

IDENTIFICATION THE SOURCES OF PAHS IN CLUJ-NAPOCA'S CITY ATMOSPHERE USING MOSS AS BIOMONITORS

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ABSTRACT. This study aims to determine the concentrations of PAHs in Cluj-Napoca's city atmosphere using moss as biomonitors. Samples from different neighborhoods of Cluj-Napoca city were collected and analyzed by gas chromatography coupled with mass spectrometry. The concentrations of individual PAHs ranged from 0.61 $\mu\text{g kg}^{-1}$ (chrysene) to 353.6 $\mu\text{g kg}^{-1}$ (Indeno(1,2,3 -cd)pyrene), and the Σ PAHs ranged from 92.61 $\mu\text{g kg}^{-1}$ to 353.6 $\mu\text{g kg}^{-1}$ with a mean value of 329.23 $\mu\text{g kg}^{-1}$. Residential areas characterized by both low population density and traffic intensity had the lowest concentration levels of Σ PAHs in moss samples, below 200 $\mu\text{g kg}^{-1}$. Levels of concentration between 200 and 300 $\mu\text{g kg}^{-1}$ were obtain in similar residential areas, but with more intense traffic. The locations that had PAHs concentration levels in moss exceeding 300 $\mu\text{g kg}^{-1}$ are either industrial areas or neighborhoods with intense traffic. In all the study's locations the main emission source, according to PAHs diagnostic ratios, is pyrogenic, mainly originating from traffic emissions. The FL/ (FL+PYR) and FLA/ (FLA+ PYR) ratios indicate that in some cases there also is a petrogenic source. In a cross examination however, it can easily be seen that the main apportion for all the location is from pyrogenic sources.

Key words: Polycyclic aromatic hydrocarbons, urban atmosphere, moss, biomonitors, Cluj-Napoca

INTRODUCTION

Air quality is generally monitored with physicochemical detectors that offer quantitative data, however these are limited by analytical difficulties (such as detection limit), by costs and maintenance equipment (Marć et al., 2015), and the measurements provide only the concentration levels that are present in the environment at the moment of sampling (Wu et al., 2014). On opposite, biomonitoring is easier to apply, has no need for electrical energy, is cheaper and can provide a larger number of samples for a larger number of pollutants, and can provide a better coverage (Iodice et al., 2016; Vukovi et al., 2015, Marć et al., 2015; Ares et al., 2012). Yet, the lack of standardized

procedure prevents the usage of this technique on a large scale (Marć et al., 2015; Ares et al., 2012).

The biomonitors are defined as the living organisms that are used with the purpose to obtain quantitative and qualitative information regarding a certain aspect of the environment (Forbes et al., 2015). Mosses are capable to quickly absorb pollutants due to the fact that they rely on air for the most important nutrients uptake because of the lack of reticular system. In addition, the lack of waxy cuticle allows the direct contact of the foliar cells with the pollutants (Wu et al., 2014), and the poorly developed vascular system and the large exposed surface ratio compared with the total mass allow a better assimilation of atmospheric polycyclic aromatic hydrocarbons (PAHs) (Forbes et al., 2015, Thomas and Simon, 1985). Mosses are often used in biomonitoring studies because they are isolated from the ground, and so, they are less exposed to contamination with solid particles (Forbes et al., 2015; Liu et al., 2005), can be found all over the world, and their slow growth allows the study of contaminants accumulation over a long period of time (Vuković et al., 2015; Wu et al., 2014).

The moss usefulness in air quality assessment was promoted by many scientists in a large variety of studies (Capozzi et al., 2016a; Lazić et al., 2016; Iodice et al., 2016; Ares et al., 2009, Liu et al., 2005; Viskari et al., 1997; Wang et al., 2009, Wegener et al., 1992, Thomas et al., 1984, Wu et al., 2014, Thomas, 1986, Holoubek et al., 2000). Of course, not only PAHs were studied, but also many other chemical contaminants with negative health effects, such as, persistent organic pollutants (POP): polychlorinated biphenyls - PCB, dioxins and furans - PCDD/F, or polybrominated diphenyl ethers - PBDE (Harmens et al., 2013), heavy metals (Capozzi et al., 2016b; Iodice et al., 2016, Wegener et al., 1992), radioactive compounds (Capozzi et al., 2016a), nitrogen oxides, carbon monoxide, volatile organic compounds (VOC), particulate matter (Iodice et al., 2016) etc.

PAHs are a family of chemical compounds made from hydrogen and carbon atoms that forming at least two fused aromatic nuclei links. After the emission, volatile PAHs remain in the gas phase, while less volatile PAHs (with five or six nuclei) are adsorbed on solid particles in the atmosphere. The deposit in the vegetal material occurs from the absorption of lipophilic compounds both in gas phase and in solid phase (Harmens et al., 2013). Due to their toxic effects on human health PAHs concentration in the environment are regulated by the European Union (Directive 2004/107/CE).

In Romania only two studies were made on pollutants concentration from air that can be found in moss. The first one studies the chlorinated pesticides (Tarcău et al., 2013), and the second one studies heavy metals, rare earths and some other microelements in the proximity of Ploiesti city (Oprea and Mihul, 2003). According to our knowledge, there are no studies in the scientific literature for the PAHs air pollution assessment by using moss in Romania.

MATERIAL AND METHODS

Sampling

The study was carried out in Cluj-Napoca, one of the largest cities of Romania, situated in the central north-western part. Eleven samples of common moss were collected from the neighborhoods presented in figure 1: Someșeni, Mărăști, IRA, Grigorescu, Mănăștur, Gheorgheni, Iris, Zorilor, Andrei Mureșanu, Parcul Central and Gruia.

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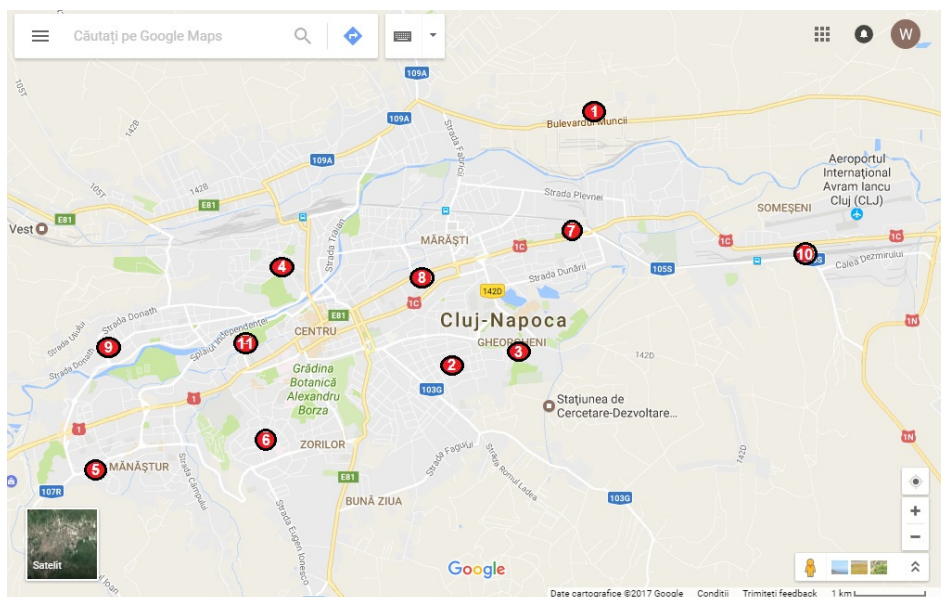


Fig. 1. Map showing the eleven sample locations in Cluj city.

1. Iris, 2. Andrei Mureșanu, 3. Gheorgheni, 4. Gruia, 5. Mănăștur, 6. Zorilor, 7. IRA, 8. Mărăști, 9. Grigorescu, 10. Someșeni, 11. Parcul Mare

All of the samples (table 1) were collected, when possible, at distances of more than 5 m from roads and houses. Whenever possible the samples were collected from parks and gardens, and when this was not possible, from the green areas that separated the block of flats from the sidewalk, maintaining of course the same distance limit.

Table 1. Samples abbreviation and location coordinates

| Sample location | Abbreviation | Latitude | Longitude |
|-----------------|--------------|--------------|--------------|
| Iris | S1 | 46°47'46.1"N | 23°37'49.4"E |
| Andrei Mureșanu | S2 | 46°45'43.1"N | 23°36'22.6"E |
| Gheorgheni | S3 | 46°46'05.2"N | 23°37'29.4"E |
| Gruia | S4 | 46°46'33.0"N | 23°34'38.9"E |
| Mănăștur | S5 | 46°45'10.5"N | 23°32'56.3"E |
| Zorilor | S6 | 46°45'25.9"N | 23°34'43.9"E |
| IRA | S7 | 46°46'55.0"N | 23°38'09.3"E |
| Mărăști | S8 | 46°46'36.7"N | 23°36'27.8"E |
| Grigorescu | S9 | 46°46'04.0"N | 23°32'59.3"E |
| Someșeni | S10 | 46°46'48.2"N | 23°40'47.2"E |
| Parcul Mare | S11 | 46°46'04.7"N | 23°34'30.8"E |

Sample processing

Due to the fact that, when the objective of a biomonitoring study with terrestrial mosses is to determine the bioconcentrated fraction, washing is ineffective (Aboal et al., 2011), and the option of dry-cleaning with a nitrogen jet is not available, the moss was cleaned by manually physical removal of contaminants: other plant remains, hair, soil, withered leaves, roots etc. The apical segments (3-4 cm long) were cut from the moss shoots and then left to dry on paper sheets at room temperature, 22° C, for 72 hours and milled afterwards.

Chemical analysis

For extraction of PAHs, 3 g of milled moss were weighed. Thirty milliliter of a mixture of n-hexane/dichloromethane (1:1 v/v) was then added. For extraction, the samples were placed in ultrasonic bath for 15 minutes. After extraction, the solvent was decanted and, filtered through a PVDF sample filter with pore size of 0.45 µm (Merck Milipore)). The extracts were then left to evaporate for 24 hours at room temperature and afterwards the residue was dissolved with 2 milliliter of n-hexane/dichloromethane (1:1) and kept in the freezer until they were analyzed.

The PAHs were analyzed by gas chromatography-mass spectrometry using a gas chromatograph model Thermo Electron Corporation (Focus GC; DSQII; TriPlus Autosampler). The separation of target compounds was performed on DB-5 column (25 m x0.25 mm x 0.25 µm i.d.). The MS had the ion source heated at 200° C, and was set on selected ion monitoring (SIM) mode. The separation was performed with two gradient of temperature as follows: from 120°C, up to 220°C with 10°C min⁻¹, and from 220°C to 300°C with 3°C min⁻¹. Injection volume was 1 µL, splitless, using Helium as carrier gas at a constant flow of 1,2 mL min⁻¹. The autosampler was set to have a 3 cycles pre-injection cleaning with 2 µL of solvent, and a 3 cycles post-injection cleaning with 2 µL of solvent.

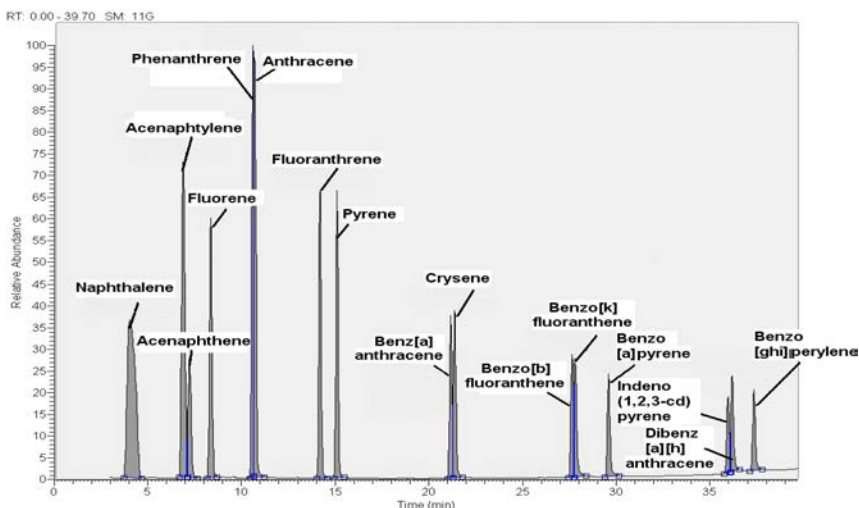


Fig. 2. Chromatogram of the PAHs standard mixture

For the qualitative and quantitative analysis a PAH standard mixture containing 16 compounds purchased by Supelco was used. The PAHs determined were: naphthalene, acenaphthene, acenaphthylene, fluorene, phenanthrene, anthracene, fluoranthene, pyrene, benz[a]anthracene, chrysene, benzo[b]fluoranthene, benzo[k]fluoranthene, benzo[a]pyrene, indeno(1,2,3-cd)pyrene, benzo[ghi]perylene and benzo[a]perylene (figure 2).

RESULTS AND DISCUSSION

The concentrations of individual PAHs (table 2) ranged from 0.61 ng g⁻¹ (chrysene – S3) to 353.6 ng g⁻¹ (Indeno(1,2,3 -cd)pyrene – S1), and the ΣPAHs ranged from 92.61 ng g⁻¹ (S8) to 353.6 ng g⁻¹ (S1) with a mean of 329.23 ng g⁻¹. In all samples the sum of high molecular PAHs, with a mean of 284.73 ng g⁻¹, was much higher than the sum of low molecular PAHs, with a mean of 44.51 ng g⁻¹, indicating that for all locations the main source is of pyrogenic origin.

Locations comparison

Residential areas characterized by both low population density and traffic intensity, like S3 – Gheorgheni, S4 – Gruia and S6 Zorilor neighborhoods, had the lowest concentration levels of ΣPAHs in moss samples, in all of these location the concentration level was below 200 ng g⁻¹. Mărăști and Grigorescu, although quite similar residential areas, obtained levels of concentration between 200 and 300 ng g⁻¹ due to the more intense traffic.

The locations that had PAHs concentration levels in moss exceeding 300 ng g⁻¹ are either industrial areas or neighborhoods with intense traffic. The highest concentration levels were in S1 - Iris and S2 – Andrei Mureșanu. Iris is the most developed industrial area of Cluj-Napoca city and the obtained results in this study are consistent with the expectations. Concentration levels in S7 – IRA (figure 3) was also consistent with the industrial activity, in a proportional ratio with the intensity of the activity, and in S10 location – Someșeni, the international airport of Cluj-Napoca city is located.

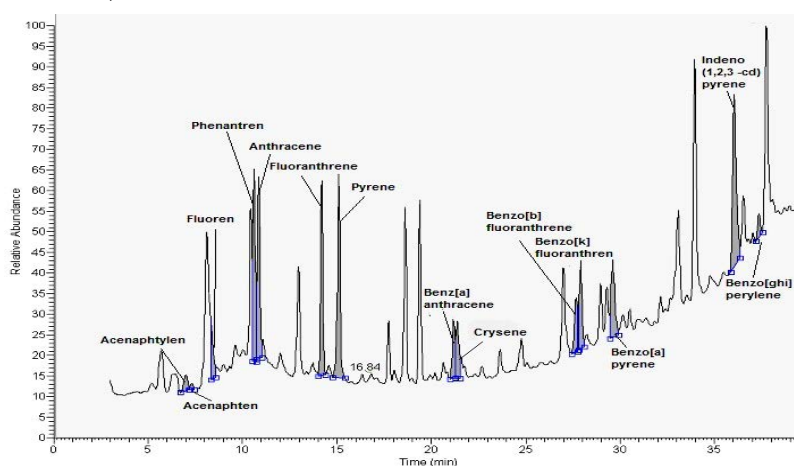


Fig. 3. Chromatogram of a real moss sample – S7 – IRA

On the other hand Andrei Mureșanu is a residential area, but the moss sample was collected from a central area of the neighborhood, in the close proximity of a busy intersection. High levels were also obtained in S11 – Parcul Mare and S8 – Mănăștur. Mănăștur is the neighborhood most densely populated from Cluj-Napoca city, so the high concentration levels are easily explained by the intense traffic and a large number of diverse heating systems. The intense traffic is also the reason why Parcul Central has high concentration levels, being situated in the city centre, which represents the access point from one neighborhood to another, also it is the subject of transitory vehicles from one city to another (e.g. from Zalău to Turda) and it is the most touristic part of the city, being an attraction for international festivals and sport competitions.

Table 2. Concentration of tested PAHs in selected zone areas

| Compound | Abbrev | Concentration (ng/g dry moss) | | | | | | | | | | |
|------------------------|--------------|-------------------------------|--------------|--------------|--------------|--------------|--------------|--------------|--------------|--------------|--------------|--------------|
| | | S1. | S2. | S3. | S4. | S5. | S6. | S7. | S8. | S9. | S10. | S11. |
| Naphtalene | NP | 0.78 | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND |
| Acenaphthylene | ACY | 3.12 | 3.42 | ND | ND | 4.65 | 1.70 | 2.66 | 1.23 | 3.82 | 3.44 | 2.79 |
| Acenaphthene | ACE | 3.74 | 2.65 | 3.01 | ND | 6.34 | 1.44 | 2.16 | 1.45 | 2.12 | 2.78 | 2.35 |
| Fluorene | FL | ND | 3.81 | 3.30 | 6.53 | 2.77 | 2.32 | 8.97 | 3.77 | 3.64 | 4.56 | 2.68 |
| Phenathrene | PHE | 15.19 | 10.34 | 4.15 | 8.46 | 3.95 | 5.33 | 24.84 | 9.18 | 7.67 | 19.32 | 10.14 |
| Anthracene | ANT | 16.45 | 27.72 | 13.44 | 22.52 | 24.88 | 12.83 | 24.41 | 21.53 | 21.68 | 37.07 | 50.46 |
| Fluoranthene | FLA | 10.15 | 9.46 | 2.69 | 9.72 | ND | 1.45 | 28.68 | 7.98 | ND | 12.95 | 3.15 |
| Pyrene | PYR | 11.19 | 15.83 | 3.22 | 8.45 | 5.38 | 1.09 | 33.87 | 7.84 | 2.90 | 11.38 | 5.78 |
| Benz[a]anthracene | BaA | 4.70 | 1.19 | 0.62 | 0.77 | 1.23 | ND | 16.64 | 1.04 | ND | 3.52 | 0.98 |
| Crysene | CHR | 4.10 | 40.21 | 0.61 | 2.96 | ND | ND | 13.85 | 1.66 | ND | 2.92 | ND |
| Benzo[b]fluoranthene | BbF | ND | ND | ND | ND | 3.28 | ND | 27.10 | ND | ND | ND | ND |
| Benzo[k]fluoranthene | BkF | 108.6 | ND | ND | 7.80 | 24.23 | ND | 26.95 | 86.29 | 24.68 | 47.07 | 88.01 |
| Benzo[a]pyrene | BaP | 8.54 | 61.58 | 18.56 | 5.07 | 13.67 | 34.66 | 42.01 | 26.26 | 15.86 | 37.55 | 2.53 |
| Indeno(1,2,3-cd)pyrene | IcdP | 353.6 | 273.8 | 107.6 | 116.6 | 271.2 | 128.5 | 122.6 | 92.6 | 199.8 | 176.4 | 206.3 |
| Dibenz[a,h]anthracene | DahA | ND | ND | ND | ND | ND | ND | ND | ND | ND | 13.90 | 16.00 |
| Benzo[ghi]perylene | BghiP | ND | ND | 6.59 | 10.31 | 11.88 | ND | 9.02 | ND | 11.94 | ND | 2.81 |
| | ΣPAHs | 540.2 | 450.0 | 163.8 | 199.2 | 373.4 | 189.3 | 383.8 | 260.8 | 294.1 | 372.9 | 394.0 |
| | min | 0.78 | 1.19 | 0.61 | 0.77 | 1.23 | 1.09 | 2.16 | 1.04 | 2.12 | 2.78 | 0.98 |
| | max | 353.6 | 273.8 | 107.6 | 116.6 | 271.2 | 128.5 | 122.6 | 92.6 | 199.8 | 176.4 | 206.3 |

ND – not detected

Table 3. *The diagnostic ration of PAHs sources in studied area*

| Isomeric ratios | Value | Source | S1. | S2. | S3. | S4. | S5. | S6. | S7. | S8. | S9. | S10. | S11. |
|------------------------------|----------|--------------------------------|------|------|------|------|------|------|------|------|------|------|------|
| Σ LMW/ Σ HMW | <1 | Pyrogenic | 0.01 | 0.02 | 0.04 | 0.03 | 0.04 | 0.03 | 0.04 | 0.03 | 0.03 | 0.03 | 0.02 |
| | >1 | Petrogenic | | | | | | | | | | | |
| Σ COMB/ Σ PAHs | ~1 | Combustion | 0.93 | 0.89 | 0.85 | 0.81 | 0.89 | 0.88 | 0.84 | 0.86 | 0.87 | 0.78 | 0.79 |
| FL/(FL + PYR) | <0.5 | Petrol emission | NA | 0.19 | | 0.44 | 0.34 | | 0.21 | 0.32 | | 0.29 | 0.32 |
| | >0.5 | Diesel emission | | | 0.51 | | | 0.68 | | | 0.56 | | |
| ANT/(ANT+PHE) | <0.1 | Petrogenic | | | | | | | | | | | |
| | >0.1 | Pyrogenic | 0.52 | 0.73 | 0.76 | 0.73 | 0.86 | 0.71 | 0.50 | 0.70 | 0.74 | 0.66 | 0.83 |
| FLA/(FLA + PYR) | <0.4 | Petrogenic | | 0.37 | | | NA | | | | NA | | 0.35 |
| | 0.4–0.5 | Fossil fuels combustion | 0.48 | | 0.46 | | | | 0.46 | 0.50 | | | |
| | >0.5 | Wood, coal, combustion | | | | 0.54 | | 0.57 | | | | 0.53 | |
| BaA/(BaA + CHR) | 0.2–0.35 | Coal combustion | | 0.03 | | | | | | | | | |
| | >0.35 | Vehicular emission/ Combustion | 0.53 | | 0.51 | 0.21 | | | 0.55 | 0.39 | | 0.55 | |
| | <0.2 | Petrogenic | | | | | | | | | | | |
| IcdP/(IcdP + BghiP) | <0.2 | Petrogenic | | | | | | | | | | | |
| | 0.2–0.5 | Petroleum combustion | | | | | | | | | | | |
| | >0.5 | Grass, wood, coal, combustion | NA | NA | 0.94 | 0.92 | 0.96 | NA | 0.93 | NA | 0.94 | NA | 0.99 |
| BaP/BghiP | <0.6 | Non-traffic emission | | | | | | | | | | | |
| | >0.6 | Traffic emission | NA | NA | 2.82 | 0.49 | 1.15 | NA | 4.66 | NA | 1.33 | NA | 0.90 |

PAH diagnostic ratio

In moss samples there is a low variability in PAHs identify, but more pronounced in each PAH quantification. The quantity of specific PAHs depends on the processes producing them. Low molecular weight PAHs are usually formed during low temperature processes (e.g. wood burning), and higher molecular weight PAH compound are formed during high temperature processes (e.g. the combustion of fuels in engines). To distinguish each source by using PAHs profile, PAH diagnostic ratios may be used. The ratios (table 3) can distinguish diesel and gasoline combustion emission, crude oil processing products and biomass burning processes (Tobiszewski and Namiejnik, 2012).

The most commonly used ratio, Σ LMW (sum of Low Molecular Weight PAHs) / Σ HMW (sum of High Molecular Weight PAHs) indicates that in all locations the main emission source is pyrogenic, so the PAHs are resulted from combustion rather than from spills. The mainly combustion source is also sustained by the results of the Σ COMB (FLA, PYR, BaA, CHR, BkF, BbF, BaP, IcdP and BghiP) / Σ PAHs ratio, the ANT/(ANT+PHE) ratio, BaA / (BaA+CHR), and IcdP/(IcdP + BghiP).

The BaP/BghiP ratio indicates that in all location PAHs mainly originate from traffic emissions, a statement strengthened by the FLA / (FLA+PYR) ratio, with the exception of S2, which seems to have a coal combustion source. These findings confirm the expected results, Cluj-Napoca being a developing city, densely populate, so with an intense traffic.

In nature there rarely is a single emission type, so the FL/ (FL+PYR) ratio indicates that in some cases there also is a petrogenic source (S3, S6 and S8). The FLA/ (FLA+ PYR) ratio indicates that for S2 and S11 the source seems to have a petrogenic origin too, but, in a cross examination of the FLA/ (FLA+PYR) and ANT/ (ANT+PHE) ratios (figure 4) it can easily be seen that the main apportion for all the location is from pyrogenic sources. The IcdP/(IcdP + BghiP) also indicates that the combustion may not only be from traffic emissions, but also from grass, wood or coal combustion.

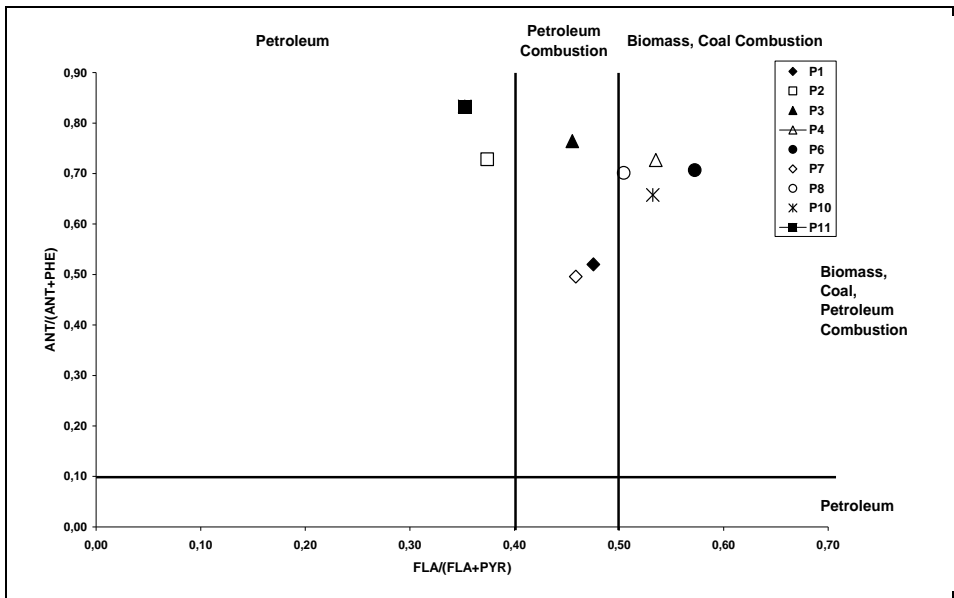


Fig. 4. Cross examination of the FLA/ (FLA+PYR) and ANT/ (ANT+PHE) ratios in the study locations

CONCLUSIONS

Residential areas characterized by both low population density and traffic intensity, like S3 – Gheorgheni, S4 – Gruia and S6 Zorilor neighborhoods, had the lowest concentration levels of ΣPAHs in moss samples, in all of these location the concentration level was below 200 μg kg⁻¹. Mărăști and Grigorescu, although quite similar residential areas, obtained levels of concentration between 200 and 300 μg kg⁻¹

due to the more intense traffic, and the locations that had PAHs concentration levels in moss exceeding $300 \mu\text{g kg}^{-1}$, are either industrial areas or neighborhoods with intense traffic.

In all the study's locations the main emission source, according to PAHs diagnostic ratios, is pyrogenic, mainly originating from traffic emissions. But in nature there rarely is a single emission type, so the FL/ (FL+PYR) and FLA/ (FLA+ PYR) ratios indicate that in some cases there also is a petrogenic source (S2, S3, S6, S8 and S11), but in a cross examination of the FLA/ (FLA+PYR) and ANT/ (ANT+PHE) ratios it can easily be seen that the main apportion for all the location is from pyrogenic sources.

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