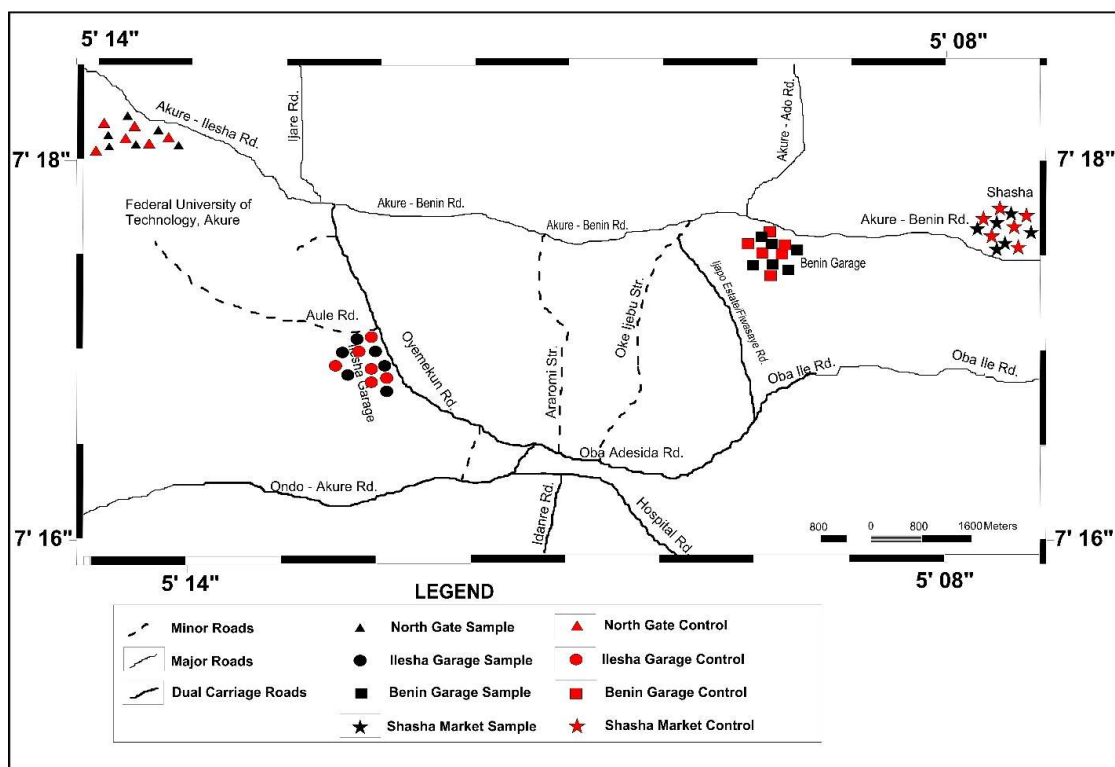
The study areas were a major open whole sale market (Shasha market) and three major motor parks/garage (FUTA North gate motor park, Ilesha motor park and Benin motor park). The study areas are within Akure metropolis of Ondo State and Nigeria. Top soil (0-15 cm) was sampled. The control samples were taken 70 m away from each study area. Six samples were collected from each study area and each control area. The six samples were homogenized to form a composite sample. A total of eight composite samples were analysed (Fig. 1). The geographical coordinates were determined using a global positioning system (GPS). The samples were collected using a stainless steel hand auger. The depth was measured using a measuring tape. The samples were carefully wrapped in foil sheets and kept in black polyethene bags, tied up and labelled immediately prior to further analysis.

### 2.2 Sample Pre-Treatment

The soil samples were spread on trays and air dried for two weeks. The dried samples were sieved using a 2 mm mesh size (British Standard) to ensure uniformity and a large surface area. The sieved samples were wrapped in foil sheets and kept in the refrigerator at  $4^{\circ}\text{C}$  until further analysis [13,14].

### 2.3 Determination of Total Soil Carbon Content (TOC)

The total organic carbon (% TOC) contents of the soil samples were determined by potassium dichromate titrimetric method. Samples were analyzed for TOC in duplicates [15].



**Fig. 1. A map showing sampling sites**

Key: North Gate Sample – NGS; North Gate Control – NGC; Ilesha Garage Sample – IGS  
 Ilesha Garage Control – IGC; Benin Garage Sample – BGS; Benin Garage Control – BGC  
 Shasha Market Sample – SSS; Shasha Market Control – SSC

## 2.4 Sample Extraction and Cleanup

Exactly 50 g of soil sample was weighed into a 250 mL beaker and 100 mL of the ratio 3:1 hexane: dichloromethane was added. The beaker was placed in a sonicator for 2 h to enable extraction of the hydrocarbons. The organic layer was poured out and the extract was dried by passing it through a funnel containing anhydrous sodium sulphate. The extract/filtrate was concentrated with nitrogen gas. A small amount of glass wool was packed into a pipette and alumina was put into the wool packed pipette. A pinch of sodium sulfate was added to the top of the packed column. A beaker was placed under the column to collect the pre-elution solvents. 10 mL of 2:1 dichloromethane: hexane mix and 15 mL of hexane was poured into the packed column and allowed to run through in order to precondition and recondition the column respectively. The extract was poured into the column and allowed to run down with the aid of 15 mL hexane in order to remove the aliphatic fractions. 20 mL of the ratio 3:1 hexane: dichloromethane was allowed to run down the

column in order to remove the aromatic fractions. The aromatic fraction was concentrated to 2 mL using nitrogen gas before gas chromatographic analysis [16].

## 2.5 Analysis of PAHs

The PAHs were quantified by external calibration performed with a Gas Chromatography-Flame Ionization Detector (GC-FID) (HP 6890 powered with HP ChemStation Rev. A 09.01, 1206 Software) with column length: 30m, column ID :0.25 µm, column film: 0.25 µm, injection temp : 250°C, detector temp: 320°C, detector: FID, initial temp 60°C for 5 min, first rate: 15°C/min for 14 min and maintained for 3 min, second rate: 10°C/min for 5 min and maintained for 4 mins, mobile phase: nitrogen gas, Nitrogen column pressure:30 psi, Hydrogen pressure: 28 psi and compressed air pressure: 32 psi. The calibration was done by firstly analyzing/injecting a mixture (0.2, 0.4, 0.6, 0.8 and 1 ppm) of mixed PAH standards into the GC-FID and plotting calibration curves for the 16 priority PAHs. The correlation coefficient was then calculated [16].

## 2.6 Quality Control and Recovery Studies

All reagents used were of analytical grade and purchased from Surechem products limited, BHC and Sigma Aldrich. All glassware's used were soaked in acid, rinsed with water, washed with detergent and rinsed with distilled water at least three times. Prior to sample analysis with the GC-FID, PAHs standard containing the 16 USEPA PAHs was analyzed and calibration curves with correlation coefficient were obtained. The correlation coefficients ranged from 0.99943-0.99992. The procedural blanks and sample duplicates were routinely analyzed and no interferences were detected.

The procedure was checked for recovery efficiencies by analyzing a soil sample spiked with PAH standards. The soil sample was spiked with 1 mg/kg (1000 µg/kg) of Acenaphthene – d12, Phenanthrene – d10, Chrysene – d10 and Perylene – d12. The percent recovery of the surrogate standards was 96.2, 98.3, 95.4 and 95.9 respectively.

## 2.7 Statistical Analysis

Statistical analysis including one-way analysis of variance (ANOVA) were performed using statistical package for social sciences (IBM, SPSS 22.0) and principal component analysis (PCA) was performed using paleontological statistics software package for education and data analysis (Past 3.01). The concentrations of PAHs were log-transformed to achieve normal distribution prior to PCA.

## 3. RESULTS AND DISCUSSION

### 3.1 Levels of PAHs

Several epidemiologic studies and research have shown increased mortality due to lung cancer in humans exposed to coke oven emissions, roofing-tar emissions, and cigarette smoke. Each of these mixtures contains benzo[a]pyrene, chrysene, benz[a]anthracene, benzo[b]fluoranthene, and dibenz[a,h]anthracene. However, it is thus impossible to evaluate the contribution of any individual PAH to the total carcinogenicity of these mixtures in humans because of the complexity of the mixtures and the presence of other carcinogens [11,12,17-19]. The  $\Sigma$ PAHs ranged from 6.184-27.046 µg/kg and 1.511-13.766 µg/kg in the

samples and controls respectively (Table 1). Shasha market had the highest concentration (27.046 µg/kg) of PAHs in both site samples and control samples. This may be attributed to the fact that the area has numerous sources of pollution such as vehicular emissions, auto workshops, food processing (roasting and cooking with coal), burning of wastes, generator fumes and others as compared to the motor parks whose major source of pollution are automobiles. North gate motor park had the lowest concentration (6.184 µg/kg) of PAHs in samples and this can be attributed to the fact that the area was recently opened to transport activities. Ilesha motor park control had the lowest concentration (1.51 µg/kg) of PAHs among the control samples and this may be attributed to the contributing factors such as bush burning, roasting and cooking with coal, vehicular emissions, etc are absent. Shasha market had the highest concentration of low molecular weight ( $\Sigma$ LMW) and high molecular weight ( $\Sigma$ HMW) PAHs. The concentration of PAHs varied according to activities ongoing in the sample areas. Areas with very high activities had higher concentrations (the concentration of PAHs increased with activity). The concentration (µg/kg) of  $\Sigma$ LMW PAHs ranged from 3.24 - 14.67 and 0.28 - 8.34 (Table 1) while the concentration (µg/kg) of  $\Sigma$ HMW PAHs ranged from 2.95 - 12.37 and 0.63 - 5.43 for site samples and control samples respectively (Table 1). Benzo (a) Anthracene, Chrysene, Benzo (b) Fluoranthene and Benzo (a) Pyrene ( $\Sigma$ PAHs 4) are carcinogenic PAHs which are used as markers. These PAHs were highest in Shasha market site sample (8.28 µg/kg) and lowest in North gate motor park site sample (2.02 µg/kg) however, it was highest in Shasha market control sample (5.09 µg/kg) and Ilesha motor park control sample (0.87 µg/kg).

The PAHs concentrations in soil samples from different areas around the world remarkably vary. The concentration of PAHs in this study was lower than those obtained by Ogoko [20] who worked on evaluation of polycyclic aromatic hydrocarbons, total petroleum hydrocarbons and some heavy metals in soils of NNPC oil depot Aba metropolis, Abia State, Nigeria which ranged from 6300 µg/kg - 7400 µg/kg. Ogunbiyi et al. [21] who researched on the assessment of polycyclic aromatic hydrocarbons concentration in sediments and soils around Ibadan, southwestern Nigeria reported the range of 1.76 µg/kg - 2926.68 µg/kg.

**Table 1. The concentration of PAHs and source diagnostic ratios of soil samples**

PAHs	BGS ( $\mu\text{g/kg}$ )	BGC ( $\mu\text{g/kg}$ )	NGS ( $\mu\text{g/kg}$ )	NGC ( $\mu\text{g/kg}$ )	SSS ( $\mu\text{g/kg}$ )	SSC ( $\mu\text{g/kg}$ )	IGS ( $\mu\text{g/kg}$ )	IGC ( $\mu\text{g/kg}$ )
Naphthalene	0.349	0.067	0.009	0.000	0.346	0.013	0.013	0.006
Acenaphthylene	0.130	0.002	0.118	0.039	0.130	0.051	0.051	0.000
Acenaphthene	0.512	0.548	0.101	0.010	0.538	0.039	0.049	0.080
Fluorene	0.406	0.007	0.086	0.010	0.527	0.071	0.071	0.005
Phenanthrene	1.210	0.042	0.161	0.037	1.270	0.404	0.509	0.015
Anthracene	0.420	0.582	0.184	0.640	0.521	1.950	2.680	0.120
Fluoranthene	8.480	0.037	2.170	0.099	9.110	3.360	4.460	0.010
Pyrene	2.270	0.260	0.408	0.316	2.230	2.450	2.830	0.043
Benzo (a) Anthracene	1.080	0.523	0.197	0.135	1.470	2.190	2.190	0.235
Chrysene	2.360	0.159	0.502	0.159	2.340	1.900	2.520	0.089
Benzo (b) Fluoranthene	1.750	0.149	0.686	0.077	1.810	0.442	0.509	0.069
Benzo (k) Fluoranthene	3.900	0.906	0.886	0.065	3.990	0.327	0.379	0.358
Benzo (a) Pyrene	2.650	2.590	0.636	0.188	2.660	0.560	0.692	0.479
Indeno {1,2,3-cd}Pyrene	0.007	0.005	0.002	0.002	0.007	0.001	0.001	0.002
Dibenzo (a,h) Anthracene	0.077	0.000	0.031	0.001	0.068	0.005	0.004	0.000
Benzo (g,h,i) Perylene	0.024	0.000	0.007	0.000	0.029	0.003	0.004	0.000
$\Sigma$ PAHs	25.625	5.877	6.184	1.778	27.046	13.766	16.962	1.511
$\Sigma$ PAHs LMW	13.777	1.545	3.237	1.151	14.672	8.338	10.663	0.279
$\Sigma$ PAHs HMW	11.848	4.332	2.947	0.627	12.374	5.428	6.299	1.232
$\Sigma$ PAHs 8	11.848	4.332	2.950	0.627	12.374	5.428	6.299	1.232
$\Sigma$ PAHs 4	7.840	3.421	2.021	0.559	8.280	5.092	5.911	0.872
Flu/Flu+Pyr	0.789	0.125	0.842	0.239	0.803	0.578	0.611	0.699
Ant/Ant+Phe	0.258	0.932	0.533	0.945	0.291	0.828	0.840	0.889
BaA/BaA+Ch	0.314	0.767	0.282	0.459	0.386	0.535	0.465	0.725
IcdP/IcdP+BghiP	0.226	1.000	0.222	1.000	0.194	0.250	0.200	1.000
Phe/Ant	2.881	0.072	0.875	0.058	2.438	0.207	0.189	0.125

The concentration of BaP in the samples was highest in Shasha market (2.66  $\mu\text{g/kg}$ ) followed by Benin motor park (2.65  $\mu\text{g/kg}$ ) however, North gate (0.64  $\mu\text{g/kg}$ ) and Ilesha motor park (0.69  $\mu\text{g/kg}$ ) were considerably lower.

### 3.2 Source Diagnostics

The release of PAHs in the environment from non-natural sources can be attributed to petrogenic and pyrogenic origins. Petrogenic PAHs are usually 2 and 3 ringed while pyrogenic PAHs have 4 rings and above [22]. A Flu/(Flu+Pyr) ratio of  $< 0.4$  suggests majorly petroleum contamination, while Flu/Flu+Pyr  $> 0.5$  suggests PAHs from combustion of grass, wood, and coal, and  $0.4 < \text{Flu/Flu+Pyr} > 0.5$  from combustion of petroleum [23,24,25]. Ant/(Ant+Phe) ratios  $< 0.1$  are indicate PAHs from petroleum contamination, while Ant/Ant+Phe ratios  $> 0.1$  indicates the PAHs are from combustion sources [24,26]. BaA/BaA + Chr of 0.35 and Icdp/(Icdp + Bghip) of 0.5 is the boundary point between petroleum and combustion sources. However, BaA/(BaA + Chr)  $< 0.2$ , between 0.2–0.35 and  $> 0.35$  means the PAHs are majorly from petroleum input, inputs

from both sources and combustion input, respectively. Icdp/(Icdp + Bghip)  $< 0.2$ , between 0.2–0.5 and  $> 0.5$  implies petroleum input, liquid fossil fuel combustion and grass, wood and coal combustion, respectively [27,28]. Phe/Ant of 3 infers motor vehicle exhaust [29]. The Flu/(Flu+Pyr) ratio ranged from 0.611-0.803 and 0.125-0.699 (Table 1) for samples and control respectively and this indicates PAHs from combustion of grass, wood, and coal. The Ant/(Ant+Phe) ratios ranged from 0.258-0.840 and 0.828-0.945 (Table 1) for samples and control respectively and this implies that contamination in the samples and control was from combustion sources. The (BaA/(BaA + Chr)) ranged from 0.282-0.465 and 0.459-0.767 (Table 1) for samples and control respectively and this implies that contamination was from combustion input majorly. The Icdp/(Icdp + Bghip) ranged from 0.194-0.226 and 0.250-1.00 (Table 1) for samples and control respectively and this implies that contamination in the samples was from petroleum input and liquid fossil fuel combustion and for control samples was from grass, wood and coal combustion. The Phe/Ant ratio ranged from 0.189-2.881 and 0.056-0.267 (Table 1) for samples and control

respectively. This infers that motor vehicle exhaust caused contamination in samples. The above data indicated that the PAHs in study areas were mainly derived from combustion (e.g. petroleum, biomass, coal).

### 3.3 Correlation between Polycyclic Aromatic Hydrocarbons (PAHs) and Total Organic Carbon (% TOC)

Although some researchers have reported a linear relationship between the concentration of PAHs and TOC in soils, several factors including organic matter composition and temperature have been reported to influence the concentration of PAHs found in soils [30]. The nonlinear relationship between the %TOC and  $\Sigma$ PAH (Table 2) concentrations in the soils infers that the PAHs were recently generated and therefore were yet to fully form a stable PAHs – organic matter matrix. However, if the PAHs in the soils may have fully integrated into the soil matrix, a good correlation can be expected [25]. The correlation analysis ( $R^2 = 0.4157$ ) indicated that there was no correlation between the PAHs and TOC. Katsoyiannis [31] reported that in an environment where there is continuous input of

fresh contamination, lack of correlation should be expected, at least until equilibrium is reached. The lack of correlations between PAHs and TOC was also reported in previous studies [22,32,33,34].

### 3.4 Principal Component Analysis (PCA)

For further investigations on PAHs which causes major variations, PCA was performed on the PAH concentrations of individual sites. PCA (Fig. 2) revealed that the first principal component PC 1 accounted for 84%, PC 2 accounted for 10%, PC 3 accounted for 4% and PC 4 accounted for 1% of the total variances.

PC 1 (Fig. 3) was characterized by naphthalene, acenaphthylene, fluorene, phenanthrene, fluoranthene, pyrene, benzo (a) anthracene, chrysene, benzo (b) fluoranthene, benzo (k) fluoranthene, benzo (a) pyrene, dibenzo (a,h) anthracene and benzo (g,h,i) perylene.

PC 2 (Fig. 4) was characterized by naphthalene, acenaphthene, benzo (k) fluoranthene, benzo (a) pyrene and indeno {1,2,3-cd} pyrene.

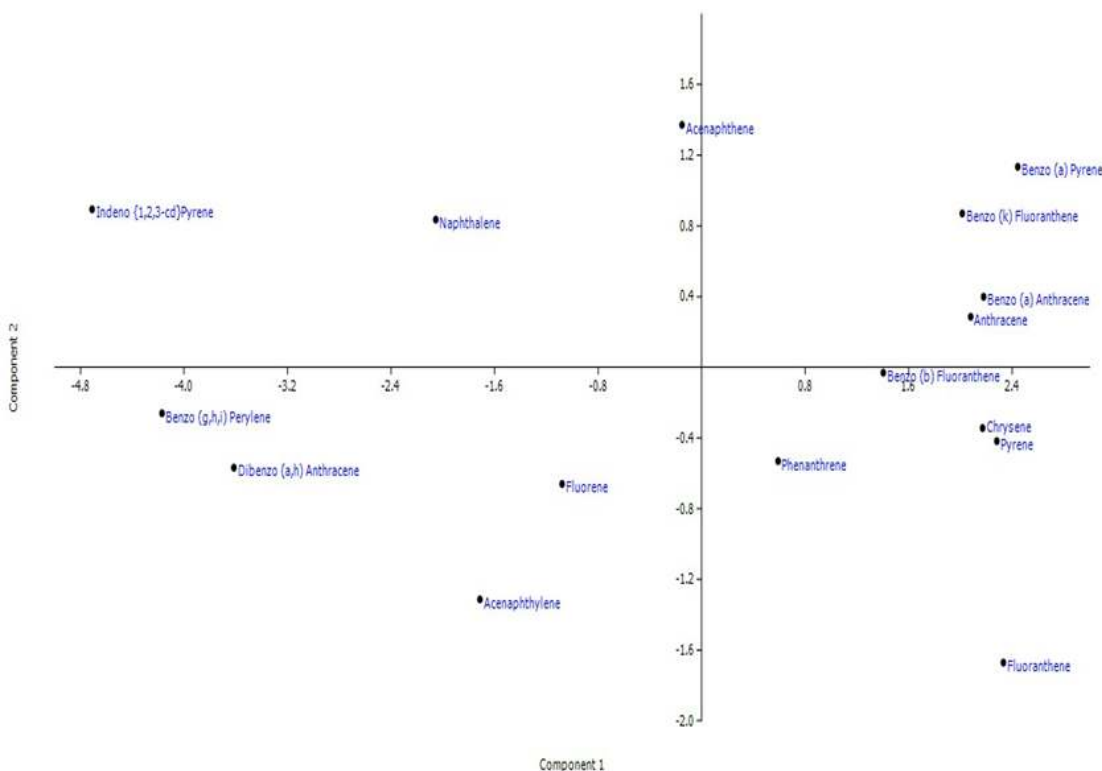
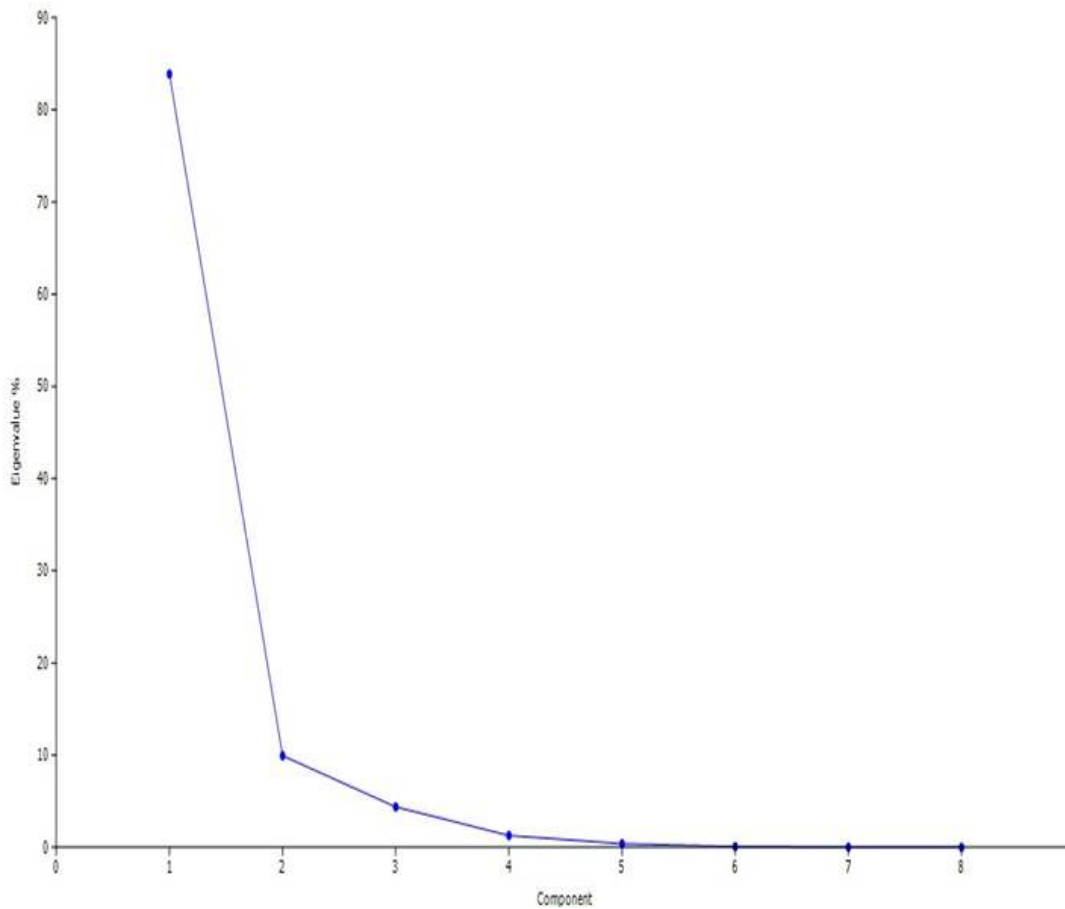


Fig. 2. Plots with PC1 and PC2 from principal component analysis

**Table 2.  $\Sigma$ PAHs and %TOC of sample areas**

Sample code	SSC	SSS	IGC	IGS	NGC	NGS	BGC	BGS
$\Sigma$ PAHs	13.766	27.046	1.511	16.962	1.778	6.184	5.877	25.625
%TOC	2.71	2.78	2.63	2.02	1.4	1.28	0.45	3.35

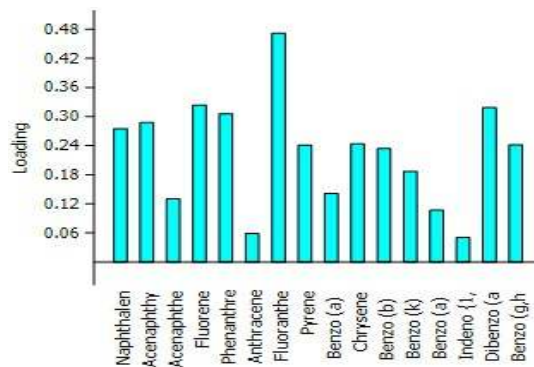


**Fig. 3. Screen plot**

Fluoranthene, pyrene, benzo (a) anthracene, chrysene, benzo (b) fluoranthene, benzo (k) fluoranthene and benzo (a) pyrene are typical markers for pyrolysis or incomplete combustion. Fluoranthene, benzo (a) anthracene and chrysene are markers for coal combustion [35,36,37]. Benzo (b) fluoranthene and benzo (k) fluoranthene are components of fossil fuels and a portion of them is associated with their combustion. Benzo (a) anthracene and chrysene are often resulted from the combustion of both diesel and natural gas. Benzo (k) Fluoranthene is an indication of diesel powered vehicles [22].

The scatter plot for PC 1 and PC 2 is presented in Fig 1. The screen plot obtained from PCA is presented in Fig 2. Factor loadings for PC 1 and

PC 2 are presented in Fig. 3 and Fig. 4 respectively.



**Fig. 4. Factor loading for PC 1**



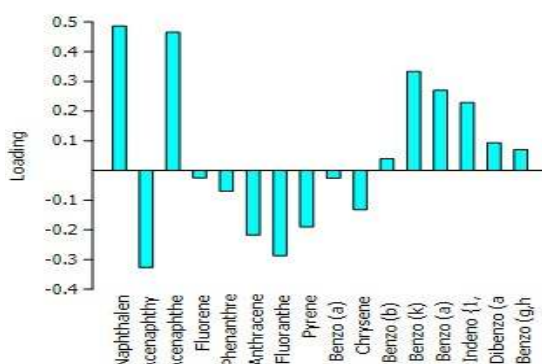


Fig. 5. Factor loading for PC 2

#### 4. CONCLUSION

This research showed that the polycyclic aromatic hydrocarbons in the site samples were all significantly higher than the control samples. Thus, the study areas were all polluted as a result of anthropogenic activities. Although the concentrations of polycyclic aromatic hydrocarbons were higher in site samples the concentrations varied according to activities ongoing in the sample areas. Areas with very high activities had higher concentrations. Shasha market sample had the highest concentration of PAHs while North gate sample had the lowest concentration of PAHs. North gate control had the lowest concentration among the control samples while Shasha market control had the highest concentration. North gate is an upcoming area while Shasha market area is already established and has numerous activities going on daily.

#### COMPETING INTERESTS

Authors have declared that no competing interests exist.

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