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ASSESSMENT OF SOIL CONTAMINATION BY POLYCYCLIC AROMATIC HYDROCARBONS (PAHS) IN BUCHAREST: SOURCES AND DISTRIBUTION

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Abstract

Polycyclic aromatic hydrocarbons (PAHs) are organic pollutants composed of two or more aromatic rings of carbon and hydrogen atoms. PAHs are characterized by low water solubility, low vapor pressure, high melting and boiling points. As the molecular mass increases, the lipophilic character increases, making them more persistent in the environment. Incomplete combustion is the main source of PAHs. This study evaluates PAHs contamination in soil samples collected from 30 locations in Bucharest, focusing on potential pollution sources such as industrial activities, vehicular emissions, and domestic sources. Industrial zones and high-traffic areas recorded the highest total PAH concentrations at 1.06 mg/kg and 1.01 mg/kg, respectively. Diagnostic ratios analysis suggests combustion as the predominant source of PAHs.

Key words*: diagnostic ratios, PAHs, soil pollution, urban contamination.*

INTRODUCTION

Polycyclic aromatic hydrocarbons (PAHs) are widespread environmental pollutants consisting of two or more benzene rings arranged in linear, clustered, or angular formations (Arey & Atkinson, 2003). They have low solubility in water, low vapor pressure, and high melting and boiling points. As their molecular weight increases, their lipophilic nature intensifies, making them more resistant to degradation (Lee & Vu, 2010).

PAHs are found in terrestrial, aquatic, and atmospheric environments (Adeniji et al., 2019). In soils and sediments, PAHs accumulate due to their lipophilic properties, binding strongly to soil particles, which serve as reservoirs for these compounds (Kuppusamy et al., 2017).

Many PAHs are mutagenic, carcinogenic, teratogenic, immunotoxic for living organisms, including microorganisms, mammals and humans (Bolden et al., 2017). Benzo(a)pyrene is considered one of the most carcinogenic PAHs and generally used as an exposure marker for risk assessments (Lee & Vu, 2010).

In 1983, the US Environmental Protection Agency (USEPA) designated 16 polycyclic aromatic hydrocarbons (PAHs) as priority pollutants, due to their high environmental concentrations, their recalcitrant nature and toxicity (Deziel et al., 2014). PAHs can be divided into two groups according to their properties and molecular weights: low molecular weight (LMW-PAHs) which have two or three aromatic rings and high molecular weight (HMW-PAHs) with four to six aromatic rings (Jia et al., 2021).

LMW-PAHs typically form during incomplete combustion of organic matter, such as in vehicle exhaust, industrial emissions, and biomass burning. Their appearance is linked to low temperatures.

HMW-PAHs tend to form in more complex combustion conditions, at high temperature as in coal or oil burning, and they are found in higher concentrations in soot (Wang et al., 2013).

Sources of PAHs are classified as either anthropogenic or natural. Some PAHs are produced by natural processes such as volcanic eruptions, vegetation fires or pyrolysis of organic matter in geological processes.

While natural sources contribute minimally to overall PAH emissions, anthropogenic sources dominate. So, anthropogenic sources are considered the main cause of pollution with these compounds and can be divided into industrial, vehicular, domestic and agricultural activities (Ravindra et al., 2008).

The most common anthropogenic sources are:

- Industrial sources when incomplete combustion, the main source of PAHs pollution can appear:
  - waste incineration,
  - iron and steel production, aluminum production, cement production, tar, asphalt, rubber production;
  - coke oven emissions, and petroleum refining (Mojiri et al., 2019);
  - paint and pigment production;
  - insecticide and fungicide production;
  - energy production (Srogi, 2007).
- Vehicular sources represented by gases from aircraft, boats, trains, vehicles (Ravindra et al., 2008)
- Domestic sources: household activities such as burning waste, burning wood or coal for heating (Gupte et al., 2016).
- Agricultural sources: burning of plant residues left after harvesting (Ravindra et al., 2008).

The aim of this study is to assess PAHs contamination in Bucharest soils, identify major pollution sources, and analyse the impact of different environmental factors on PAH distribution.

MATERIALS AND METHODS

Soil sampling

A total of sixty topsoil samples were collected from depths of 0-10 cm and 10-20 cm across 30 locations in Bucharest and its surrounding areas, including Chitila, Jilava, and Popești-Leordeni. These sites were selected to target various pollution sources, such as industrial zones, high-traffic areas, and residential neighborhoods.

The industrial sites, represented by sampling points S1, S2, S10, S12-S19, S21, S23, S24, S26 and S27, cover a range of facilities including paint factories, concrete factories, incinerators, rubber manufacturing units, a power station, pharmaceutical plants, an emulsion station, as well as the IMGB and Faur-Laminorului industrial platforms.

High traffic areas are covered by sampling points S4, S5, S6, S8, S9, S11, S25 and S28, where vehicle emissions are a significant source of pollution. In addition, the Ruteni and Vidra landfill sites are designated as sampling points S3 and S22, respectively. Residential areas and urban green spaces were also considered, with sampling points S20 and S7 representing these environments.

All sampling sites were georeferenced using GPS for accurate spatial analysis (Figure 1).

Samples were collected in March when air temperatures averaged 15°C.

Each sample was a composite of five subsamples taken from the four corners and the centre of a $5 \times 5\ m^2$ plot.

The samples were placed in glass containers, shielded from light, and stored under refrigeration until analysis. In proximity to potential pollution sources (e.g., the Green Global Chitila waste dump, S1), additional samples were taken at distances of 200 m and 400 m from the source.

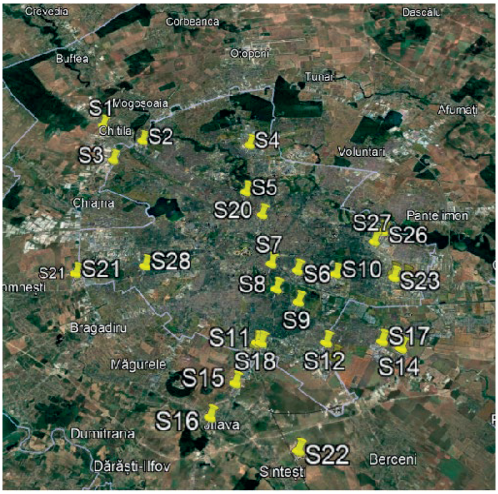

Figure 1. Map of the sampling points

Soil physicochemical properties

Soil properties were analysed using standard methods:

- pH: potentiometric method (SR-7184-13);
- Organic carbon (C, %): wet oxidation method (Walkley-Black, STAS 7184/21-82);
- Mobile Phosphorus (PAL, mg/kg): Egner-Riehm-Domingo method (STAS 7184/19-82);
- Electrical Conductivity (EC, μS/cm): aqueous extract and conductometric method (STAS 7184/7-87);

- Nitrate ($N\text{-}NO_3$, mg/kg): potentiometric method (ICPA methodology);
- Particle size distribution: wet/dry sieving and sedimentation (STAS 7184/7-87).

The soils studied are classified into the following textural classes: sand, sandy loam and loam (Table 1). The chemical properties of the studied soils are summarised in Table 2.

Table 1. Textural classes of the soil analysed in the study

Textural classes	Number of collection points
Sand	10
Sandy Loam	22
Loam	28

Table 2. Chemical properties of the analysed soil samples

Chemical properties of the analysed soil sample (N = 60)	Range
pH	4.98-8.56
C (%)	0.86-6.19
N (%)	0.09-0.499
EC (μS/cm)	23.3-1521
$N\text{-}NO_3$ (mg/kg)	2.68-65
P (mg/kg)	16.34-271.2

The analysed soils exhibit a broad spectrum of chemical properties, which is typical for urban soils (Preda et al., 2010). The pH varied from slightly acidic to alkaline, organic carbon levels ranged from extremely low to high, as did electrical conductivity (EC). Nitrate levels fluctuated between low and high, while phosphorus and nitrogen availability ranged from low to very high, respectively from low to high.

PAHs extraction from soil and analysis

PAHs are analysed from soil according to the standards set forth in the European Standards (SR EN 17503:2022). To extract these compounds from soil, an automated Soxhlet extractor and a mixture of hexane and acetone (in a 1:1 ratio) were employed. The extract is washed with water for chromatography to remove acetone and it is passed through anhydrous sodium sulphate. If necessary to remove the sulphur, copper powder can be used. The determination of PAHs was performed on a Knauer ultrahigh pressure liquid chromatograph (UHPLC) with UV-VIS detection, at 254 nm. The concentration for each compound were quantitatively determined by comparison the peak area of the standard with that of the samples.

In accordance with Order 756/1997, the following priority PAHs are to be quantified: anthracene, benzo(a)anthracene, benzo[b]fluoranthene, benzo[k]fluoranthene, benzo[a]pyrene, benzo[ghi]perylene, chrysene, fluoranthene, indeno(1,2,3)pyrene, naphthalene, and phenanthrene, as well as pyrene. The pair consisting of benzo[*ghi*]perylene and indeno(1,2,3)pyrene cannot be determined with UV-VIS detector. The separation of the other compounds was performed with a C18 column (2.1 mm x 100 mm x 1.8 μm) operated at 40°C. The mobile phase is composed of water and acetonitrile and the gradient of this mobile phase is presented in Table 3.

Table 3. Flow and gradient of the mobile phase

Time (minute)	Flow (ml/min)	Acetonitrile (%)	Water (%)
0	0.4	50	50
1.6	0.4	55	45
2.9	0.4	60	40
3.7	0.4	70	30
8.5	0.4	85	15
13	0.4	50	50
13	0.4	50	50

Quality assurance/Quality control

To ensure the accuracy and reliability of the results, quality control and quality assurance procedures included duplicate samples, blank samples, and certified reference materials. The limit of quantification for PAHs ranged from 0.02 mg/kg to 0.05 mg/kg. Reagents are of chromatographic grade. To minimize the contamination, all the glassware is rinsed with hexane, acetonitrile and acetone.

RESULTS AND DISCUSSIONS

PAHs concentrations and patterns in the analysed soil samples

The total concentration of the PAH compounds in soil ranged between 0.047 mg/kg and 1.060 mg/kg, with an average value of 0.212 mg/kg (Figure 2). According to Order 756/1997, 33.3% from analysed soil samples have normal concentration (<0.1 mg/kg), 66.7% have concentrations ten times higher than normal values. The highest PAHs concentrations were observed at S4 (Ficusului Blvd), a high-traffic area, and at S12 and S13 (IMGB), a well-known industrialized zone in Bucharest (Figure 2). Similar studies in other European cities have reported comparable PAHs concentrations, suggesting common urban pollution patterns. For example, the total PAHs content was reported to range from 0.148 mg/kg to 3.410 mg/kg in Torino, from 0.218 mg/kg to 4.490 mg/kg in Ljubljana (Morillo et al., 2007), and from 0.450 mg/kg to 5.650 mg/kg in the soil of the River Seine basin in Paris (Motelay-Massei et al., 2004). These results are significantly lower compared to those found in soils with specific pollution sources. For instance, near a tar works in the UK, the total concentration of PAHs ranged from 6.6 mg/kg to 872 mg/kg (Lorenzi et al., 2010). This suggests that the study data presented represents diffuse pollution sources.

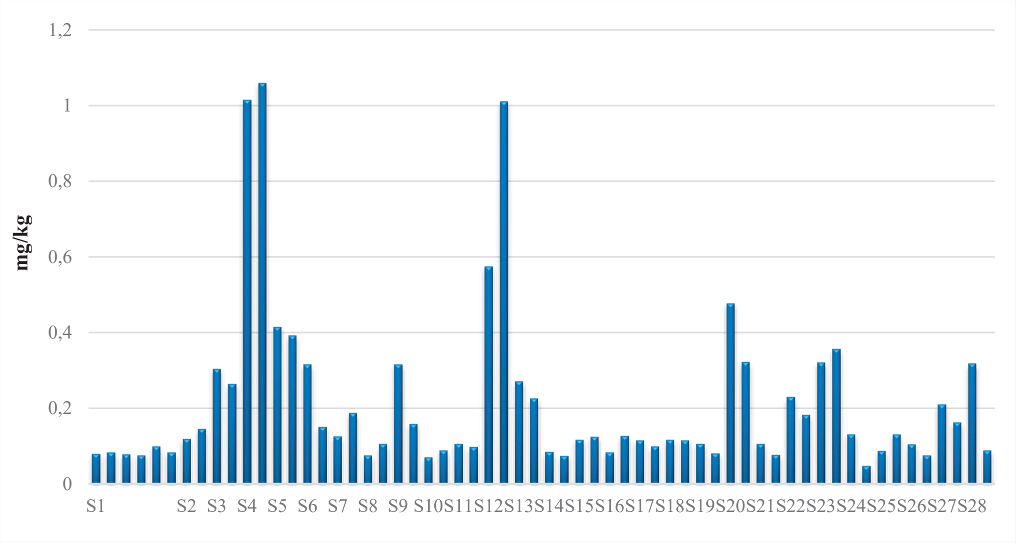

Figure 2. Total PAHs concentration in soil samples collected from Bucharest

The mean concentration of the individual PAH compounds increases in order: benzo(a)pyrene, benzo(k)fluoranthene, naphthalene, anthracene, phenanthrene, pyren, benzo(b)fluoranthen, benzo(a)anthracene, fluoranthen (Figure 3).

The highest concentrations were reported for fluoranthene, benzo(a)antracene and benzo(b)fluoranthene. Similar results were obtained by Wang (2013) in a study regarding the soil contamination with PAHs in Beijing.

The range of the concentration for the individual PAHs are presented in Table 4. The PAH distribution in Bucharest soils shows a minor contribution from LMW-PAHs (2-3 rings), such as naphthalene, phenanthrene, and anthracene. Instead, 4-ring polynuclear aromatic hydrocarbons (fluoranthene, pyrene, chrysene, and benzo(a)anthracene) are dominant (Figure 4). Among this group, fluoranthene and benzo(a)anthracene has the highest concentration.

Regarding 5-ring PAHs, benzo(b)fluoranthene has the highest contribution to the total PAH content.

Naphthalene is one of the most volatile PAHs, meaning it can evaporate relatively quickly from the soil surface. Naphthalene contaminates 46.7% from the analysed soil sample. The highest concentration was recorded in the soil sample collected from the S3 (Ruteni landfill). Naphthalene can be found in landfills due to its presence in various waste materials, including plastics, coal tar, petroleum products, and mothballs. Jia & Batterman (2010) also reported the presence of naphthalene in a landfill site.

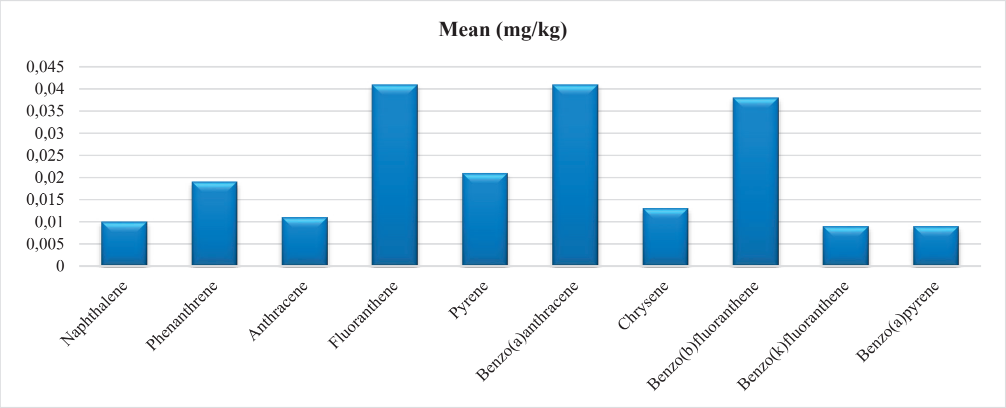

Figure 3. Concentration of the individual PAH compounds in the soil samples collected from Bucharest

Table 4. Range and mean value of concentration of the individual PAHs in studied soil samples

PAH compounds	Type of PAH	Range (mg/kg)		Mean (mg/kg)
		Min.	Max.	
Naphthalene	LMW	ND	0.104	0.010
Phenanthrene	LMW	0.004	0.072	0.019
Anthracene	LMW	ND	0.089	0.011
Fluoranthene	HMW	0.012	0.280	0.041
Pyren	HMW	ND	0.128	0.021
Benzo(a)anthracene	HMW	0.004	0.632	0.041
Chrysen	HMW	ND	0.054	0.013
Benzo(b)fluoranthene	HMW	ND	0.525	0.038
Benzo(k)fluoranthene	HMW	ND	0.054	0.009
Benzo(a)pyrene	HMW	ND	0.032	0.009

LMW = Low molecular weight
HMW = High molecular weight
ND = Not detection

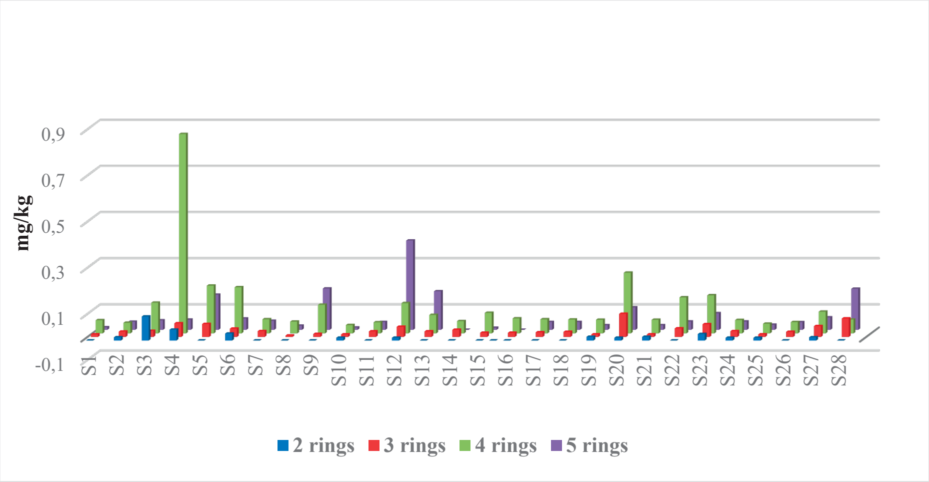

Figure 4. Profiles of the PAHs in the soil samples by number of rings

Phenanthrene is the most thermodynamically stable of the 3-ringed PAHs. This can be the reason it contaminates all the soil samples, even at small concentrations that slightly exceed the upper limit of the normal threshold (<0.05 mg/kg). Similar results were reported by Vane et al. (2014). The highest concentrations were identified in a boulevard with heavy traffic congestion, S28 (Prelungirea Ghencea).

In contrast, there is a major presence of HMW-PAHs (4-5 rings), including fluoranthene, pyrene, benzo[a]anthracene, chrysene, and particularly benzo[b]fluoranthene (Figure 4). This suggests a dominance of HMW-PAHs, likely indicating sources such as combustion processes rather than petrogenic inputs (McCready et al., 2000).

Fluoranthene, a 4 rings PAH contaminates all the soil samples. 80% from collected samples have concentrations that exceed the upper threshold of normal values (<0.02 mg/kg). Fluoranthene is relatively stable in soil, it strongly binds to soil particles, especially in organic-rich soils (Abdel-Shafy & Mansour, 2016). The highest concentration of fluoranthene was recorded in S4, once again in an area with heavy car traffic.

70% from soil samples are contaminated with pyrene, another 4 rings PAH. The highest concentration (0.128 mg/kg), which is considered a normal value according to Order 756/1997 (<0.5 mg/kg), was recorded in the IMGB industrial area. Pyrene is not the most toxic PAH, but it can act as a precursor to more harmful compounds like benzo[a]pyrene, which is carcinogenic (Gabriele et al., 2021).

Chrysene contaminates almost all soil samples, with 60% of them having normal concentrations (<0.02 mg/kg). The highest concentration (0.054 mg/kg) was recorded in S5 (Kiseleff Boulevard).

Benzo(a)anthracene has also 4 aromatic rings in the molecule and it can persist in soil for long periods due to its low volatility and stability. It can adhere strongly to soil particles, making it resistant to degradation. It is considered a possible human carcinogen (Group 2B by the International Agency for Research on Cancer, IARC) (Abdel-Shafy & Mansour, 2016). Long-term exposure can increase the risk of cancers, especially if the compound enters the human body through ingestion or inhalation. 63% from the studied soil samples have concentrations that exceed the upper threshold of normal values (<0.02 mg/kg). The highest concentrations (0.632 mg/kg) were obtained in sample collected from S4 (Ficusului Boulevard). This value exceeds the normal upper threshold (<0.02 mg/kg) but remains below the alert threshold (2 mg/kg).

From the 5-ring PAHs, benzo(b)fluoranthene, benzo(k)fluoranthene and benzo(a)pyrene were evaluated. The compound with a major contribution to the total content of PAH is benzo(b)fluoranthene. It is relatively persistent in the environment. It does not readily degrade in the presence of sunlight, air, or water, and can remain in soil or sediments for a long time. The highest concentration (0.535 mg/kg) was obtained in the sample collected from the IMGB area. This value exceeds the normal upper threshold (<0.02 mg/kg) but remains below the alert threshold (2 mg/kg).

The highest concentrations of benzo(k)fluoranthene and benzo(a)pyrene (0.054 mg/kg, respectively 0.032 mg/kg) were recorded in S5 (Kiseleff Boulevard). These concentrations slightly exceed the normal value (<0.02 mg/kg) according with Order 756/1997.

Effect of soil properties on the total PAHs concentration

PAHs are hydrophobic and have a high affinity for organic matter. Soils rich in organic matter tend to adsorb and retain PAHs more effectively, reducing their mobility and bioavailability (Yang et al., 2010). Higher organic carbon content is often correlated with higher PAH concentrations due to stronger sorption (Du et al., 2022). No correlation found in this study between the total content of PAHs in soil and organic carbon. A lack of a correlation is not an uncommon finding for soils (Ribes et al., 2003; Bucheli et al., 2004; Heywood et al., 2006) and has been attributed to a state of non-equilibrium. Low molecular weight PAHs (2-3 rings) associated with soot tend to partition and equilibrate more easily between the vapor phase and the soil organic matter, whereas high molecular weight PAHs (4-6 rings) remain more strongly bound to particles (Bucheli et al., 2004).

The influence of pH on PAHs content in soil is indirect, as PAHs are nonpolar compounds and do not ionize in response to pH changes. However, pH affects PAHs behaviour by influencing microbial degradation and their interactions with metal ions or organic matter.

Pawar (2015) was observed that soil with pH 7.5 was most suitable for the degradation of phenanthrene, anthracene, fluoranthene, and pyrene. Thus, on Theodor Pallady Boulevard, the pH is 7.54, the phenanthrene content is 0.011 mg/kg, anthracene is below the limit of quantification, and fluoranthene has the lowest concentration: 0.012 mg/kg.

Soils with high EC can adsorb more PAHs due to electrostatic interactions with organic matter. Studies show a positive correlation between PAH content in soil and conductivity, instead nitrate, which acts as an electron acceptor, has played a significant role in the oxidation of PAHs (Du et al., 2022).

In soil samples collected from Bucharest, no correlation can be established between PAHs and EC or nitrate, likely because the total concentration of PAHs is more influenced by pollution sources, which are highly diverse in Bucharest.

Identifying PAHs sources using isomer ratios

Diagnostic ratios are used to distinguish between pyrogenic (fire-related) and petrogenic (oil or fossil fuel-related) sources of polycyclic aromatic hydrocarbons.

The key point mentioned is that while these diagnostic ratios are useful, their application can be limited because there may be overlap in the ratios from both pyrogenic and petrogenic sources.

This overlap can make it challenging to pinpoint specific types of pyrogenic or petrogenic sources using these ratios alone. (Stout et al., 2004; Galarneau, 2008). In the real world, PAHs often originate from multiple sources, making it difficult to pinpoint one specific source. For instance, both vehicular emissions and industrial activities might contribute to contamination in the same area. This mixing complicates the identification and assessment of specific sources. Despite this, ratios are widely used and applied in various environments for source apportionment estimations of PAHs (Dickhut et al., 2000; Yunker et al., 2002; Tobiszewski & Namieśnik, 2012).

The following diagnostic ratios were used to indicate possible sources (Davis et al., 2019):

- ∑LMW/∑HMW PAHs;
- Anthracene/ (Anthracene + Phenanthrene);
- Fluoranthene/ (Fluoranthene + Pyrene);
- Benzo[*a*]anthracene/(Benzo[*a*]anthracene + Chrysene).

The ratio of LMW to HMW PAHs aims to distinguish between petrogenic and pyrogenic sources. Values < 1 suggest pyrogenic sources, while a value > 1 suggests petrogenic sources (Zhang et al., 2008). In this study the ratio ranged from 0.03 to 0.91, suggesting the pyrogenic sources may be most impactful in soil collected (Figure 5).

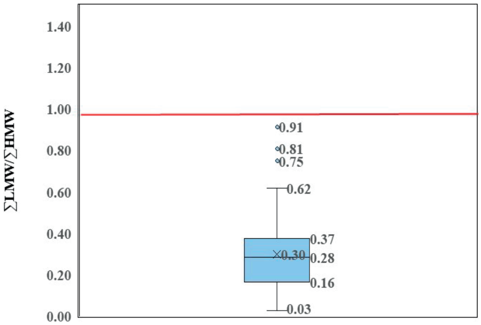

Figure 5. PAH diagnostic ratio plot of ∑LMW/∑HMW

The Anthracene/(Anthracene + Phenanthrene) ratio distinguishes between petrogenic and combustion sources, as values <0.1 indicate petrogenic and those > 0.1 indicate combustion (Yunker et al., 2002). Values obtained for this ratio ranged between 0.1 and 0.62. The value 0.1 is obtained in sample collected from S 28, Prelungirea Ghencea (Figure 6).

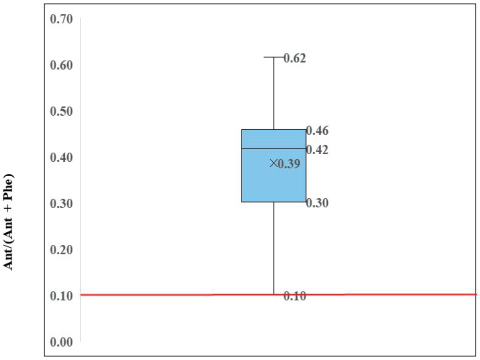

Figure 6. PAH diagnostic ratio plot of Ant/(Ant+Phe)

The Fluoranthene/(Fluoranthene + Pyrene) ratio provides valuable insight into distinguishing between petroleum sources, petroleum combustion, and other combustion processes. Ratio values < 0.4 indicate petrogenic sourcing, values between 0.4 and 0.5 are indicative of petroleum combustion, while values > 0.5 suggest wood, grass, and/or coal combustion (Yunker et al., 2002). This ratio is greater than 0.5 for all the soil samples collected from Bucharest (Figure 7).

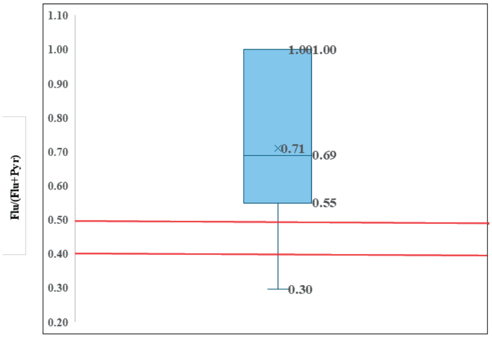

Figure 7. PAH diagnostic ratio plot of Flu/(Flu+Pyr)

The Benzo[*a*]anthracene/(Benzo[*a*]anthracene + Chrysene) ratio distinguishes between petrogenic and combustion (pyrogenic) sources, with a range indicating mixed sourcing. Values below 0.2 suggest petrogenic sources, values between 0.2 and 0.35 indicate mixed sources, while values above 0.35 point to combustion sources (Yunker et al., 2002). According to Figure 8, the same theory is confirmed once again, indicating that the source of PAH compounds is combustion.

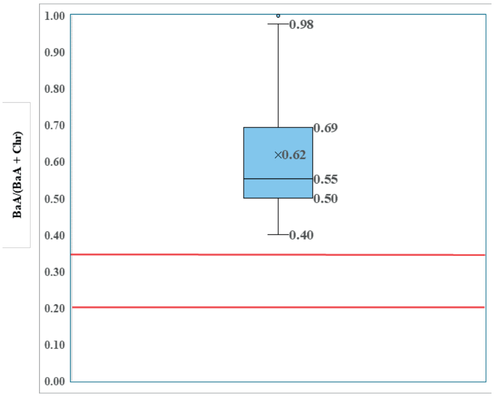

Figure 8. PAH diagnostic ratio plot of BaA/(BaA+Chr)

CONCLUSIONS

33.3% from analysed soil samples have normal concentration (<0.1 mg/kg), 66.7% have concentrations ten times higher than normal values.

The highest values of PAHs concentration were observed in S4 (Ficusului Blvd.) a very crowded boulevard and S12, S13 (IMGB), a well-known industrialized area from Bucharest.

The PAH distribution in Bucharest soils shows a minor contribution from LMW- PAHs (2–3 rings). Of these, phenanthrene is the contaminant present in all the analysed samples, even at low concentrations.

In contrast, there is a major presence of HMW-PAHs (4-5 rings), including fluoranthene, pyrene, benzo[a]anthracene, chrysene, and particularly benzo[b]fluoranthene.

The application of the three types of diagnostic ratios led to the same conclusion, namely that the source of PAHs pollution of the studied soil samples could be the combustion process as: vehicle exhaust (gasoline and diesel engines), residential heating (coal, wood, oil burning), industrial processes.

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