


# Concentration and distribution of polychlorinated biphenyls in rice paddy soils

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**Abstract** To monitor and evaluate the risk of polychlorinated biphenyls (PCBs) contamination in Pohang, Korea, the concentration and distribution of 29 PCBs in paddy soils were determined using high-resolution gas chromatography/high-resolution mass spectrometry. The overall concentrations of  $\Sigma_{29}$  PCBs in the paddy soils of the areas close to the heavily industrial city of Pohang (268–1833  $\text{pg g}^{-1}$  dw) were higher than those in the paddies from Anseong (106.6–222.6  $\text{pg g}^{-1}$  dw) in Korea. In Pohang, the major contributors to the  $\Sigma_{29}$  PCBs were the non-dioxin-like PCBs, including the PCBs 28, 52, 70, 101, 118, 138, 153, and 180, which correspond to 48–62% of the total PCBs. The toxic equivalency (TEQ) values obtained from the 12 dioxin-like PCBs from Pohang (0.03–1.03  $\text{pg}$

$\text{TEQ g}^{-1}$  dw) showed that PCB 126 contributed the highest toxicity, possibly posing a risk to the living organisms. The results of both principal component and cluster analysis based on the PCB homologue patterns demonstrated that each sampling site showed a similar PCBs contamination pattern, and Aroclor 1254, which is likely used by small and big steel factories, was identified as a major source of PCB contamination in Pohang.

**Keywords** Industrial area · Monitoring · Pohang · Polychlorinated biphenyls

## Introduction

Since the late 1920s, the synthesized polychlorinated biphenyls (PCBs) have been used in many industries, including petrochemical refineries, shipbuilding, and steel. The PCBs were defined as persistent organic pollutants (POPs) [1] because they do not easily degrade in the environment, causing adverse effect on the living organisms including humans. The PCB congeners are generally classified into two groups: dioxin-like (DL)-PCBs and non-dioxin-like (NDL)-PCBs. The toxicities of DL-PCBs have been exhaustively investigated, and the toxicity equivalency factors (TEF) values were assigned to DL-PCBs for health risk assessment. In contrast, the toxicological characterization of NDL-PCBs is still needed for the complete health risk assessment [2] because their high percentages are also found in food and human samples [3–5]. The NDL-PCBs 138, 153, and 180 are relatively less hydrophobic, because of which they are less strongly bound to the sediments, and are more easily available to the aquatic living organisms. According to a previous study, the exposure to the NDL-PCBs 101, 153, and 180 propelled the escalation of oxidative stress in chondrocytes by reducing their antioxidant ability [6].

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It was estimated that the amount of  $\Sigma_{22}$  PCBs released worldwide from 1930 to 2016 was 0.1 Mt [7]. Even though PCBs have never been produced in Korea, 0.3 tons of  $\Sigma_{22}$  PCBs was emitted into the atmosphere from 1946 to 2015 primarily because of the importation of the PCB-containing equipment to Korea for several industrial usages [7]. The previous studies on the sources of PCB contamination reported that the municipal solid waste incinerators and iron and steel industries were the major emitters of dioxins, such as PCBs, in Korea as well as other countries. A previous study demonstrated that the high levels of  $\Sigma_8$  PCBs in the air of Pohang were derived from the steel complex [8]. Furthermore, a study on the soil samples from the rural, city, and industrial sites in Ulsan, Korea, suggested that the high levels of PCBs might be strongly related to the surrounding industrial complexes, including the shipbuilding industry and petrochemical refinery [9]. However, to the best of our knowledge, only a few studies have been carried out to determine the concentrations of PCBs in the paddy soils of Korea, even though rice is the main staple food of Korea. Given that the soil is an essential reservoir of atmospherically deposited PCBs, which might be transported into the food web, the determination of the PCB levels and distributions in paddy soils is urgently required to protect human health and living organisms in the heavily industrial areas. According to a recent study on PCBs in the paddy soils nearby a transformer oil-related factory in Anseong, Korea, the  $\Sigma_{29}$  PCBs concentrations ranged from 106.65 to 222.67  $\text{pg g}^{-1}$  dw [10], which were not the high-risk values.

The PCB levels in the soils nearby industrial areas were investigated by many researchers around the world, because the vegetables or grains grown in an area are deeply linked to the public health [11, 12]. It was reported that the  $\Sigma_{33}$  PCB concentrations in wheat, rice, soil, and air from Pakistan were 0.15–2.22, 0.05–9.21, 0.70–30.5  $\text{ng g}^{-1}$  dw, and 41–299  $\text{pg m}^{-1}$ , respectively [11].

The objectives of this research were (1) to perform the monitoring of PCBs in the rice paddy soils from Pohang, one of the highly polluted industrial cities in Korea, by determining the concentration of 29 PCB congeners and homologue patterns, (2) to perform the risk assessment using the TEF system, and (3) to uncover the PCB contamination sources using the principal component analysis (PCA) and clustering analysis (CA).

## Materials and methods

### Sample preparation and instrumental analysis

Rice paddy soils (0–10 cm) were sampled from 20 sites in Pohang City, Gyeongsang Province, Korea. The sampling locations in Pohang are shown in Fig. S1, and the grid

references are listed in Table S1. The world's fourth largest steel producer, POSCO, was established in Pohang with a population of 0.52 million in 1968. Now, Pohang is one of the biggest industrial cities with many small and big steel makers in Korea.

Before being passed through a 2-mm sieve, the soil samples were air-dried at the room temperature. The Soxhlet extraction was performed on the 10-g soil samples with the optimized sample preparation procedure and gas chromatography/high-resolution mass spectrometry described in our previous study. The United States Environmental Protection Agency (US EPA) criteria (method 1668C) were used to analyze each PCB congener. The quantification of the chemicals was carried out by using the isotope dilution method with calibration standards (EC9605-CVS and 68C-CVS, Wellington Laboratories, Canada). The compounds of interest were 12 DL-PCBs (77, 81, 126, 169, 105, 114, 118, 123, 156, 157, 167, and 189), 6 indicator PCBs (28, 52, 101, 138, 153, and 180), and 14 NDL-PCBs (33, 44, 52, 70, 81, 114, 118, 128, 170, 187, 194, 195, 199, and 206). The recovery rates of the surrogate standards ranged from 62.2 to 110.8%.

### Calculation for toxic equivalency quantities (TEQ)

The TEF system was used for the risk assessment of PCB congeners found in this study. The overall toxicity, or the toxic equivalency (TEQ), of a mixture was calculated by adding the concentrations of all congeners ( $C_i$ ) and multiplying by its  $\text{TEF}_i$  [13].

$$\text{TEQ} = \Sigma[C_i] \times \text{TEF}_i$$

### Statistical analysis

Two hypotheses were developed for this study. First, each sampling site from Pohang has the same tendency of PCB contamination. Second, the PCB contaminations were derived from the Aroclors 1254 and 1260 used in the industrial activities in Pohang. These two hypotheses were verified based on the results derived from both PCA and CA with a statistical package for the social sciences (IBM SPSS 23, USA). The score and loading values obtained from the PCA analysis were used for visualization with the score and loading plots. The profiles of each PCB homologue group (tri-, tetra-, penta-, hexa-, hepta-, octa-, and nonaCBs) obtained from this study and those of the Aroclors 1242, 1254, and 1260 from a previous study [14] were used for the statistical analyses. The different rates of the Aroclor profiles produced by different companies were utilized for the statistical analyses. The concentration of

each homologue was normalized by the concentration of  $\Sigma_{29}$  PCBs for each sample. The identification of the PCB sources in Pohang was based on the data from PCA and CA. To assess the differences between the PCB concentrations of the sites and cities, the rank-sum tests were performed using SigmaPlot 12.5 (Systat Software, USA). Differences were considered to be statistically significant if the “*p* value” was less than 0.05.

**Results**

**$\Sigma_{29}$  PCBs, 7 PCBs, and toxic equivalency quantities of 12 DL-PCBs**

The concentrations of  $\Sigma_{29}$  PCBs (Table S2) at 20 sites in Pohang ranged from 268 to 1833 pg g<sup>-1</sup> dw. Site 10 (1838 pg g<sup>-1</sup> dw) and site 17 (1834 pg g<sup>-1</sup> dw) showed the highest PCB concentrations, and site 14 (1281 pg g<sup>-1</sup> dw) and site 12 (1248 pg g<sup>-1</sup> dw) showed the second highest PCB concentrations. Out of the 29 PCB congeners determined, the PCBs 28, 52, 70, 101, 118, 138, 153, and 180 were predominating (mean: 39–80 pg g<sup>-1</sup> dw) in the most sampling sites. These congeners, except for PCB 70, were known to be the indicator PCBs. The indicator PCBs detected in this study accounted for 48–63% of the  $\Sigma_{29}$  PCBs, except for site 8 (34%).

The TEQ values for the 12 DL-PCBs at each site from Pohang were calculated based on the TEQ calculation formulae. The TEQ values (Table 1) ranged from 0.03 to 1.03 pg TEQ g<sup>-1</sup> dw. Site 7 showed the highest TEQ value (1.03 pg TEQ g<sup>-1</sup> dw) and site 12 the second highest (0.89 pg TEQ g<sup>-1</sup> dw).

**Distribution and possible source identification of PCBs**

Among the homologues determined for each sampling site from Pohang (Fig. 1), the pentaCBs (mean 21.0%) and hexaCBs (mean 25.3%) were the most dominant, followed by the tetraCBs (mean 18.3%) and triCBs (mean 18.0%). Based on these PCB homologue profiles, the PCB contamination in Pohang was found to be more associated with Aroclor 1254 than with Aroclors 1260 and 1242. This was supported by the results of PCA (Fig. 2).

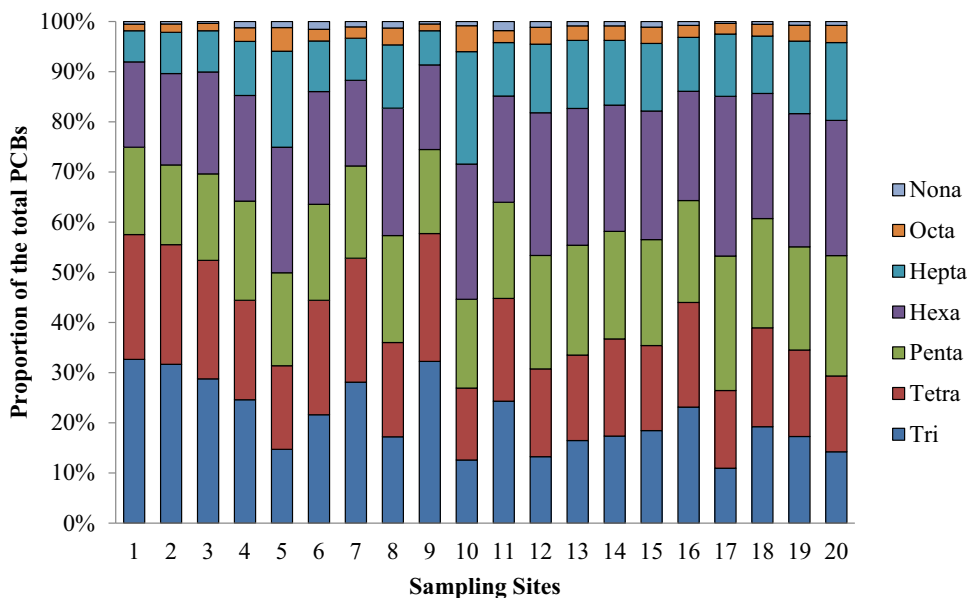
**Discussion**

Recently, many unexpected compounds in the environments have been determined using advanced instruments [15, 16], and in this study, all PCBs were determined using high-resolution mass spectrometry (HR-MS) in conjunction with gas chromatography.

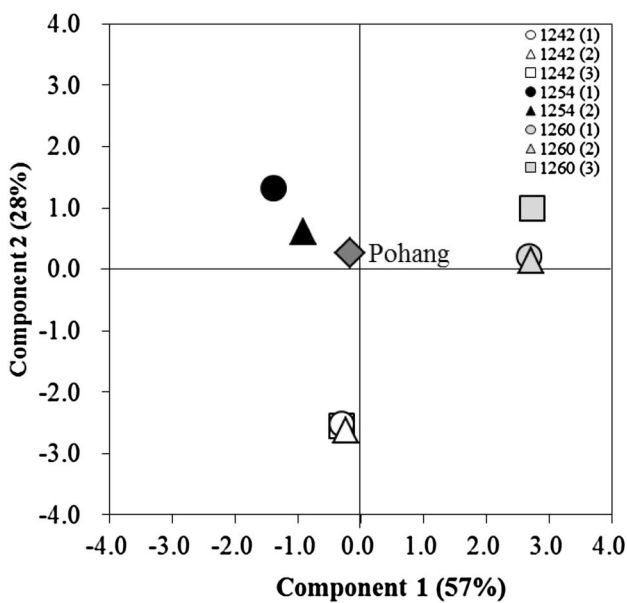
**Table 1** Toxic equivalency quantities of 12 DL-PCBs determined in soil samples from Pohang

PCBs	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20
<i>Non-ortho-substituted PCBs</i>																				
77	0.0003	0.0003	0.0005	0.0007	0.0009	0.0008	0.0015	0.0009	0.0004	0.0014	0.0008	0.0027	0.0005	0.0024	0.0009	0.0017	0.0013	0.0012	0.0006	0.0014
81	0.0001	0.0001	0.0001	0.0002	0.0002	0.0002	0.0007	0.0000	0.0001	0.0002	0.0001	0.0008	0.0001	0.0004	0.0002	0.0004	0.0003	0.0002	0.0001	0.0002
126	0.0307	0.0474	0.0423	0.1043	0.1200	0.1047	0.9434	0.1361	0.0464	0.2366	0.1429	0.7937	0.0910	0.2936	0.1416	0.2981	0.2065	0.2059	0.0950	0.2783
169	0.0000	0.0056	0.0048	0.0124	0.0126	0.0000	0.0843	0.0168	0.0057	0.0167	0.0113	0.0810	0.0099	0.0313	0.0139	0.0295	0.0090	0.0150	0.0000	0.0197
<i>Mono-ortho-substituted PCBs</i>																				
105	0.0003	0.0003	0.0004	0.0006	0.0006	0.0004	0.0004	0.0010	0.0003	0.0016	0.0008	0.0017	0.0005	0.0015	0.0008	0.0014	0.0024	0.0011	0.0005	0.0011
114	0.0000	0.0000	0.0000	0.0000	0.0000	0.0001	0.0000	0.0000	0.0000	0.0000	0.0001	0.0000	0.0000	0.0000	0.0000	0.0000	0.0002	0.0001	0.0000	0.0001
118	0.0005	0.0005	0.0007	0.0013	0.0012	0.0007	0.0006	0.0019	0.0006	0.0035	0.0016	0.0037	0.0010	0.0037	0.0018	0.0029	0.0058	0.0023	0.0011	0.0022
123	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0001	0.0000	0.0001	0.0000	0.0001	0.0000	0.0000	0.0001	0.0000	0.0000	0.0000
156	0.0001	0.0001	0.0001	0.0002	0.0002	0.0001	0.0002	0.0003	0.0001	0.0007	0.0003	0.0007	0.0002	0.0005	0.0003	0.0005	0.0013	0.0004	0.0002	0.0004
157	0.0000	0.0000	0.0000	0.0001	0.0000	0.0000	0.0001	0.0001	0.0000	0.0001	0.0001	0.0002	0.0000	0.0002	0.0001	0.0001	0.0002	0.0001	0.0000	0.0001
167	0.0000	0.0000	0.0000	0.0001	0.0001	0.0000	0.0001	0.0001	0.0000	0.0003	0.0001	0.0003	0.0001	0.0002	0.0001	0.0002	0.0005	0.0002	0.0001	0.0002
189	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0001	0.0000	0.0000	0.0001	0.0000	0.0001	0.0000	0.0001	0.0000	0.0001	0.0001	0.0000	0.0000	0.0001
Σ	0.0320	0.0543	0.0489	0.1200	0.1358	0.1071	1.0314	0.1573	0.0535	0.2614	0.1581	0.8849	0.1034	0.3340	0.1598	0.3349	0.2277	0.2264	0.0978	0.3037

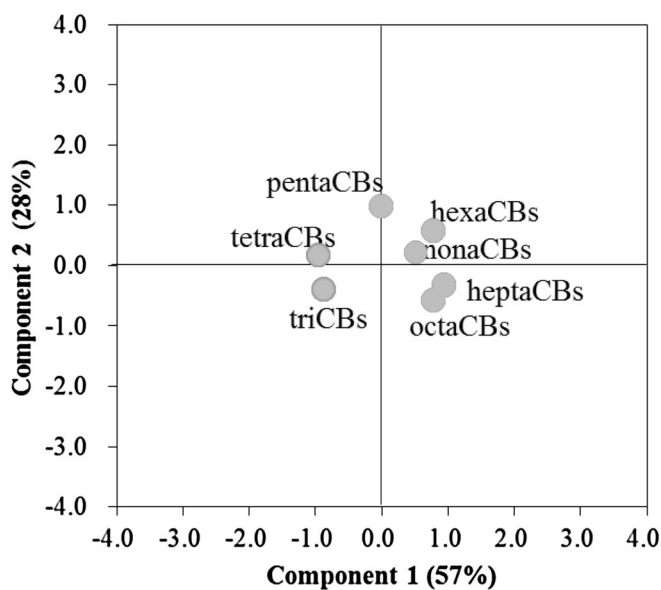
**Fig. 1** Location of sampling sites from Pohang in Korea



**(A) Score Plot**



**(B) Loading Plot**



**Fig. 2** (A) Score plot and (B) loading plot of PCA obtained using the homologue profiles from Pohang and Aroclors 1242 (1, 2, and 3), 1254 (1 and 2), and 1260 (1, 2, and 3) studied by Shin and Kim [23]

**$\Sigma_{29}$  PCBs, 7 PCBs, and toxic equivalency quantities of 12 DL-PCBs**

Of the 20 sampling sites, the mean  $\Sigma_{29}$  PCB concentration of sites 11–20 (group 2) ( $940 \text{ pg g}^{-1} \text{ dw}$ ) was higher than that of the remaining sites (group 1) ( $550 \text{ pg g}^{-1} \text{ dw}$ ); this was supported by the statistical analysis (Mann–Whitney rank-sum test,  $p < 0.05$ ) (Fig. S1). It is believed that this finding was strongly linked with the location of each site

because the sites in group 2 were located near the steel industry complex, compared with those in group 1.

The residual concentrations of the  $\Sigma_{29}$  PCBs in the soil samples from Pohang ( $268\text{--}1833 \text{ pg g}^{-1} \text{ dw}$ ) were much higher than those ( $107\text{--}223 \text{ pg g}^{-1} \text{ dw}$ ) from Anseong [10]. The  $\Sigma_{29}$  PCB concentrations from Pohang were lower than the high-risk PCB levels for residential ( $0.23 \text{ mg kg}^{-1}$ ) and industrial ( $0.94 \text{ mg kg}^{-1}$ ) soil setup by US EPA [17].

The major contributors to the  $\Sigma_{29}$  PCBs in Pohang were NDL-PCBs, including PCB 28, 52, 70, 101, 118, 138, 153, and 180, which corresponded to 48–62% of the total. Combined contribution of PCB 153 and 138 to the sum of the 7 indicators was more than 30%, which was consistent with the previous studies showing the high percentages of the congeners in food groups such as fish [18, 2]. Brazova et al. demonstrated that the PCBs 153 and 138 were the biggest contributors to the sum of 6 indicators in European catfish. Another study also showed that PCB 153 contributed 53% to the sum of NDL-PCBs in catfish [4], because the PCB mixture released by various industrial activities surrounding the lake reflected fish contamination. In this study, the congener patterns of soil contamination should also reflect the presence of the congeners in the atmosphere, closely associated with unintentional emissions due to the historical use of commercial PCB mixtures by the steel industry. The presence of abundant congeners was also derived from their persistent properties (approximately 10-year half-life for PCB 101, 7 years for PCB 118, and 18 years for both PCBs 138 and 153) [19].

The DL-PCB congener distribution in the soil samples showed the predominance of PCBs 118, 105, 156 and 77 (49, 23, 9, and 9%, respectively) in terms of concentration, which was in agreement with the previous studies on PCBs in fish [4]. Even though the concentration of PCB 126 was low, the congener contributed highly to the TEQ values at all soil sampling sites from Pohang (90%). The DL-PCB TEQ values in Pohang (0.03–1.03 pg TEQ g<sup>-1</sup> dw) were higher than those in Anseong (0.04–0.11 ng TEQ kg<sup>-1</sup> - dw), but much lower than those of the agricultural soils in Delhi, India (0.01–105.54 pg g<sup>-1</sup>) [20], as well as the WHO threshold level (20 ng TEQ kg<sup>-1</sup>).

The measured values of 0.03–1.03 pg TEQ g<sup>-1</sup> are much lower than the physiological effects of PCBs in livers of aquatic mammals as 160–1400 pg TEQ g<sup>-1</sup> [21]. Kannan et al. [21] measured the toxicity level of the PCBs in seals, European otters, and mink. However, they suggested that the dietary threshold levels could be 1.4–1.9 pg TEQs g<sup>-1</sup>. On the other hand, [22] also showed that the TEQ level as 10 ng TEQ (BaP) g<sup>-1</sup> caused DNA damage in earthworms (*Eisenia fetida*). With this regard, the TEQ levels determined in Pohang seem to be safe to animals including humans because of lower levels than the previous studies.

### Distribution and possible source identification of PCBs

The pentaCBs and hexaCBs were detected as the two most dominant homologues in the soil samples from Pohang, which was consistent with the results from Anseong [10]. The triCBs and tetraCBs also presented high ratios,

indicating the possible degradation of the highly chlorinated PCBs. However, the PCB homologue pattern was more associated with Aroclor 1254 than with Aroclors 1260 and 1242, which was confirmed by the data obtained from PCA (Fig. 2) and CA (Fig. S2). For source identification, the proportions of each homologue group identified in the soil samples from Pohang and commercial products (Aroclors 1242, 1254, and 1260) [14] were compared. The technical mixtures used for the statistical tests were known to be the transformer oils mostly consumed in the Korean power stations [23]. According to the result of PCA (Fig. 2) obtained from the PCB profiles, the variances of the first and second principal components were 57 and 28%, respectively. The score plot demonstrated the first hypothesis that all soil samples were closely related to each other and with Aroclor 1254 (1 and 2), which was confirmed by CA. The dendrogram indicated that Pohang and Aroclor 1254 (1 and 2) were classified into group 1. The Aroclors 1260 (1, 2, and 3) and 1242 (1, 2, and 3) were classified into groups 2 and 3, respectively, because the PCB profiles of each Aroclor with the same number were slightly different [24]. The PCA loading plot also confirmed that the soil samples and Aroclor 1254 were characterized with tetra-, penta-, and hexaCBs. These plots showed that the octaCBs contributed highly to Aroclor 1260, and the triCBs and tetraCBs were big contributors to Aroclor 1242.

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### Compliance with ethical standards

**Conflict of interest** The authors have declared that no competing interests exist.

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