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Original Article

Study on the Occurrence of Polycyclic Aromatic Hydrocarbons in Vehicular Soot and Outdoor Dust Samples: Concentrations, Profiles, and Source Estimation

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Abstract: Polycyclic aromatic hydrocarbons (PAHs) are pollutants of global concern due to their omnipresence in the environment and their negative effects on ecosystems and human health. Investigations on the occurrence of PAHs in emission sources and related environmental compartments are essential to understanding the behavior and fate of these pollutants. In this study, concentrations of 17 PAHs were analyzed in vehicular soot and outdoor dust samples collected from northern Vietnam. The samples were processed using ultrasound-assisted extraction and solid-phase extraction techniques. PAH concentrations were determined using a gas chromatography/mass spectrometry (GC/MS) method with internal standard quantification. Concentrations of 17 PAHs decreased in the order of gasoline soot (median 844,000; range 380,000–2,336,000 ng/g) > diesel soot (8500; 7200–176,000 ng/g) > outdoor dust (410; 130–2100 ng/g). The most predominant compounds were pyrene, benzo[ghi]perylene, fluoranthene, benzo[b+j]fluoranthene, and phenanthrene. Several PAH molecular indices in soot and dust samples were calculated and compared. Vehicular exhausts are important sources of PAHs in urban outdoor dusts. Our results have confirmed the pyrogenic origins of PAHs and the possibility of using molecular indices in PAH source apportionment. Further studies should be performed to characterize source-to-sink profiles of PAHs and related compounds in Vietnam.

Keywords: PAHs, vehicular soot, outdoor dust, molecular indices, Vietnam.

1. Introduction

Polycyclic aromatic hydrocarbons (PAHs) have been frequently monitored over the last five decades due to their widespread occurrence and various deleterious effects on environmental and human health [1, 2]. PAHs mainly originated from pyrogenic sources (i.e., incomplete combustion of organic materials) and petrogenic sources (i.e., petroleum products) [3, 4]. Traffic

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activities are important sources of PAHs, which release these pollutants through vehicular exhaust, tire abrasion, and spillage of oily materials [5, 6]. Traffic emissions have been characterized as major contributors to PAH levels found in outdoor dusts from various Asia countries like Japan, Taiwan, India, Thailand, Myanmar, and Vietnam [5-10]. Boonyatumanond et al., (2007) analyzed PAHs in street dusts from Bangkok, Thailand and several types of source materials like gasoline soot, diesel soot, roadside aerosols, used engine oils, tire particles, and asphalt [6]. Among these sources, tire debris and diesel vehicle exhaust were identified as the principal PAH sources in Bangkok [6]. Meanwhile, other studies reported that emissions from gasoline-powered vehicles were major contributors to PAHs in Vietnamese road dust [8-10]. These findings suggest that the simultaneous determination of PAHs in source-related samples and environmental matrices is crucial to characterizing pollution sources and behavior of these contaminants.

The occurrence, potential sources, and exposure risks of PAHs in outdoor dusts from Vietnam have been reported by some previous studies [7-10]. The source estimation of PAHs in these studies was mainly performed using diagnostic ratios or molecular indices of selected compounds. In which, reference indices of PAH sources were mainly retrieved from the literature without actually measured values. To our knowledge, there is only one study that analyzed PAHs in vehicle soot samples from Vietnam [11]. Hoang et al., (2020) reported that total concentrations of 19 unsubstituted PAHs and 15 methylated derivatives (Me-PAHs) in one pooled gasoline soot sample (obtained from three gasoline-powered motorbikes) and one pooled diesel soot sample (obtained from three diesel-powered trucks) were 800,000 and 12,000 ng/g, respectively [11]. One pooled road dust sample was also analyzed, showing a total PAH level of 930 ng/g [11]. The PAH profile of road dust sample was dominated by several compounds, including benzo[ghi]perylene, suggesting the influence of gasoline exhaust [11]. However, the sample numbers reported by Hoang et al., (2020) were quite small and can only provide preliminary information and limited discussion.

In this study, we analyzed concentrations of 17 PAHs, such as naphthalene (Nap), acenaphthylene (Acy), acenaphthene (Ace), fluorene (Flu), phenanthrene (Phe), anthracene (Ant), fluoranthene (Flt), pyrene (Pyr), benz[a]anthracene (BaA), chrysene (Chr), three benzofluoranthene isomers (BFs, including Bb+jF and BkF), benzo[a]pyrene (BaP), dibenz[a,h]anthracene (DA), indeno[1,2,3-cd]pyrene (IP), and benzo[ghi]perylene (BP), in gasoline soot, diesel soot, and outdoor dust samples collected from northern Vietnam. Concentrations, profiles, and molecular indices of PAHs were evaluated to characterize potential relationships between vehicular emissions and road dust contamination. Results of this study can update the database on the emission sources and environmental occurrence of PAHs in Vietnam.

2. Experimental

2.1. Chemicals and Materials

Three sets of PAH standards were used, including native standards (H-QME-01; AccuStandard, USA), surrogate standards (CIL-ES-2044; Cambridge Isotope Laboratories, USA), and internal standard (CIL-DLM-261; Cambridge Isotope Laboratories, USA). The working and calibration standard solutions were prepared by diluting the stock solutions with hexane. Organic solvents (i.e., hexane, acetone, and dichloromethane) at chromatographic grade were purchased from Fisher Scientific Korea (South Korea). The solid-phase extraction (SPE) cartridges (SPE Cartridge-SIL, silica gel 500 mg/3 mL) were provided by Mialab (Vietnam).

2.2. Sample Collection

The outdoor dust samples (n = 10) were manually collected around five campuses of the

Vietnam National University, Hanoi, using non-plastic brushes and pans. The obtained samples were transferred into paper bags and sealed in polyethylene (PE) zipper bags. The streets investigated in this study include Le Thanh Tong (R1, R2), Nguyen Trai (R3, R4), Luong The Vinh (R5, R6), Xuan Thuy (R7, R8), and Hoa Lac (R9, R10) (Figure 1). Five samples were taken from the road surface (R1, R3, R5, R7, R9) and five samples were taken inside the university campuses (R2, R4, R6, R8, R10). The vehicular soot samples were collected by shaving exhaust pipes with a stainless-steel spatula. The soot samples were wrapped in aluminum foil and sealed in PE zipper bags. Diesel soot samples were collected from four trucks (D1, D3, D4, D5) and one forklift (D2) in Bac Giang and Quang Ninh Provinces, respectively. Gasoline soot samples (G1, G2, G3, G4, G5) were taken from five motorbikes in Hanoi. All the samples were transported to the laboratory and stored at –4 °C until analysis.

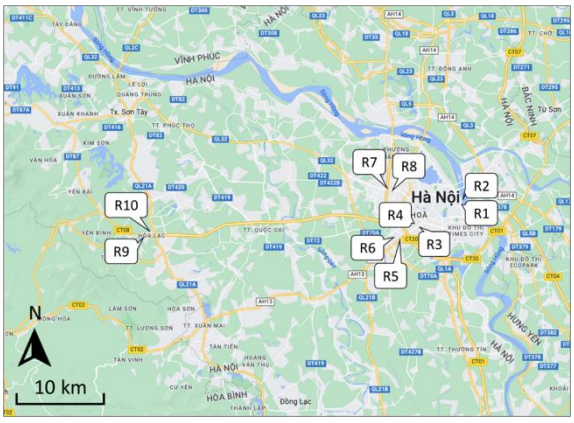

Figure 1. Sampling locations of outdoor dust samples in Hanoi, Vietnam.

2.3. Chemical and Instrumental Analysis

Chemical analysis was performed according to our previous procedures with minor modifications [10, 11]. The dust sample (<100 μm, 200 mg) was spiked with surrogate standards and extracted with acetone (2 mL, 10 min) and acetone/hexane mixture (1:1, v/v, 2 mL, 10 min) using an ultrasonic water bath (WUC-32; Jiayuanda, China). The two extract portions were combined, nitrogen evaporated, and reconstituted in 500 μL hexane before clean-up. The dust extract was purified by passing through a silica gel SPE cartridge and PAHs were eluted by 3 mL dichloromethane/hexane (1:3, v/v) mixture. The eluate was spiked with internal standard and nitrogen evaporated to 500 μL before quantification.

The soot sample (100 mg) was extracted three times with a total of 6 mL acetone (each time with 2 mL acetone in 10 min) using the same ultrasonic bath. The three extract portions were combined and hexane was added to make 10 mL extract. Diesel soot extract (1 mL, corresponding to 10 mg soot) or gasoline soot extract (100 μL, corresponding to 1 mg soot) were taken and transferred to a concentration tube, spiked with labeled standards, and solvent-exchanged into 1 mL hexane before quantification.

PAHs were quantified using a gas chromatography/mass spectrometry (GC/MS) system (GC 8890 MS 5977B) and a DB-5 column (30 m × 0.25 mm × 0.25 μm) (Agilent Technologies, USA). The MS detector was operated in electron ionization and selected ion monitoring modes. The GC/MS instrumental parameters were retrieved from our previous studies [10,11]. Statistical analysis was performed using Microsoft® Excel® (Microsoft Office Home and Student 2019) and Minitab® 21.3 Statistical Software (Minitab, LLC.).

3. Results and Discussion

3.1. Concentrations of PAHs in Vehicular Soot and Outdoor Dust Samples

PAHs were detected in vehicular soot and outdoor dust samples at total concentrations ranging from 130 to 2,336,000 ng/g (Figure 2a). The highest PAH levels were found in the gasoline soot (median 844,000; range 380,000–2,336,000 ng/g), followed by diesel soot (8500;

7200–176,000 ng/g) and outdoor dust (410; 130–2100 ng/g). All the target PAHs were found in the soot samples at a detection frequency of 100% and individual concentrations ranging from 4.1 to 723,000 ng/g. The most predominant PAHs detected in the soot samples were BP, IP, BaP, BFs, Flt, and Pyr, with maximum concentrations of up to several hundred ppm. For the outdoor dust samples, except for Flu, Acy, Nap, and Ace (with detection frequencies of 90%, 90%, 40%, and 10%, respectively), the remaining compounds were detected in all the samples. Individual concentrations of PAHs in the dust samples ranged from <1.0 to 360 ng/g. Major PAHs in the dust samples were BP, Flt, Pyr, Phe, and BFs. PAH concentrations in the soot samples were about two to three magnitude orders higher than those in the dust samples, suggesting vehicular emissions are important sources of these compounds. In addition, these two matrices shared several key compounds, indicating their source-to-sink relationship.

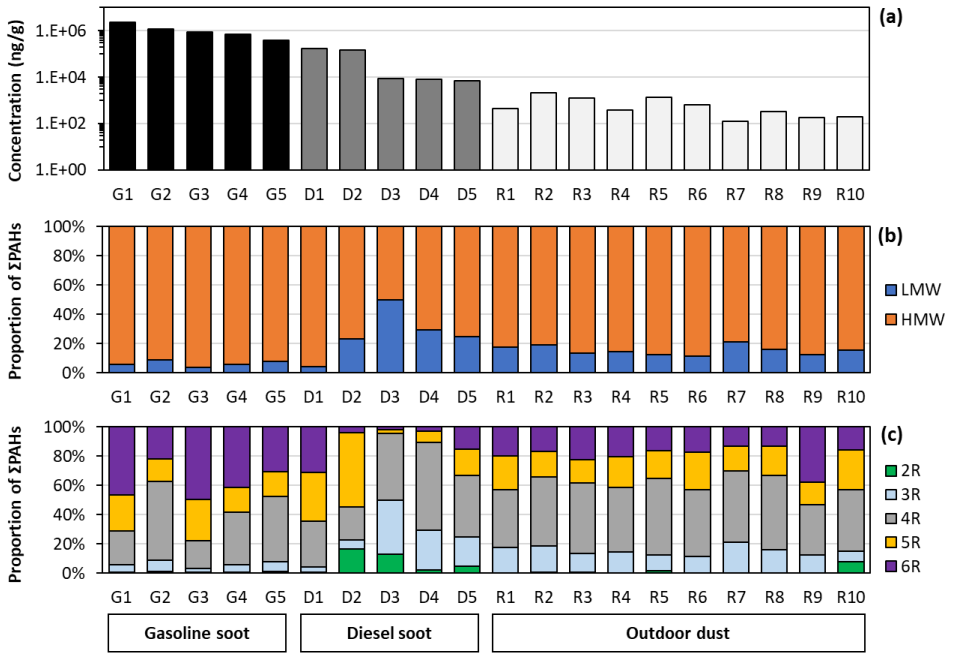

Figure 2. The occurrence of PAHs in vehicular soot and outdoor dust samples: (a) concentrations of total PAHs; (b) proportions of LMW and HMW PAHs; (c) proportions of PAHs by ring numbers.

Concentrations of PAHs in vehicle soot samples were reported in some previous studies [5, 6, 11-13]. Takada et al., (1991) reported concentrations of 11 PAHs in three diesel soot (16,000–197,000 ng/g) and one gasoline soot (147,000 ng/g) samples in Japan [5]. Concentration of 14 PAHs in one pooled vehicle soot sample (n = 70) in the USA was 136,000 ng/g and dominated by BP (23,800 ng/g), Pyr (22,000 ng/g), Phe (20,500 ng/g), Flt (17,200 ng/g), and IP (10,900 ng/g) [12]. Higher PAH concentrations were measured in gasoline soot of passenger cars and motorbikes (127,000–5,228,000 ng/g) than in diesel soot of trucks and tractors (77,000–317,000 ng/g) [13]. Boonyatumanond et al., (2007) measured higher PAH levels in gasoline soot (sum of 15 PAHs and 4 Me-PAHs: 520,000–6,901,000 ng/g) than in diesel soot (5600–670,700 ng/g) [6]. Hoang et al., (2020) also found higher PAH

concentration in motorbike gasoline soot (sum of 19 PAHs and 15 Me-PAHs: 800,000 ng/g) than in diesel soot (12,000 ng/g) [11]. Levels of PAHs detected in our gasoline soot (380,000–2,336,000 ng/g) and diesel soot samples (7200–176,000 ng/g) were within the range documented for similar samples from other countries. It should be noted that vehicles investigated in our study are of different categories (i.e., gasoline-powered motorcycles and diesel-powered trucks and forklift), with dissimilar formation mechanisms, emission pathways, engine working principles, exhaust system efficiency, and soot characteristics. Several previous studies also reported higher PAH levels in gasoline soot than in diesel soot [6, 11, 13]. Higher PAH levels found in gasoline soot than in diesel soot can be explained by several reasons: i) Gasoline contains higher PAH precusors (e.g., aromatic hydrocarbons) than diesel; ii) Gasoline soot has smaller particle size and adsorption capacity for PAHs than diesel soot; and iii) Combustion conditions, such as temperature and oxygen content, in gasoline-powered engines are more suitable for PAH formations than in diesel-powered engines.

The PAH levels in our outdoor dust samples (130–2100 ng/g) fall in the ranges previously reported for Vietnamese road dusts [7-10]. Interestingly, PAH concentration ranges in Hanoi road dusts show an insignificant temporal variation over the last two decades. Total PAH levels in road dust samples collected in Hanoi in 2011 (76–5500 ng/g), 2012–2013 (260–4700 ng/g), and 2016 (170–5000 ng/g) imply relatively constant emission degree and stable concentrations [8-10]. The narrow concentration range of PAHs in our study (sampling in 2022) was largely due to the selection of sampling locations and limited sample size. Previous studies focused on the main roads with high traffic density in Hanoi to clarify the relationships between PAH levels and urbanization degree, showing a clear urban-rural gradient [7-10]. In the present study, we investigated five pairs of outdoor dust samples collected around 5 campuses, including 5 on-road samples and 5 in-campus samples. As shown in Figure 2a, the Nguyen Trai and Luong The Vinh sites had higher PAH levels on roads compared to campuses, implying direct contamination of road surfaces with vehicular exhausts. In contrast, two Le Thanh Tong and Xuan Thuy sites had higher PAH levels on campuses compared to roads. One possible reason for this observation is that the Le Thanh Tong and Xuan Thuy campuses also serve as parking areas of the university, resulting in relatively high vehicle intensity and PAH emission. The Hoa Lac campus is located in a suburban area with comparable PAH levels between on-road and in-campus samples. In general, PAH concentrations in Vietnamese road dusts were within the ranges reported in other Asian countries like Myanmar (84–4100 ng/g), Japan (max. 3200 ng/g), Taiwan (max. 9400 ng/g), and India (650–1800 ng/g) [7, 8].

3.2. Profiles of PAHs in Vehicular Soot and Outdoor Dust Samples

High-molecular-weight (HMW) PAHs (4–6 rings; 50–97% of total PAHs) were more dominant than low-molecular-weight (LMW) compounds (2–3 rings; 3–50% of total PAHs) (Figure 2b). However, the proportions of these two groups were not similar between sample categories. Percentages of HMW PAHs were the highest in the gasoline soot samples (94% ± 2%), followed by dust samples (85% ± 3%), and diesel soot samples (74% ± 16%). Conversely, LMW-PAHs were more abundant in the diesel soot samples (26% ± 16%) than in the dust (15% ± 3%) and gasoline soot samples (6% ± 2%). PAHs are formed in the vehicle engines and released into the environment (i.e., air and road dust) in the form of a complex mixtures distributed between gas and particulate phases. LMW PAHs mainly occur in the gas phase due to their high volatility, while HMW PAHs mainly associated with the particulate phase (i.e., soot particles and dust) due to their lower volatility. Therefore, our soot and dust samples

were dominated by PAHs containing 4–6 rings, which were in accordance with those reported by previous studies conducted in other countries [6-8, 13].

The ring-number profiles of PAHs varied between the sample categories (Figure 2c). In the gasoline soot samples, 6-ring PAHs accounted for the highest percentages (38% ± 12%), followed by 4-ring (35% ± 15%), 5-ring (20% ± 6%), 3-ring (5.2% ± 1.9%), and 2-ring (1.1% ± 0.3%) compounds. Meanwhile, the diesel soot samples mainly accumulated 4-ring PAHs (40% ± 14%), followed by 5-ring (22% ± 20%), 3-ring (19% ± 14%), 6-ring (11% ± 12%), and 2-ring (7% ± 7%) compounds. Ring profiles in the dust samples showed a peak at 4-ring compounds (45% ± 5%), followed by 5-ring (20% ± 4%), 6-ring (19% ± 7%), 3-ring (14% ± 4%), and 2-ring (1.1% ± 2.4%) PAHs. The differences in PAH profiles between soot and dust samples are likely attributed to the mixing effects of multiple emission sources and the transformation process of these compounds in the environment.

The compound-specific profiles of PAHs are presented in Figure 3. For the whole dataset, the most predominant compounds were Pyr (17% ± 7%), BP (14% ± 10%), Flt (13% ± 5%), Bb+jF (12% ± 7%), Phe (10% ± 6%), IP (8% ± 4%), and Chr (8% ± 2%). Proportions of BP and IP in the gasoline soot samples were higher than those found in the dust and diesel soot samples. The PAH profiles in the diesel soot samples showed the highest variation. Three diesel soot samples (D3, D4, D5) had similar profiles with major compounds as Pyr (20–30%), Phe (14–23%), and Flt (12–19%). The diesel soot sample D1 was dominated by Bb+jF (23%), BP (16%), and IP (15%). The remaining soot sample D2 (forklift) had higher percentages of Bb+jF (33%) and Nap (17%). Moderate proportions were observed for BaP (5% ± 3%), BaA (4% ± 2%), Nap (3% ± 5%), and BkF (3% ± 2%). Compounds detected at the lowest percentages were Flu (1.4% ± 1.6%), DA (1.1% ± 0.6%), Acy (0.8% ± 1.0%), and Ace (0.1% ± 0.2%).

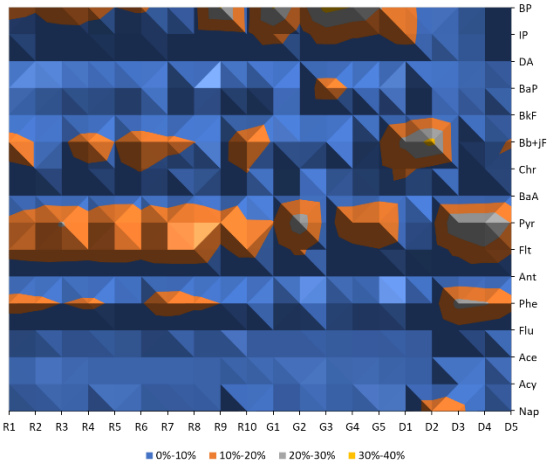

Figure 3. Distribution profiles of PAHs in the vehicular soot and outdoor dust samples.

We applied hierarchical cluster analysis of PAH profiles in outdoor dust and vehicle soot samples to estimate their potential relationships based on PAH pattern similarity (Figure 4). Almost all dust samples (except for the sample R10) showed similar PAH profiles and matched with two gasoline soot samples (G2 and G5), partly indicating gasoline exhausts as a potential source of PAHs in outdoor dusts. The dust sample R10 (inside the Hoa Lac campus) was grouped with two diesel soot samples (D1 and D2), implying emission from diesel-powered vehicles of transportation and construction activities. The remaining groups were attributed to gasoline emissions (G1, G3, G4) and diesel emissions (D3, D4, D5).

3.3. Molecular Indices of PAHs in Outdoor Dust and Vehicle Soot Samples

To provide more details on the PAH source estimation, molecular indices were calculated for the dust and soot samples (Table 1). As shown in Table 1, the gasoline soot samples exhibited molecular indices with less variation than the diesel soot samples. The LMW/HMW

indices lower than 1.0 (except for one diesel soot sample) have confirmed the dominance of pyrogenic sources of PAHs in the investigated samples. The indices that showed the largest difference between the gasoline and diesel soot were Phe/Ant, Ant/(Ant + Phe), BaA/Chr, IP/BP, and BaA/BaP. The remaining indices, including Flt/Pyr, Flt/(Flt + Pyr), BaA/(BaA + Chr), IP/(IP + BP), and BaP/BP exhibited less variation.

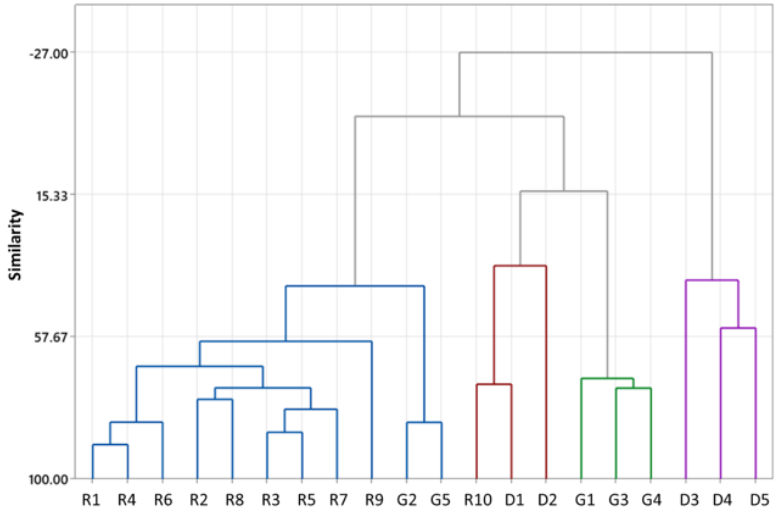

Figure 4. Hierarchical cluster analysis of PAH profiles in outdoor dust and vehicle soot samples of this study.

Table 1. Molecular indices (median and range) of selected PAHs in outdoor dust and vehicle soot samples of this study

Indices	Gasoline soot	Diesel soot	Outdoor dust
LMW/HMW	0.061 (0.036–0.097)	0.33 (0.045–1.0)	0.18 (0.13–0.26)
Phe/Ant	2.56 (1.83–3.06)	11.8 (8.48–23.1)	11.4 (7.27–41.0)
Ant/(Ant + Phe)	0.13 (0.11–0.17)	0.063 (0.040–0.154)	0.048 (0.023–0.092)
Flt/Pyr	0.50 (0.46–0.57)	0.63 (0.44–0.92)	1.07 (0.56–1.34)
Flt/(Flt + Pyr)	0.33 (0.31–0.36)	0.38 (0.31–0.48)	0.52 (0.36–0.57)
BaA/Chr	0.94 (0.90–1.31)	0.52 (0.18–0.65)	0.46 (0.36–0.91)
BaA/(BaA + Chr)	0.48 (0.47–0.57)	0.34 (0.15–0.39)	0.32 (0.26–0.48)
IP/BP	0.46 (0.39–0.50)	0.96 (0.43–1.86)	0.61 (0.23–1.12)
IP/(IP + BP)	0.31 (0.28–0.33)	0.49 (0.30–0.65)	0.38 (0.18–0.53)
BaA/BaP	0.60 (0.32–2.23)	1.96 (0.14–3.56)	1.06 (0.73–1.68)
BaP/BP	0.26 (0.19–0.45)	0.30 (0.23–6.67)	0.35 (0.13–0.79)

In most cases, the road dust samples showed different indices compared to the soot samples, indicating effects of weathering process and contributions of other sources. The Phe/Ant and Ant/(Ant + Phe) indices of dust samples were closer to diesel samples than

gasoline samples; however, these indices were more suitable to discriminate pyrogenic/petrogenic sources rather than gasoline/diesel sources [14]. The Flt/(Flt + Pyr) ratios can be used to distinguish petrogenic sources (<0.4), fossil fuel combustion (0.4–0.5), and biomass and coal combustion (>0.5) [15]. Although Flt and Pyr were major compounds detected in the soot samples, they are also abundant in other pyrogenic sources like coal combustion, wood combustion, incineration, and oil burning 3. The BaA/Chr and BaA/(BaA + Chr) ratios in the dust samples approached the diesel soot rather than gasoline soot. Meanwhile, the IP/BP, IP/(IP + BP), and BaA/BaP values distributed between the ranges of gasoline and diesel soot. These results indicate that the occurrence of PAHs in outdoor dust is regulated by mixed sources with higher contributions of pyrogenic inputs than petrogenic releases. The use of molecular indices is efficient to distinguish pyrogenic and petrogenic sources of PAHs, but no single indice can be applied to characterize specific sources of the same thermal origins.

4. Conclusion

Concentrations of 17 PAHs were determined in vehicle soot and outdoor dust samples collected from northern Vietnam. Elevated PAH levels of up to mg/g were detected in gasoline soot samples, which were about one to two magnitude orders higher than those in diesel soot samples. PAHs were also measured in road dust at low μg/g levels. PAH profiles were characterized for samples, showing the abundance of HMW compounds over LMW ones. The most predominant PAHs were Pyr, BP, Flt, Bb+jF, Phe, IP, and Chr; however, the profiles varied between sample categories and even between samples of the same category. Traffic activities and urbanization levels are estimated as important factors influencing PAH contents and patterns in outdoor dust samples. Molecular indices were calculated, showing significant contribution of pyrogenic sources over petrogenic inputs. Additional investigations are needed to provide more comprehensive insights into PAH emission sources and their relative contributions.

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