

## Distribution Ratios of Polycyclic Aromatic Hydrocarbons (PAHs) in Urban Soils

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### Authors' contributions

This work was carried out in collaboration between both authors. Authors MHES and JBS designed the study, managed the experimental process wrote the protocol and wrote the first draft of the manuscript. Author JBS managed the literature searches, wrote and reviewed the manuscript draft. Author MHES prepared sample analyses of the study, performed by GCMSTQD analysis and identified the distribution of PAHs. Both authors read and approved the final manuscript.

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### ABSTRACT

Distribution ratios and sources of polycyclic aromatic hydrocarbons (PAHs), including Fluoranthene (Flt), Pyrene (Pyr), Benzo(a)pyrene (BaP), Benzo(k)fluoranthene (BkP), Indeno(1,2,3-cd)pyrene (IP), Benzo(ghi)perylene (BgP) in urban surface and subsurface soil collected from Riyadh city, Saudi Arabia was studied. The sum of the six PAHs ranged from 30.5-1016.6 ng g<sup>-1</sup> with an average of 286.6 in surface samples and from 25.5-1501.7 ng g<sup>-1</sup> with an average of 287.9 ng g<sup>-1</sup> in subsurface samples. The magnitude of the PAHs was found in the following order: Pyr > Flt > BgP > IP > BkF > BaP. The ratios of benzo(a)pyrene to benzo(ghi)perylene (BaP/BghiP) indicated that diesel and gasoline vehicular exhausts were the predominant local emission sources of PAHs. The maximum values of Ind/(Ind+BgP) and Flt/(Flt+Pyr) indicated predominantly that the sources of these PAHs are pyrogenic sources due to combustion. It was also observed that soil samples of industrial areas possess the highest PAHs concentrations, and that the PAHs concentrations decreased with increasing distance from Riyadh city.

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## 1. INTRODUCTION

Soil acts as a sink for many contaminants, including polycyclic aromatic hydrocarbons (PAHs). The persistence of these compounds varies based on the source, distribution and localized soil environment. Therefore, it becomes of first importance to detect these compounds in the soils, assess the extent of contamination and make recommendations based on the findings. The assessment of soils becomes particularly important in the Kingdom of Saudi Arabia. The desert environment in this area is impacted by dust storms and increased amounts of suspended particulate matter in the air. There have been established reports of the occurrence of PAHs in the dust in such environments.

A number of PAHs listed as priority pollutants by the United States Environment Protection Agency (USEPA) and the European Union (EU) PAHs are wide-spread environmental pollutants and have been detected in surface waters, sediments, soils, plants, and both rural and urban air worldwide [1-3]. PAHs can occur naturally, but their origin is primarily attributed to anthropogenic processes, such as the burning of fossil fuels and other organic substances [4-6]. The major toxic effects of PAHs include cancers in various tissues, immunity suppression, loss of fertility, and cardiovascular diseases. Among these effects, the carcinogenicity of PAHs has been well-examined and accepted. Some PAHs listed by the U.S. Environmental Protection Agency (EPA) as priority pollutants are reported to be photo-mutagenic [7]. The PAHs taken up in skin can photosensitize under sunlight and, coupled with the genotoxic effects of UV in sunlight, could potentially be carcinogenic; this has also been clearly demonstrated *in vivo* [8-10]. Thus, when coupled with the dust and increased particulate matter in the arid and desert environment, PAHs could be a serious health concern. Moreover, there are reports from the region and across the world of the presence of PAHs in suspended particulate matter [11-13]. An increasing number of epidemiological studies, together with the US EPA's recent review, suggest that the suspended particulate matter in the atmosphere is leading to a rise in premature mortality [14-16]. Due to the omnipresence of these contaminants, the potential for them to enter the food chain is high. There have been reports from across the world, including the Kingdom of Saudi Arabia, revealing that PAHs

were detected in fish due to the contamination of the Arabian Gulf with oil spills [17,18]. The frontier of toxicological research on the presence of PAHs and other types of organic pollutants has been expanded by the availability and sophistication of modern analytical instrumentation. PAHs were detected in human breast milk in a number of studies, indicating the risk to mothers and breast-fed neonates [19-22].

Although environmental pollutants can originate from various sources, the soil is a major sink for many environmental pollutants, including PAHs. PAHs are introduced into the soil from atmospheric deposition after both local and long-range transport, causing accumulation in soils even in remote places that are far from point sources, such as industrial sites [23]. Other potential sources of soil contamination by PAHs include disposal from public sewage treatment plants, leachate from coal storage sites, and the use of soil compost and fertilizers [24,25]. Domestic use of fuel for cooking has also been shown to be another source of PAHs, and therefore, PAHs have been detected in food and can thus enter the food chain [26]. Urbanization and the increased combustion of organic material have a direct impact on the generation of atmospheric pollutants; therefore, there are specific guidelines by organizations such as the United Nations Economic Commission for Europe and the United Nations Environment Programme regarding the monitoring of PAHs [27,28]. In the Saudi Arabian context, this study on PAHs is one of the few first initiatives to monitor and estimate the contaminations of PAHs. Saudi Arabia has fast become an industrialized country, and with the undertaking of industrialization and the corresponding connection to the production of PAHs, the contamination rate could be high. If a direct relationship between the emission of greenhouse gases (GHG) and the production of PAHs is assumed, the potential presence of PAHs in the Saudi environment is high. It has been stated that the emission of greenhouse gases in the country has various causes. The generation of electricity using hydrocarbon sources such as petroleum has been indicted as one of the major sources of GHG emissions in Saudi Arabia. The emissions due to electricity generation are equal to those from the automotive industry. Electricity generation, road transport, desalination, petroleum refining, cement production, the

cement industry, the petrochemical industry, aviation and the production of iron and steel correspond to 26, 25, 15, 10, 5, 3, 3, 3, and 2% of the total greenhouse gas emissions, respectively [29].

El-Mubarak et al. [30] reported that the ambient air of Riyadh contained PAHs at concentrations from 1.383 to 13.470 ng m<sup>-3</sup>. The PAHs found are characteristic for emissions from traffic with diesel being a predominant source.

Analyses of fine soil particles from the Riyadh metropolitan area show that both anthropogenic and natural biogenic sources contribute to their organic matter contents. Anthropogenic sources are characterized by the presence of n-alkanes with hopane and sterane biomarkers, and unresolved complex mixture (UCM) from urban traffic emissions and plasticizers from plastic litter and detritus (e.g., shopping bags). The anthropogenic sources comprise between 70–92% in late winter and 64–96% in late summer of the total extractable hydrocarbons. The abundances of anthropogenic organic compounds in the city depend on the location, season, and types of urban activities. They are at relatively low levels in the rural samples. The natural sources of organic compounds are primarily from higher plant wax (i.e., vegetation) and amount to 1–13% in late winter and 0.0–18% in late summer of the total hydrocarbons of the soils and sands [31,32].

The influence of different UV wavelengths irradiation on photolysis of PAHs was investigated on sandy soil under two wavelengths (254 and 306 nm) UV irradiation for six PAHs. In addition, kinetic model and influence of several parameters on PAHs photolysis have been studied. The results obtained indicated that UV radiation with a wavelength of 306 nm was more efficient in the photolysis of the polycyclic aromatic hydrocarbons. Our results showed that fluoranthene (Flt) was the fastest in decomposition, had the greatest value for the coefficient of photolysis ( $7.4 \times 10^{-3} \text{ h}^{-1}$ ), and had less half-life, reaching 94 h when using a wavelength of 254 nm. The results indicated that the pyrene (Pyr) was more resistant to photolysis in comparison with endeno(1,2,3-cd) pyrene (IP) and fluoranthene (Flt). The results indicate that photolysis is a successful way to remediate the six studied PAHs compounds [32,33].

## 2. MATERIALS AND METHODS

### 2.1 Soil Sampling and Preparation

Overall Riyadh city, 448 soil samples of 224 locations, covering the urban and industrial areas, were collected at surface (0-5 cm) and subsurface (5-10 cm) layers (Fig. 1). Soil samples were collected by using core and soil cylinders made from stainless steel. Approximately 2 kg of each soil sample was collected from each sampling site. After collection, the soil samples were immediately transported to the laboratory in glass containers with a minimum headspace of air.

### 2.2 Analytical Methods

A 21 g  $\pm 0.01$  soil sample was weighed on an analytical balance in a 100 mL beaker, and was mixed with diatomaceous earth and 2 gm of sodium sulfate anhydrous to absorb moisture from the sample. The sample mixture was transferred to a 35 mL stainless steel Accelerated Solvent Extractor (ASE) cell (Dionex, Sunnyvale, CA, USA). Each ASE cell was pre-cleaned by rinsing with GC grade acetone and dichloromethane mixture, while diatomaceous earth was baked overnight at 460°C for 12 hrs. The cells were capped, loaded in the ASE and extracted with 40 mL GC grade acetone/dichloromethane set in the method as 1 part of each in the ASE. Extraction was done at a temperature of 100°C and pressure of 1500 psi. The duration of 1 cycle was 5 min and 60% of solvent mature flush volume. The extract collected in the ASE vial was transferred to a concentration flask and concentrated to 0.5-1 mL in a rotary evaporator (Buchi, Germany).

Silica SPE barrels (cartridges) were prepared and conditioned with 5 mL methanol, dichloromethane, and hexane. The extract was fractionated in the silica SPE by elution with 15 mL hexane followed by 10 mL dichloromethane. The collected fractions were concentrated with a stream of nitrogen gas to dryness and transferred quantitatively to a GC vial, the volume was adjusted to 1.0 mL using the same solvent used for fractionation and injected to the GC /MSMSTSQ8000 instrument.

Analyte separation, detection, and identification was performed by GC-MSMS TRACE 1310 gas chromatograph coupled to a TSQ8000, triple quadrupole mass spectrometer (Thermo Fisher

Scientific, Waltham, MA, USA). The instrument had an "SSL Instant Connect" module and a TriPlus autosampler., DB-5MS column (30 m x 0.25 mm x 0.25  $\mu\text{m}$  film thickness). The inlet temperature was 270°C with splitless 2  $\mu\text{l}$  injection and the oven temperature was ramped from 50°C to 310°C (3 min) at a rate of 10°C/min then to 325°C (10 min) at a rate of 4°C/min. Target analytes were determined with selected-ion-monitoring (SIM) mode using electron impact (EI) with guard temperature set point 190°C and source 350°C. Specific target ions and 2-3 qualified ions of each analyte were used in the identification of PAHs. Quantitation data analysis was done for each analyte using the calibration method developed for this purpose.

### 2.3 Quality Control

The procedural blanks, spiked blanks, sample duplicates were routinely analyzed with field samples. Limits of detection (LOD) were calculated as at least 3 times the noise level of the chromatogram in blank sample. The procedure was checked for recovery efficiencies by analyzing uncontaminated soil spiked with PAH standards. Surrogate standards d8-Nap, d10-Ace, d10-Phe, d12-Pyr, d12-Chr, d12-Per, d12-DBA and d14-DBaP) were added to all soil samples to monitor the efficiency of sample extraction, cleanup and analysis in every sample. The average recoveries of surrogate standards added to soil samples were calculated (Desired recovery range is 90% - 105%).

## 3. RESULTS AND DISCUSSION

### 3.1 Levels and Distribution of PAHs in Urban Soil Samples

The minimum, maximum and average concentrations of the six PAHs in urban soil samples are shown in Table 1. The sum of 6 PAHs ranged from 30.5-1016.6 with an average of 286.6 in surface samples and from 25.5-1501.7  $\text{ng g}^{-1}$  with an average of 287.9  $\text{ng g}^{-1}$  in subsurface samples (Fig. 2). Among various investigated PAHs, pyrene was found to be present at the highest concentrations (87.95  $\text{ng g}^{-1}$ ) at surface and (75.97  $\text{ng g}^{-1}$ ) at subsurface layers while benzo(a)pyrene recorded the lowest concentration (10.64  $\text{ng g}^{-1}$  at surface layer and 17.91  $\text{ng g}^{-1}$  at subsurface layer). The average percentages of Flt, Pyr, BaP, BkF, IP and BgP

were 28.7-29.9%, 31.1-33.7%, 3.68-4.98%, 5.37-5.72%, 7.48-7.68%, and 21.1-21.8%, respectively in the collected soil samples (Fig. 3). The amounts of PAHs were found in the following order: Pyr > Flt > BgP > IP > BkF > BaP. It has been reported that Flt and Pyr could be originated from incinerators, combustion, oil burning and petrol powered vehicles [34-36,33].

The obtained maximum concentrations of the investigated PAHs in surface and subsurface soil samples compared with the results of other countries found in the literature varied. While the concentration of total PAHs in this study was lower than what was published to be found in USA (58680  $\mu\text{g kg}^{-1}$ ), as indicated by [37], the maximum concentrations of PAHs were higher than those found in urban soil samples in Delhi, India; Tokushima, Japan, Hong Kong, China; Kota Bharu, Malaysia; Sanghai, China; Valasske, Czeck Republic [38-42,34].

Interim sediment quality guidelines (ISQGs) and probable effect levels (PELs) for PAHs in comparison with the obtained data from this study are presented in Table 2. The results indicated that the maximum measured concentrations of Fluoranthene and Pyrene exceed the guideline values of ISQGs and PELs. Additionally, the maximum measured concentrations of Benzo(k)fluoranthene, Benzo(g,h,i)perylene and Indeno(1,2,3-cd)pyrene exceed the guideline values of threshold effect concentration (TEC).

It was observed that soil samples of industrial areas possess the highest PAHs concentrations, and the PAHs concentrations decreased with increasing distance from the Riyadh city (Fig. 4), mainly due to motor vehicle exhausts and industrial activities.

### 3.2 Pollution Source

The anthropogenic release of PAHs can be attributed to petrogenic and pyrogenic origins [41]. The ratios of selected PAHs are generally reported to be a good index for identifying pollution source. The ratios of benzo(a)pyrene to benzo(g,h,i)perylene (BaP/BghiP) indicated that diesel and gasoline vehicular exhausts were the predominant local emission sources of PAHs. Our results showed that the ratios of benzo(a)pyrene to benzo(g,h,i)perylene (BaP/BghiP) ranged from 0.06 to 27.2 with an average of 4.24 in surface soil samples and

ranged from 0.02 to 34.2 with an average of 4.0 (Table 3), indicating that the source of pollution in most soil samples having a ratio of  $> 0.6$  might be originated from traffic sources. Additionally, the ratios  $Ind/(Ind+BghiP)$  and  $Flt/(Flt+Pyr)$  are mainly used to distinguish between pyrogenic and petrogenic sources. The value of  $Ind/(Ind+BghiP) > 0.5$  indicates combustion sources. The values of  $Flt/(Flt+Pyr) > 0.4$

indicates pyrogenic sources. The values of  $Ind/(Ind+BghiP)$  ranged from 0.01 to 0.85 in surface samples and from 0.01 to 0.76 in subsurface layer. Additionally, these values of  $Flt/(Flt+Pyr)$  are in the range of 0.003-0.99 in surface layer and of 0.17-0.99 in subsurface layer. The maximum values indicate predominantly pyrogenic sources and combustion.

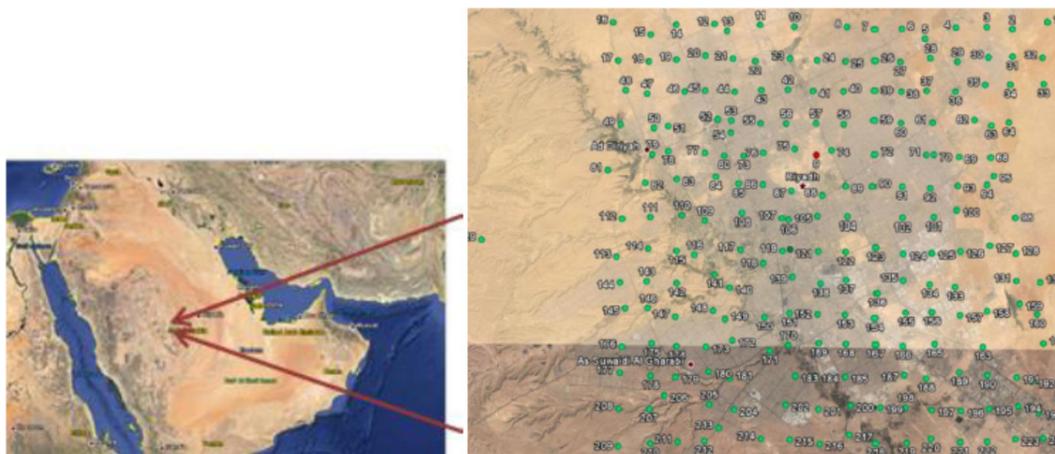


Fig. 1. Urban soil samples locations map in Riyadh City, and location of the remote soil sample, central of Saudi Arabia

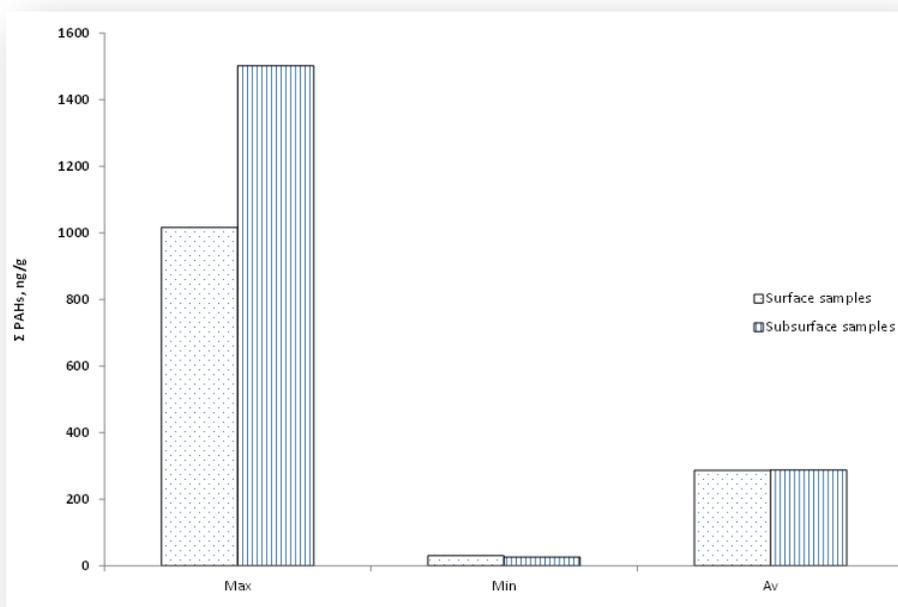


Fig. 2. Maximum (Max), minimum (Min) and average (Av) concentrations of  $\Sigma PAHs$

**Table 1. PAHs concentration (ng g<sup>-1</sup>) Maximum, minimum, and average in urban soil in Riyadh city**

PAHs Compounds	Surface 0-5 cm			Subsurface 5-10 cm		
	Max	Min	Average	Max	Min	Average
Fluoranthene Flt	129.9	2	75.52	172.3	2	70.69
Pyrene Pyr	189.9	2	87.95	211.9	2	75.97
Benzo(a)pyrene BaP,	112.6	3.7	10.64	112.6	3.9	17.91
Benzo(k)fluoranthene BkF	166	2	18.07	185.2	2	25.42
Indeno(1,2,3-cd)pyrene IP	311.4	4.4	22.11	559.9	3.6	30.60
Benzo (g,h,i) perylene BgP	373.9	2	74.64	371.4	10.3	68.22

**Table 2. Interim sediment quality guidelines (ISQGs) and probable effect levels (PELs) for PAHs (µg·kg<sup>-1</sup>dw)**

PAHs	Canadian Marine and estuarine sediments (µg·kg <sup>-1</sup> dw)		TEC threshold effect concentration (TEC)	WDNR USA Sediment ug/kg dry	USEPA Soil (ng/g)	In this study	
	ISQG Canadian interim sediment quality guidelines (ISQGs)	PEL Probable effects levels (PELs)		MEC midpoint effect concentration (MEC)			PEC probable effect concentration (PEC)
Fluoranthene	113	1494	423	1,327	2,230	2300	2-129.9
Pyrene	153	1398	195	858	1,520	1700	2-189.9
Benzo(a)pyrene	88.8	763	150	800	1,450	0.015	3.7-112.6
Benzo(k)fluoranthene	NA	NA	240	6,820	13,400	1.5	2-166
Benzo(g,h,i)perylene	NA	NA	170	1,685	3,200	NA	2-373.9
Indeno(1,2,3-cd)pyrene	NA	NA	200	1,700	3,200	NA	4.4-311.4

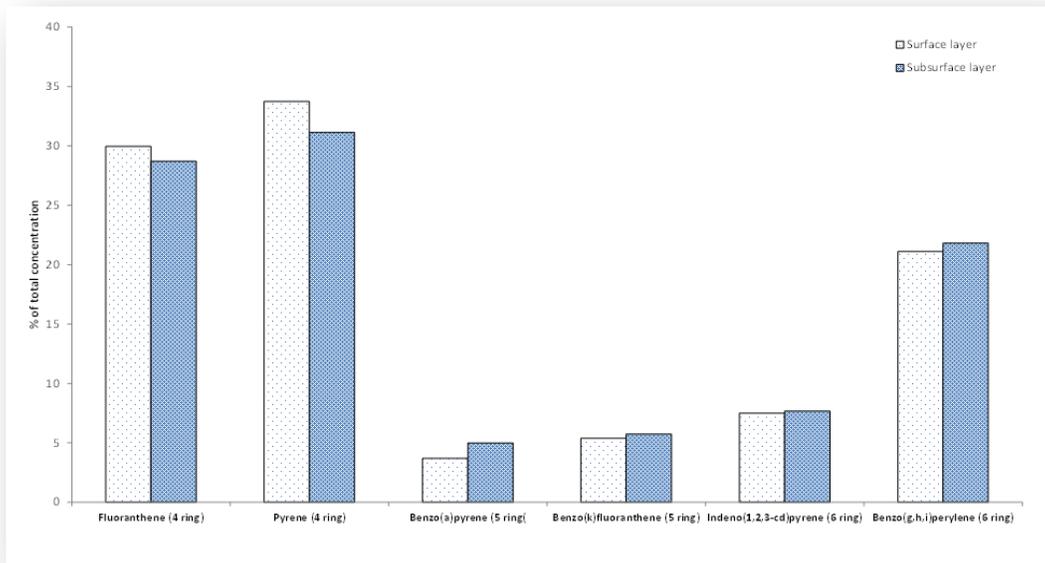


Fig. 3. Percentages of total PAHs content in tested soil samples in Riyadh city

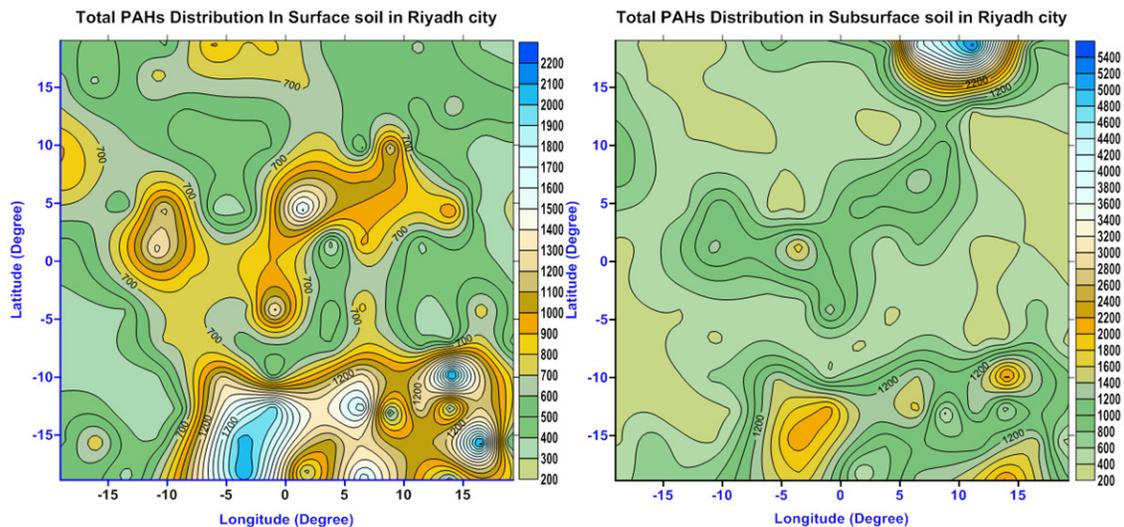


Fig. 4. Total PAHs distribution in surface and subsurface soil in Riyadh city

Table 3. The ratios of BaP/BghiP, Ind/(Ind+BghiP) and Flt/(Flt+Pyr)

PAHs compounds	Surface 0-5 cm			Subsurface 5-10 cm		
	Max	Min	Average	Max	Min	Average
BaP/BghiP	27.2	0.06	4.2402	34.2	0.02	3.997061
Ind/(Ind+BghiP)	0.85	0.01	0.2817	0.76	0.01	0.28834
Flt/(Flt+Pyr)	0.99	0	0.4723	0.99	0.17	0.469407

#### 4. CONCLUSION

Levels, distribution and sources of 6 polycyclic aromatic hydrocarbons (PAHs) were analyzed in

surface and subsurface soil samples collected from Riyadh, Saudi Arabia. The total concentrations of 6 PAHs (RPAHs) ranged from 30.5-1016.6 with an average of 286.6 in surface

samples and from 25.5-1501.7 ng g<sup>-1</sup> with an average of 287.9 ng g<sup>-1</sup> in subsurface samples. The ratios of benzo(a)pyrene to benzo(g,h,i)perylene (BaP/BghiP) and indeno(1,2,3-cd) pyrene to benzo(g,h,i)perylene (IDP/BghiP) indicated that diesel and gasoline vehicular exhausts were the predominant local emission sources of PAHs. Additionally, soil samples of industrial areas possess the highest PAHs concentrations, and the PAHs concentrations decreased with increasing distance from the Riyadh city, mainly due to motor vehicle exhausts and industrial activities. Our results suggest that the remediation of contaminated soils with PAHs in Riyadh city should be taken into account.

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### COMPETING INTERESTS

Authors have declared that no competing interests exist.

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