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Monitoring polycyclic aromatic hydrocarbon concentrations and distributions in rice paddy soils from Gyeonggi-do, Ulsan, and Pohang

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Abstract

Polycyclic aromatic hydrocarbons (PAHs) are ubiquitous pollutants that are released by incomplete combustion of carbon-containing materials. The top soils of rice paddies were collected from Gyeonggi-do (18 sites), Ulsan (20 sites), and Pohang (19 sites) in Korea to assess the spatial distribution and potential sources of PAHs. The total concentrations of 15 PAHs in the soils were 19.53–672.93, 125.01–3106.27, and 51.94–8106.21 mg/kg in Gyeonggi province, Ulsan, and Pohang, respectively. The concentration of 7 key carcinogenic PAHs were followed the order: Pohang (38.54–4826.63 ng/g) > Ulsan (28.54–1561.39 ng/g) > Gyeonggi province (19.53–206.51 ng/g). Three-ring PAHs were predominant in the soils from Gyeonggi-do while 3–5 ring compounds were abundant in the agricultural soils from the two industrial regions (Ulsan and Pohang). The PAH isomeric diagnostic ratios indicated that PAH contamination in the two cities mainly originated from pyrogenic sources. The principal component analysis indicated that pyrogenic coal burning and residential biomass combustion were major contributors to the soil contamination in the two cities. The transportation of PAHs through the air from industrial complexes and high volume of traffic may influence the PAHs distribution in the soils of the two cities in Korea.

Keywords: Contamination, Distribution, Polycyclic aromatic hydrocarbons, Soil, Source

Introduction

Polycyclic aromatic hydrocarbons (PAHs) are abundant environmental pollutants produced by various human activities including the incomplete combustion of carbon-containing materials [1]. PAHs are hardly degraded by microorganisms due to their stable aromatic ring structures and some PAHs are known to be carcinogenic and mutagenic [2]. PAHs are easily dispersed and undergo long-range atmospheric transport to remote regions such as the Arctic [3, 4]. Once PAHs are released into the environment, they can be extensively dispersed

through the air and water and deposited in the soil. A large quantity of PAHs released into the atmosphere deposits in soil via dry and wet deposition [5, 6].

As a major reservoir of pollutants, the soil system is an important indicator of long-term environmental problems. Because of their persistency and hydrophobicity, PAHs are prone to enrichment in the soil matrix long after being adsorbed by the soil and sediment organic matter [7, 8]. PAHs in soils can be transferred into agricultural products, further threatening human health [9, 10]. Previous studies have shown that 3-ring PAHs are readily absorbed by maize roots but 2- and 4–6 ring PAHs are less likely to be absorbed into the plants [11]. Therefore, studies regarding agricultural soil PAH contamination must be performed to decrease the risk of human exposure and environmental contamination.

Recently, various studies have been published regarding the concentrations, distributions, and sources of

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PAHs in soils in a wide range of regions throughout the world. PAH contamination of urban soils have been reported in Miami in the USA [12], Esbjerg in Denmark [13], Ulsan in South Korea [5], Shanghai [7], Nanjing [14], and Beijing [15, 16] in China, and Dhanbad in India [17]. To identify the source of PAHs in soils, a wide variety of studies determined the specific isomeric ratios of PAHs [5, 18–20]. The ratio of low molecular weight PAHs (LMWPAHs) to those with high molecular weight (HMWPAHs), anthracene/(anthracene + phenanthrene), and fluoranthene/(fluoranthene + pyrene) are commonly used [5, 8, 21, 22].

Emissions of PAHs have greatly increased by the extensive industrialization in Ulsan and Pohang [5, 23, 24]. Contamination with PAHs in the agricultural lands surrounding these areas is highly likely to occur since many are located near roads or adjacent to industrial areas [25, 26]. A previous study showed that the major contributors to PAHs in road dust in Ulsan were emissions from industrial activities and automobile exhaust [24]. However, a limited number of studies have been performed regarding PAHs in agricultural soils in these industrial regions. Therefore, it is essential to monitor continuously the contamination for human safety.

This study aimed to investigate the concentrations and spatial distributions 16 PAHs in rice paddy soils collected from Gyeonggi province (18 sites) and two industrial areas, Ulsan (20 sites) and Pohang (19 sites) in South Korea. Based on the determined PAH concentrations, pollution sources were identified using specific diagnostic ratios of PAHs and principal component analysis (PCA).

Materials and methods

Standards and sample preparation

The 15 major PAHs identified by the EPA include acenaphthylene (Acy), acenaphthene (Ace), fluorene (Flu), phenanthrene (Phe), anthracene (Ant), fluoranthene (Flt), pyrene (Pyr), benzo[*b*]fluoranthene (BbF), benzo[*k*]fluoranthene (BkF), BaP, indeno[1,2,3-*cd*]pyrene (Ind), dibenzo[*a,h*]anthracene (DahA), and benzo[*g,h,i*]perylene (BghiP). The mixture of 16 PAHs was purchased from Accustandard (CT, USA). The surrogates including acephthene-d10, phenanthrene-d10, chrysene-d10, fluorene-d10, and benzo[*a*]pyrene-d12 were obtained from Sigma (MO, USA).

Rice paddy soils (0–10 cm) were collected from 18 sites in Gyeonggi province, 20 sites in Ulsan City, and 19 sites in Pohang City, Gyeongsang Province, South Korea. The grid references of the sampling locations from all sites are listed Additional file 1: Table S1. After the overlying vegetation was removed, the soils were sampled at five points and placed into the same plastic bag. In the laboratory,

before passing through a 2-mm sieve, each sample was mixed thoroughly and air-dried at room temperature.

The PAH analysis in soil was carried out based on the U.S EPA method. The soil samples (50 g) were added to a flask and 1 mg/L of surrogate (100 μ L) was spiked into each sample. After NaCl (10 g) and dichloromethane (100 mL) were added, the samples were shaken for 15 min using a mechanical shaker. After the extracts were filtered and passed through sodium sulfate to remove residual water, they were evaporated using a rotatory evaporator. For clean-up, silica SPE cartridges were used prior to gas chromatography-mass spectrometry (GC-MS) analysis.

Instrumentation

An Agilent 6890 N GC (Agilent Technologies, Santa Clara, CA, USA) furnished with an Agilent 7683B injector (Agilent Technologies, Santa Clara, CA, USA) and an Agilent 5975 mass selective detector (MSD) was used for PAH analysis. In the GC system, a DB-5MS capillary column (30 m \times 0.25 mm i.d. \times 0.25 μ m film thickness) was installed to separate the analytes. The injection volume was 1 μ L for each sample using split mode (3:1) at an injector temperature of 300 $^{\circ}$ C. The transfer line and ion source temperatures were 280 and 200 $^{\circ}$ C, respectively. The oven temperature was first held at 65 $^{\circ}$ C for 2 min, increased to 140 $^{\circ}$ C (held for 10 min) at a rate of 10 $^{\circ}$ C/min, and finally to 290 $^{\circ}$ C (held for 9 min) at a rate of 10 $^{\circ}$ C/min. Helium was used as a carrier gas at a constant flow rate of 1.2 mL/min. Mass spectra were acquired using electron ionization and selective ion monitoring modes. The ion mass conditions used for PAH quantification is shown in Table 1.

Quality control and data analysis

Quantitation was performed using an external standard calibration method (five-point calibration; 0.05, 0.10, 0.25, 0.50, and 0.10 mg/L) and correlation coefficients ($R^2 \geq 0.999$) for the calibration curves. The analytical methods optimized in this experiment were validated for both precision and accuracy. The method detection limits (MDLs) for the PAHs at 10 μ g/kg fortification level ranged from 4 to 18 ng/g and limits of quantification (LOQs) ranged from 12 to 52 ng/g. Matrix spike experiments ($n=3$) were performed at the 10 μ g/kg spiking level and the recovery rates were ranged from 60 to 120% with relative standard deviations (RSDs) $\leq 10\%$ except Phe ($128\% \pm 1\%$) and Flt ($133\% \pm 6\%$). MDLs, LOQs, and recoveries for each PAH with RSDs were listed in Additional file 2: Table S2.

The statistical package IBM SPSS 22.0 software for the PCA analysis and SigmaPlot 12.0 for rank sum tests were used to analyze the data [5, 18, 19]. For the PCA analysis,

Table 1 The ion mass conditions used for PAH quantification

| PAHs | RT (min) | Q1 | Q2 (ratio) | Surrogate | I.S. |
|------------------------|----------|-----|-------------|--------------------|------|
| Acenaphthylene | 14.134 | 152 | 151 (18.98) | A | I |
| Acenaphthene | 15.218 | 153 | 154 (96.00) | | |
| Fluorene | 19.608 | 166 | 165 (90.44) | | |
| Phenathrene | 25.035 | 178 | 176 (18.12) | B | |
| Anthracene | 25.232 | 178 | 176 (17.76) | | |
| Fluoroanthene | 28.906 | 202 | 101 (23.32) | C | II |
| Pyrene | 29.502 | 202 | 101 (25.78) | | |
| Benzo[a]anthracene | 32.647 | 228 | 114 (23.41) | | |
| Chrysene | 32.73 | 228 | 114 (22.11) | | |
| Benzo[b]fluoranthene | 35.132 | 252 | 126 (33.41) | D | |
| Benzo[k]fluoranthene | 35.194 | 252 | 126 (36.27) | | |
| Benzo[a]pyrene | 35.921 | 252 | 126 (37.28) | | |
| Indeno[1,2,3-cd]pyrene | 39.459 | 276 | 138 (54.57) | | |
| Dibenzo[a,h]anthracene | 39.62 | 278 | 139 (44.32) | | |
| Benzo[g,h,i]perylene | 40.461 | 276 | 138 (65.03) | | |
| Acenaphthene-d10 | 15.026 | 164 | A | Surrogates | |
| Phenanthrene-d10 | 24.952 | 188 | B | | |
| Chrysene-d10 | 32.667 | 240 | C | | |
| Perlene-d12 | 36.071 | 264 | D | | |
| Fluorene-d10 | 19.317 | 176 | I.S.-I | Internal Standards | |
| Benzo[a]pyrene-d12 | 35.835 | 264 | I.S.-II | | |

the data were normalized with the sum of Phe, Ant, Flt, Pyr, BaA, Chr, BbF, and BkF. All the data processing was performed using Microsoft Excel (Microsoft Inc., Redmond, WA, USA).

Results

Concentrations and spatial distribution of PAHs in rice paddy soils

The concentrations of US EPA priority PAHs (15 PAHs) in rice paddy soils in Gyeonggi province and two industrial areas (Ulsan and Pohang) in Gyeongsang province were determined (Additional file 3: Fig. S1). The distribution and concentration of the 15 PAHs (Σ_{15} PAHs) and 7 carcinogenic PAHs (Σ_7 PAHs; BaA, Chr, BbF, BkF, BaP, Ind, and DahA) in the top soils of Gyeonggi province, Ulsan, and Pohang are shown in Fig. 1 and Additional file 4: Table S3. The spatial distribution of the Σ_{15} PAHs varied and ranged between 19.53 and 672.93 ng/g (mean: 253.13 ng/g; median: 230.36 ng/g) for Gyeonggi

province, 125.01–3106.27 ng/g (mean: 810.41 ng/g; median 606.33 ng/g) for Ulsan, and 51.94–8106.21 ng/g (mean: 2383.97 ng/g; median: 1888.84 ng/g) for Pohang. The concentrations of the Σ_{15} PAHs in Gyeonggi province were statistically different from those found in Ulsan (rank sum test $p < 0.001$) and Pohang (rank sum test $p < 0.001$). However, the PAH concentrations in Ulsan were not statistically different from those observed in Pohang (rank sum test $p = 0.001$).

The concentrations of the Σ_7 PAHs (19.53–206.51 ng/g) among 15 PAHs determined in Gyeonggi province were statistically different from those in observed in Ulsan (28.54–1561.39 ng/g, rank sum test $p = 0.012$) and Pohang (38.54–4826.63 ng/g, rank sum test $p < 0.001$). Unlike the Σ_{15} PAHs, the concentrations of the Σ_7 PAHs in the Ulsan soil samples were statistically different from those sampled from Pohang (rank sum test $p = 0.004$). These results indicated that the soils in Pohang were more contaminated with Σ_7 PAHs than those in Ulsan and Gyeonggi province.

PAH profiles in rice paddy soils

The composition profiles of the 16 PAHs in Gyeonggi province, Ulsan, and Pohang are presented in Fig. 2. The 16 PAHs are generally classified into five groups: 2-ring, 3-ring, 4-ring, 5-ring, and 6-ring PAHs, based on the number of aromatic rings. The sequence of the PAHs proportion in rice paddy soils of Gyeonggi province was composed of 3-ring (69.9%)–4-ring (17.7%)–5-ring (12.3%) (Fig. 2a). The paddy soils of Ulsan mainly consisted of 3-ring (60.1%)–4-ring (24.0%)–5-ring (14.9%)–6-ring (1.0%) (Fig. 2b). The paddy soils of Pohang were also primarily composed of 3-ring (37.2%)–4-ring (44.6%)–5-ring (14.8%)–6-ring (3.4%) (Fig. 2c). Phe in 3 ring PAHs was dominant (66%) in the soils from Gyeonggi province, while Phe and Ant in 3-rings and Flt and Pyr in 4-rings were dominant compounds in the soil samples from Ulsan. BaA, Chr, BbF, and BkF were next dominant PAHs in Ulsan soils. Phe, Ant, Flt, Pyr, BaA, Chr, BbF, and BkF were dominant in Pohang soils.

Source identification using PAH isomeric ratios

The sources of PAHs can be identified based on their concentrations and patterns in the soil. Ratios of LMW/HMW > 1 suggest that PAH contamination primarily originates from petrogenic sources (hydrocarbons related with petroleum). On the other hand, LMW/HMW ratios of < 1.0 are a result of pyrogenic PAH generation the burning of petroleum, wood, and coal [27]. The ratios of LMW/HMW in Gyeonggi province, Ulsan, and Pohang are shown in Fig. 3. The LMW/HMW ratios in the soils ranged from 0.22 to 18.74 with a mean value of 2.40 for Gyeonggi province, 0.09–10.55 with a mean of 2.48 for

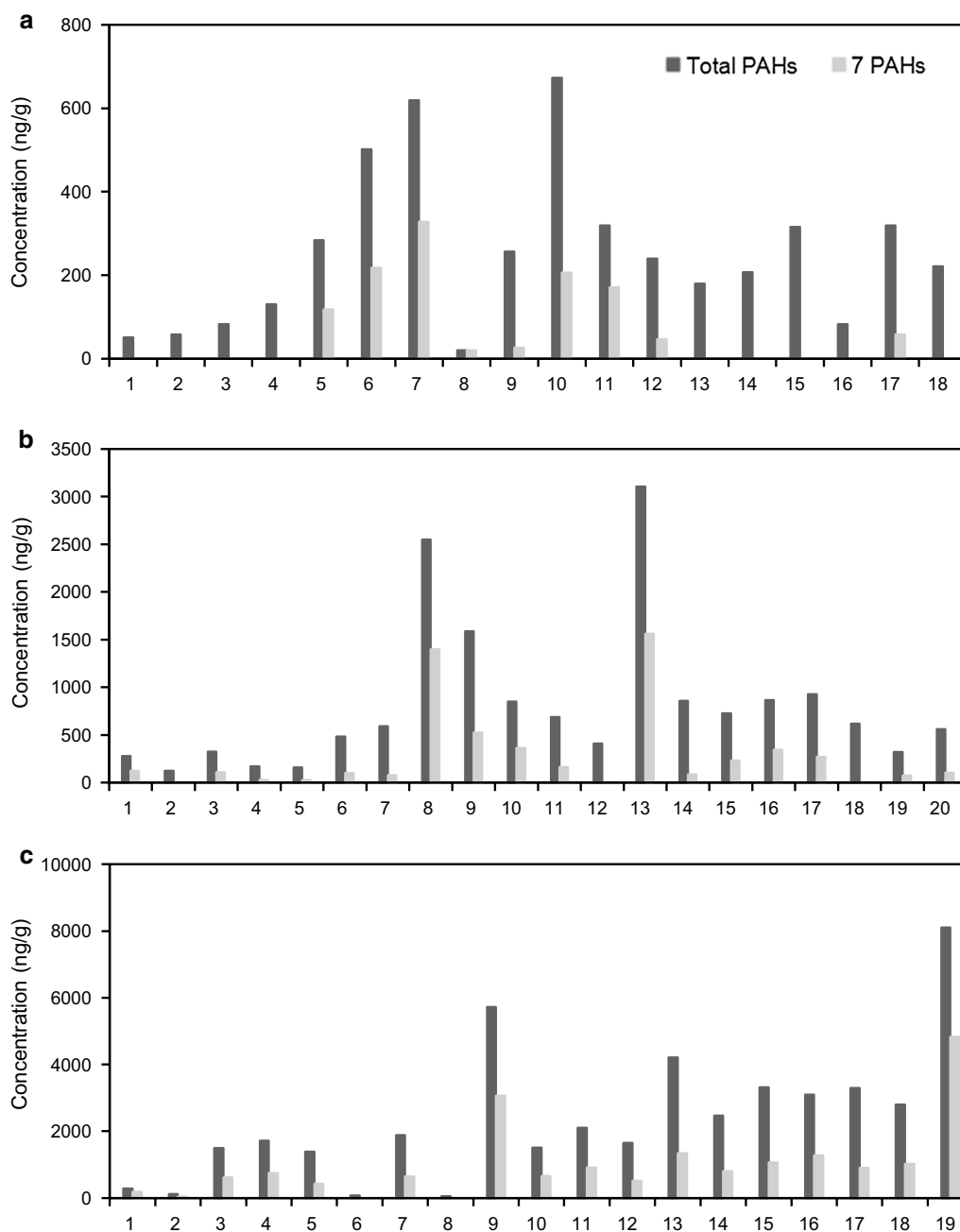


Fig. 1 Concentrations of the Σ_{16} and Σ_7 PAHs in the sample collected from sites in **a** Gyeonggi province, **b** Ulsan, and **c** Pohang

Ulsan, and 0.23–1.98 with a mean of 0.50 for Pohang. Except for 5 sites (sites S4, S5, S11, S14, and S18) in Ulsan and two sites (S2 and S11) in Pohang, analysis of most sites indicates that the PAHs in rice paddy soils were pyrogenically derived.

The ratios of Ant/(Ant + Phe) and Flt/(Flt + Pyr) were determined using the measured PAH concentrations from Ulsan and Pohang soils to identify the

pollution source. The diagnostic ratios were not calculated in the soils from Gyeonggi province because Ant, Flt, and Pyr were not detected except Phe. The cross plot of Ant/(Ant + Phe) against Flt/(Flt + Pyr) is shown in Fig. 4. Ratios of Ant/(Ant + Phe) > 0.1 and Flt/(Flt + Pyr) < 0.4 indicate pollution caused by heavy fuel burning. In contrast, ratios of Ant/(Ant + Phe) > 0.1 and Flt/(Flt + Pyr) > 0.5 correspond to a pyrogenic pollution

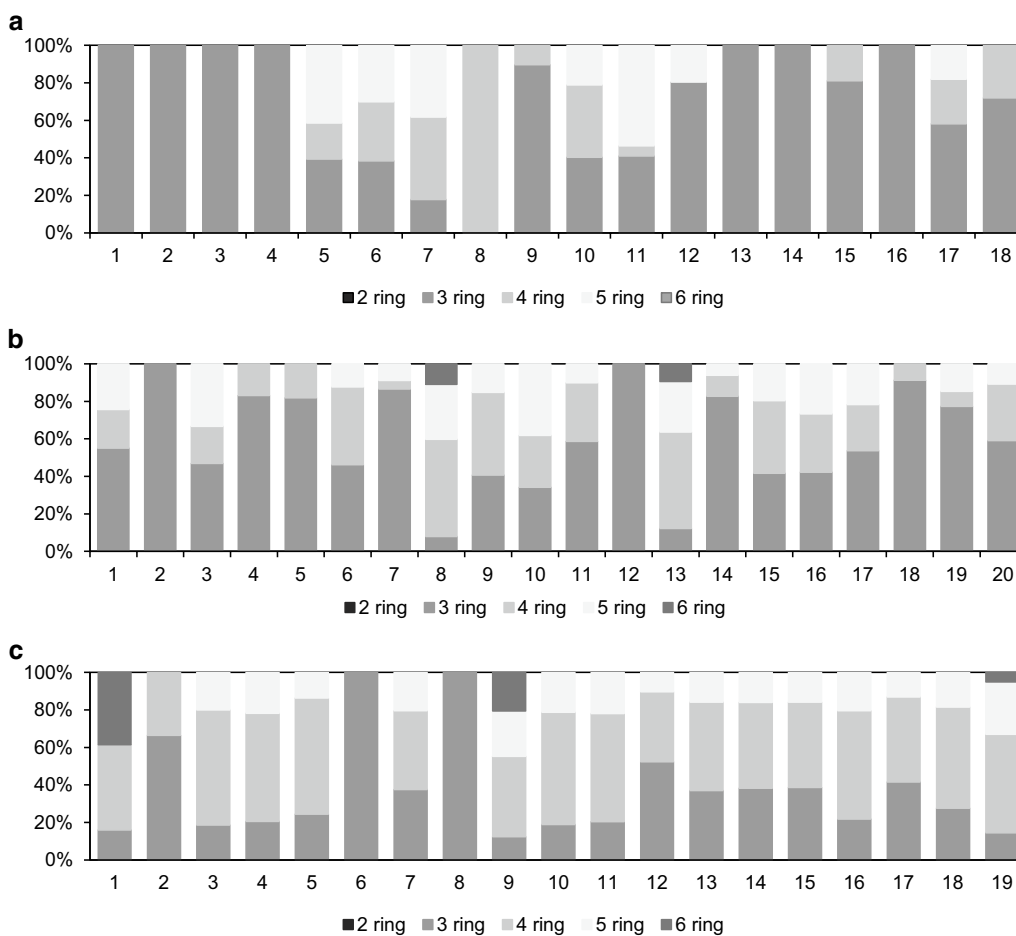


Fig. 2 Composition profiles of the 16 PAHs in samples taken from sites in **a** Gyeonggi province, **b** Ulsan, and **c** Pohang

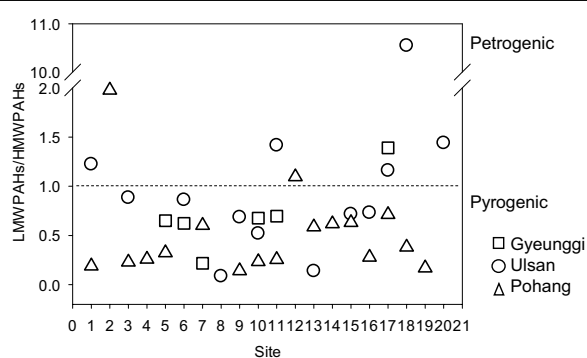


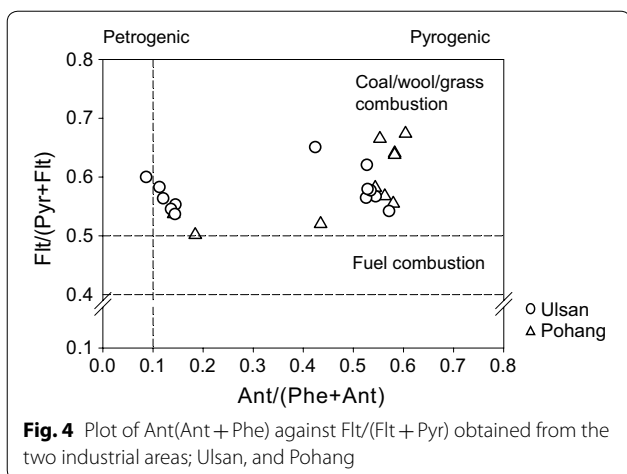
Fig. 3 Ratios of low molecular weight PAHs (LMWPAHs)/high molecular weight PAHs (HMWPAHs) in samples taken from sites in Gyeonggi province, Ulsan, and Pohang

sources originating from grass, wood, or coal combustion. When the Flt/(Flt + Pyr) ratio is between 0.4 and 0.5, petroleum combustion is the most likely pollution

source. In this study, the values of Flt/(Flt + Pyr) and Ant/(Ant + Phe) ranged from 0.5 to 0.7 in both cities. These results indicated that pyrogenic sources, including coal/wood/grass combustion, were the main sources of PAH contaminants.

Source identification using PCA

From the individual PAH (Phe, Ant, Flt, Pyr, BaA, Chr, BbF, and BkF) concentrations, PCA was performed to reduce the set of original variables and to extract a small number of potential factors for analysis of the relationship among the observed variables for further investigation of possible PAH sources. Two principal components (PC1 and PC2) were extracted with eigenvalues of > 1, constituting the majority (> 62%) of the total variance in the agricultural soils from the two industrial cities. The loading and score plots are shown in Fig. 5a, b. PC1 accounted for 45% of the total variance with a high load of BbF, Flt, Pyr, and BaA and PC2 accounted for 18% of the variance with a high load of BkF, BbF, Flt, and Pyr.



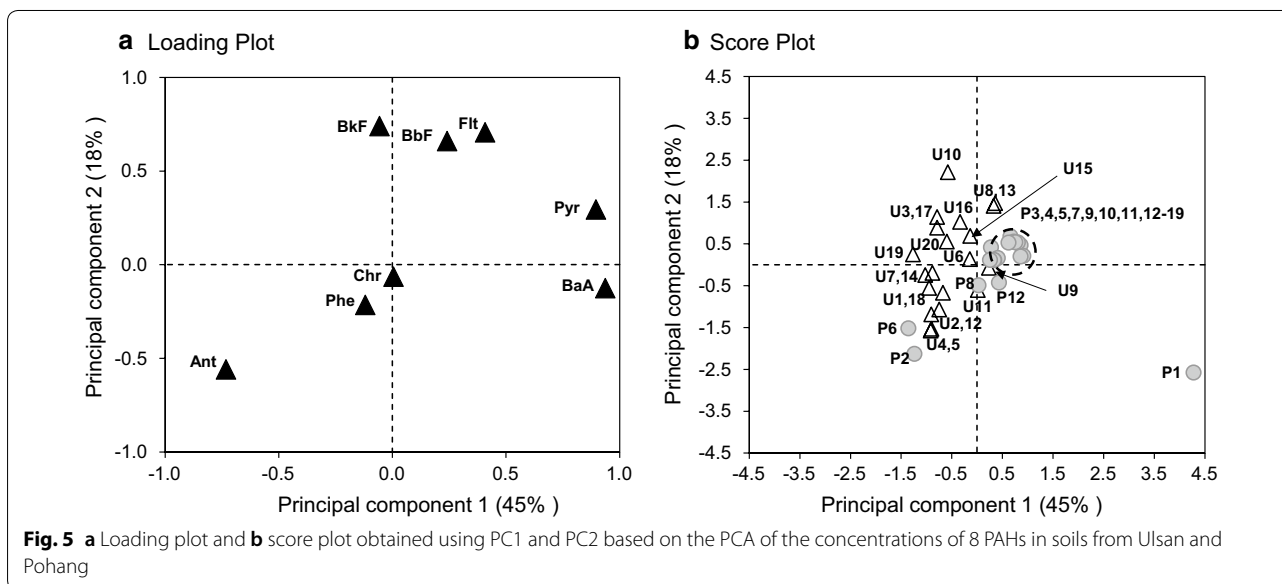
Discussions

The concentrations of the Σ_{15} and Σ_7 PAHs showed that the rice paddy soils from both industrial cities were significantly contaminated due to long-term industrial activities and large volumes of traffic compared to Gyeonggi province, which contains a couple of factories with more residential areas. The results presented herein are consistent with previous studies which showed that the PAHs distribution in soils reflected regional disparities derived from economic growth and population density on a continental scale in China [28]. For example, PAH concentrations in soils obtained from eastern China were higher compared to those sampled from western China due to differences in economic growth [29]. On a regional scale in China, the distribution of PAHs is mainly related

to the extent of urbanization. It was reported that PAH concentrations in soils declined along an urban–suburban–rural gradient.

The average levels of the Σ_{15} PAHs obtained in this study were compared with those reported in previous studies from other countries and the values are listed in Additional file 5: Table S4. The levels of PAHs in Gyeonggi province soils (19.53–672.93 ng/g) were lower than those of agricultural areas in other countries. In nine sampling sites out of 18 in the province, 7 carcinogenic PAHs in the agricultural soils were not detected, indicating that the soils were not possibly greatly contaminated with PAHs compared to other cities. The Σ_{15} PAH concentrations in Ulsan soils (125.01–3106.27 ng/g) were similar to rural areas (21.5–3350 ng/g) in Nanjing, China [14] and agricultural areas (1230–2950 ng/g) in Accra, Ghana [19], but were lower than those (228.6–14,722.1 ng/g) in Tianjin, China [30]. Pohang soils contained higher amount of PAHs (51.94–8106.21 ng/g) than rural area in Nanjing, China [14] and agricultural areas in Accra, Ghana [19], but were lower than those found in Tianjin, China [30]. The agricultural lands in Accra are severely contaminated with PAHs because they are located near roads with heavy traffic. This comparison indicated that Ulsan soils contained moderate PAH contamination compared to other countries (Additional file 5: Table S4). Pohang soils were severely contaminated with PAHs compared to other countries as well as other areas in South Korea due to the massive industrial complexes present in the city.

Gyeonggi province soils contained mostly 3-ring PAHs, but 3–5 ring compounds were dominant in the



contaminated soils in the two industrial regions, Ulsan and Pohang, suggesting that the sources of PAHs originated from the long-lasting industrial activities and heavy traffic derived from rapid industrial growth. Because of water solubility, 2-ring PAHs, and to a lesser extent 3-ring PAHs are more available for biological degradation and uptake [31]. On the other hand, due to low water solubility and volatility, 5- or more rings PAHs are primarily in solid state, bound to soils, sediments or air particulate matters [32]. In solid state, they are less available for biological degradation or uptake, causing increase in their persistency in the environment. It is likely that higher ring PAHs in the two city soils were accumulated from the long-term industrial activities.

The PCA results indicated that most sampling sites in Ulsan were contaminated with Ant, Phe, Chr, and BkF while most sites in Pohang were contaminated with Phe, Ant, Flt, Pyr, BaA, Chr, and BbF, associated with the adjacent steel industry complex. This is consistent with reports from previous studies that showed Phe, Ant, Pyr, Flu, BaA, BbF, DahA, BaP, and BkF are involved in the pyrolytic processes used in the steel industry and associated incomplete combustion emissions [33, 34]. It was reported that Flu, Pyr, BaA, BaP and Chr are derived from coal combustion [35], whereas Phe, Ant, Flu and Pyr are generally released from low-temperature pyrogenic processes [36]. In addition to the analysis of PAHs in the paddy soils, various pesticides are also needed to analyze in the paddy soils because sum of organic pollutants increase their toxicities on ecological concerns [37, 38].

In conclusion, the soil samples from Ulsan and Pohang in South Korea showed higher PAH concentrations than those from Gyeonggi province. The province has less industrial activities than two industrial cities. The major sources of the PAHs in Ulsan and Pohang soils might be due to the long-term industrial activities. Based on diagnostic isomeric ratios, the sources of PAHs in the agricultural soils were mainly coal combustion and pyrogenic processes. Considering that various pollutants including PAHs in soils can be mainly derived from particulate matters in atmosphere, contaminants in paddy soil also should be monitored for food safety for residents around the areas.

Additional files

Additional file 1: Table S1. Grid references of the sampling sites in Korea.

Additional file 2: Table S2. Method detection limits (MDLs), limits of quantification (LOQs), recoveries with relative standard deviations (RSDs) obtained from each PAH compound at the 100 µg/kg spiking levels using the developed method.

Additional file 3: Figure S1. Chromatograms of (A) standards of polyaromatic hydrocarbons (1 ppm), (B), Pohang (10th sample site), (C) Ulsan (13th sample site), and (D) Gyeonggi-Do (9th sample site).

Additional file 4: Table S3. Concentrations of all 16 PAHs and the 7 carcinogenic PAHs from Ulsan, Pohang, and Gyeonggi province in Korea.

Additional file 5: Table S4. The concentration range (mean) of the Σ 16 PAHs in soil samples reported in the literature from several other countries.

Authors' contributions

HJJ, YCK, and SHY carried out sampling and analysis of polycyclic aromatic hydrocarbons in soils. LK, HC, TOK, and SEL interpreted results and drafted the manuscript. All authors read and approved the final manuscript.

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Competing interests

The authors declare that they have no competing interests.

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