

## Polychlorinated Dibenzo-*p*-dioxins and Polychlorinated Dibenzofurans in Marine Sediments from Mokpo Coastal Water of Korea

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Concentrations of polychlorinated dibenzo-*p*-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs) were determined in marine sediments from Mokpo coastal water of Korea. Total and toxic equivalent (TEQ) concentrations of PCDD/Fs ranged from 19.4 to 175 pg/g dry weight and ranged from 0.195 to 2.87 pg WHO-TEQ/g dry weight, respectively. These PCDD/F concentrations were below the safety sediment value (20 pg TEQ/g dry weight) of chronic toxicity. A highly significant correlation between the concentrations of PCDD/Fs and total organic carbon (TOC) in marine sediments was observed. Overall PCDD/F concentrations in the sediments were elevated in rivers and at inner locations close to harbors, indicating that these contaminants derived from local discharges of human activities. PCDD/F concentrations measured in our study were lower than those in several industrialized areas in Korea. Non-parametric multidimensional scaling (MDS) ordination showed that combustion processes from industrial complexes are the major source of PCDD/Fs in Mokpo coastal water.

Key words: PCDD/Fs; Marine sediment, Mokpo coastal water; Multidimensional scaling (MDS); Combustion process

### Introduction

Mokpo, located in the southwest corner of Korea, has Muan in the north and a number of islands in the west coast. Mokpo coast is characterized by muddy flat with wide range of tides. Fresh water flows into this coast mainly from the Yeongsan River at the eastern end of the coast. The Yeongsan, Yeongam and Geumho embankments have been constructed for drainage since 1981 (Kim et al., 1994). The inner part of the coast has the Daebul industrial complex, which was founded in 1996 and produces chemical products, nonferrous metals, machinery, and food products (Yoon, 2001). Samho industrial complex located in the outer part of the coast comprised of shipyards and machine factories. The rapid industrialization of this area can be accompanied by a significant environmental deterioration that may lead to a variety of social and health problems.

Polychlorinated dibenzo-*p*-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs) are widely distributed contaminants found in various environmental zones as well as in humans (Hutzinger et al., 1985). These contaminants are released into the environment by various combustion and manufacturing processes, such as municipal solid waste incinerators (MSWIs) (Olie et al., 1977), steel mills (Tysklind et al., 1989), automobiles (Marklund et al., 1987), chemical production processes (Hutzinger et al., 1985) and chloralkali processes (Kannan et al., 1998). PCDD/Fs have been shown to cause developmental toxicity, cancer, and endocrine disruption (Kociba et al., 1976).

PCDD/Fs derived from various sources can be transported to aquatic environments via riverine inputs and atmospheric deposition (Moon et al., 2005). Due to their lipophilic and hydrophobic characteristics, PCDD/Fs adsorb strongly to suspended particles and then deposited to bottom sediments.

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Consequently, analysis of sediments can provide information on recent sources of these contaminants in the marine environment (Jimenez et al., 1998; Hilscherova et al., 2003; Masunaga et al., 2003). The existence of PCDD/Fs and related compounds in marine sediments from industrialized and heavily populated areas in Korea is an issue of concern (Im et al., 2002; Koh et al., 2004; Ryoo et al., 2005). Until now, the studies on environmental contamination in Mokpo coastal waters have only been focused on the water quality (Yoon, 2001) and trace metals (Kim and Sin, 2002). To our knowledge, the present study is the first effort to investigate concentrations and potential sources of toxic organic contaminants such as PCDD/Fs in Mokpo coastal waters. This study was conducted as part of a nationwide survey on persistent organic pollutants (POPs) in Korea's Coa-

stal environment. The objective of this study was to describe the concentrations, congener patterns and sources of PCDD/Fs in marine sediments from Mokpo coastal waters of Korea.

## Materials and Methods

### Sampling strategy

Surface sediments (0-4 cm) were sampled at 29 locations from Mokpo coast in March 2006 (Fig. 1). Sediment samples were collected systematically from the inner to the outer parts of the coast (M1-M13), harbor zone (H1-H4), Yeongsan River (R1), wastewater treatment plant (WWTP) and some streams (R2-R5) of the Mokpo coast. Our sampling area covered the locations near Samho industrial complex (S1-S4) and small industrial complex (C1-C2) that

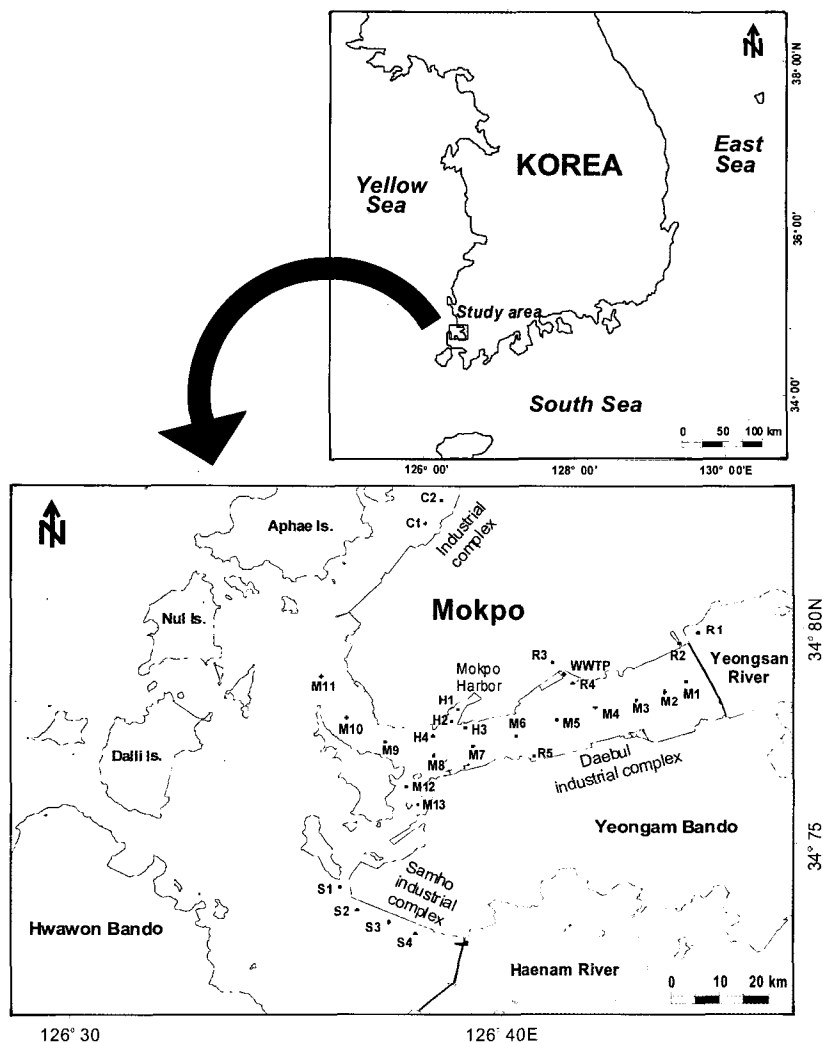


Fig. 1. Map showing sampling locations (enlarged map) of marine sediments from Mokpo coastal water in Korea.

are located in the northern part of the Mokpo City. Surface sediments were sampled by using a boxcorer deployed from a research vessel. The collected samples were wrapped individually in aluminum foil and then frozen immediately in a refrigerator on board the vessel. Marine sediment samples were transported to the laboratory and were kept in a freezer at  $-20^{\circ}\text{C}$  until analysis.

### Sample preparation

Marine sediments were freeze-dried and passed through a 2 mm sieve. Detailed descriptions of sample extraction and clean-up procedures have been reported elsewhere (Moon et al., 2005; 2006). Briefly, 20 g of sediment samples were extracted in a Soxhlet apparatus using 200 mL of 10% acetone (ultra residue analysis, J. T. Baker, Phillipsburg, NJ, USA) in toluene (ultra residue analysis, J. T. Baker, Phillipsburg, NJ, USA) for 24 h after spiking with  $^{13}\text{C}$ -labelled internal standards of PCDD/Fs (EPA-1613LCS, Wellington Laboratories Inc., Guelph, Ontario, Canada). The extracts were pre-cleaned on a multi-layer silica gel (70-230 mesh, Neutral, Merck) column containing  $\text{AgNO}_3$ -silica gel,  $\text{H}_2\text{SO}_4$ -silica gel and KOH-silica gel with 160 mL of hexane (dioxin analysis grade, Wako, Japan). The eluted fractions were cleaned up on an activated neutral alumina (70-230 mesh, Neutral, Merck) column with successive portions of 3% methylene chloride (dioxin analysis grade, Wako, Japan) in hexane and 50% methylene dichloride in hexane. The second fraction was concentrated to less than 1 mL, and left at room temperature for one or two days to evaporate to dryness. The residue was dissolved with 50  $\mu\text{L}$  of *n*-nonane (Pesticide residue analysis, Fluka, Switzerland) and determined for PCDD/Fs.

### Instrumental analysis

Detailed descriptions of instrumental analysis have been described elsewhere (Moon et al., 2005; 2006). Briefly, identification and quantification were performed by using a high-resolution gas chromatograph interfaced with high-resolution mass spectrometer (HRGC/HRMS; JMS 700D, JEOL, Tokyo, Japan). The capillary column used was an SP 2331 (60 m length, 0.25 mm i.d., 0.2  $\mu\text{m}$  film thickness, Supelco) for the separation of tetra- to hexa-CDD/Fs. Hepta- to octa-CDD/Fs were analyzed using a DB5-MS (60 m length, 0.25 mm i.d., 0.25  $\mu\text{m}$  film thickness; J&W Scientific) capillary column.

The total organic carbon (TOC) content in marine sediments was analyzed using a CHN Elemental Analyzer (PerkinElmer, Model 2400; Boston, MA,

USA), after removal of calcium carbonate with 1 N HCl. Grain size analysis was performed using a particle size analyzer (Sympatec Windox4; Sympatec GmbH, Clausthal Zellerfeld, Germany).

### Quality assurance/quality control (QA/QC)

All of the labeled internal standards were detected with no interferences. Solvents injected before and after the injection of standards showed negligible contamination or carryover. Procedural blanks were processed in the same way as the samples. Blanks did not contain quantifiable amounts of the target compounds. The limit of detection was calculated as three times the signal-to-noise ratio, which ranged from 0.008 to 0.04 pg WHO-TEQ/g dry weight. The recoveries of spiked internal standards were  $82 \pm 19\%$  (mean  $\pm$  standard deviation) for toxic congeners of PCDD/Fs in all of the sediments. The concentrations of toxic equivalents (TEQs) for PCDD/Fs are given using World Health Organization-toxic equivalency factors (WHO-TEF) (Van den Berg et al., 1998).

## Results and Discussion

### Sediment characteristics

Grain size and TOC in marine sediments from Mokpo coastal waters are summarized in Table 1. Sediments collected in our study were fine clay to fine sand, with grain sizes of 5.1-17.6  $\mu\text{m}$ . Except some locations such as Stations R1, R2 and M13, all of the sediment samples showed homogenous distribution of grain size with values less than 10  $\mu\text{m}$ . The TOC in marine sediments ranged from 0.4 to 2.4% with a mean value of 0.8%. Station R2 (Samhyang River; 2.4%) had the highest content of TOC in our study, however other locations contained low levels of TOC with values less than 1.0%. A significant correlation between the concentrations of PCDD/F and TOC in marine sediments was observed ( $r^2=0.723$ ,  $p=0.0001$ ) (Fig. 2). This is because PCDD/Fs are associated with organic-rich particles in marine coastal waters. Koh et al. (2004) reported a highly significant correlation ( $r^2=0.90$ ,  $p=0.004$ ) between PCDD/F and TOC in the Hyeongsan River sediments from Yeongil Bay. Our finding suggests that sediment characteristics such as TOC can be an important factor governing the levels of PCDD/Fs in marine coastal waters.

### Spatial distribution of PCDD/Fs

Concentrations of 2,3,7,8-substituted and total PCDD/Fs in marine sediments from Mokpo coastal waters are summarized in Table 1. Toxic congeners of

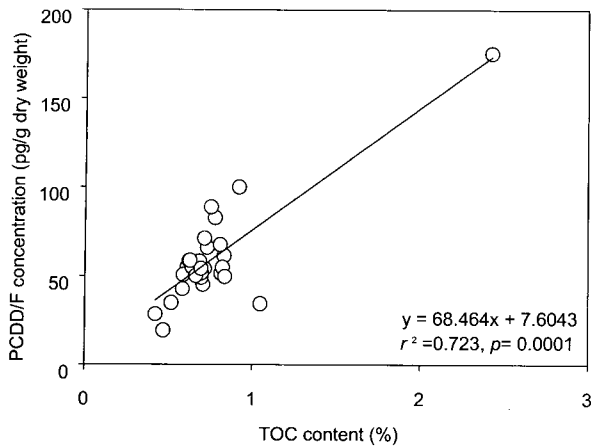


Fig. 2. Correlation between the concentrations of PCDD/F and TOC in marine sediments from Mokpo coastal water in Korea.

PCDD/Fs were detected in all the sediment samples taken from study area, indicating that PCDD/Fs are widespread contaminants in coastal waters. Total concentrations of PCDD/Fs in marine sediments from Mokpo coastal waters ranged from 19.4 to 175 pg/g dry weight with an average of 59.6 pg/g dry weight. The TEQ concentrations of PCDD/Fs in marine sediments ranged from 0.195 to 2.87 pg TEQ/g dry weight with an average of 0.755 pg TEQ/g dry weight. Evers et al. (1996) has proposed 20 pg TEQ/g dry weight as a safety sediment value of chronic toxicity, to evaluate a toxic potency for marine coastal environment. In this study, the concentrations of PCDD/F in all of the sediment samples from Mokpo coastal waters were below the toxicity value, indicating that

this coast is relatively safe with regard to PCDD/F contamination.

The overall PCDD/F concentrations in the sediments from Mokpo coastal waters were elevated in rivers and at inner locations close to the harbors (Fig. 3). However, in general, there was no great difference in PCDD/F contamination among the sampling locations in the surveyed coast. In particular, most of the sediments showed low PCDD/F concentrations, less than 1 pg TEQ/g dry weight except those from some locations. The highest TEQ concentration was found at Station H1, which is located at inner part of Mokpo Harbor and decreased gradually toward the outer part of the bay (H1-H4). Station R2 (Samhyang River) had a relatively high concentration of PCDD/Fs, indicating that this river can be the main route for PCDD/F contamination in Mokpo coastal waters. However, sediments from the Yeongsan River had relatively a low concentration (0.41 pg TEQ/g dry weight), suggesting that this river is not the main source of PCDD/F contamination in Mokpo coast. The sediments (Stations WWTP, C1-C2, S1-S4) collected near WWTP and some industrial complexes in Mokpo coast showed relatively low PCDD/F concentrations, although the importance of industrial complexes has been emphasized as a potential source of PCDD/F contamination in some studies (Moon et al., 2002a; Koh et al., 2004).

A few studies have reported PCDD/F contamination in river and marine sediments from Korean coastal waters. The PCDD/F concentrations (0.195-2.87 pg TEQ/g dry weight) in our study were comparable with those in sediments from Ulsan Bay,

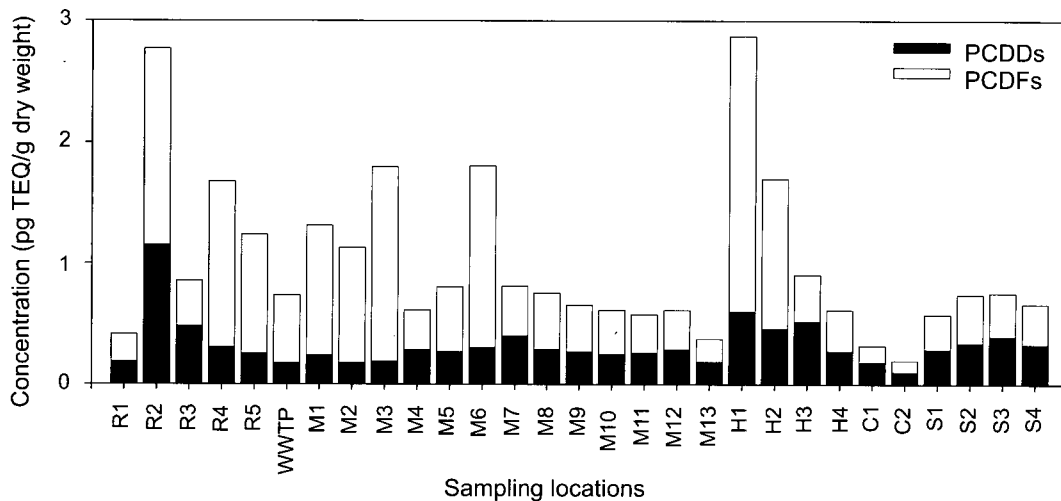


Fig. 3. Distribution of PCDD and PCDF concentrations in marine sediments from Mokpo coast in Korea. Detailed sampling locations are described in Fig. 1.

Table 1. Summary of sample characteristics and concentrations of PCDD/Fs in marine sediments from Mokpo coastal water in Korea

	Min	Max	Median	Mean $\pm$ SD
Grain size ( $\mu\text{m}$ )	5.1	17.6	6.0	7.3 $\pm$ 3.2
TOC (%)	0.4	2.4	0.7	0.8 $\pm$ 0.3
<b>Toxic 2,3,7,8-substituted congeners (pg TEQ/g dry weight)</b>				
2,3,7,8-TCDD	0.013	0.436	0.045	0.062 $\pm$ 0.084
1,2,3,7,8-PeCDD	0.048	0.471	0.132	0.145 $\pm$ 0.081
1,2,3,4,7,8-HxCDD	0.005	0.058	0.011	0.015 $\pm$ 0.010
1,2,3,6,7,8-HxCDD	0.011	0.075	0.022	0.026 $\pm$ 0.013
1,2,3,7,8,9-HxCDD	0.004	0.070	0.032	0.032 $\pm$ 0.013
1,2,3,4,6,7,8-HpCDD	0.003	0.185	0.031	0.049 $\pm$ 0.044
OCDD	0.007	0.029	0.019	0.018 $\pm$ 0.005
2,3,7,8-TCDF	0.001	0.003	0.002	0.002 $\pm$ 0.001
1,2,3,7,8-PeCDF	0.005	0.062	0.020	0.022 $\pm$ 0.011
2,3,4,7,8-PeCDF	0.038	0.490	0.117	0.133 $\pm$ 0.087
1,2,3,4,7,8-HxCDF	0.005	1.43	0.046	0.228 $\pm$ 0.425
1,2,3,6,7,8-HxCDF	0.003	0.347	0.035	0.056 $\pm$ 0.071
1,2,3,7,8,9-HxCDF	0.004	1.02	0.034	0.130 $\pm$ 0.240
2,3,4,6,7,8-HxCDF	0.005	0.125	0.022	0.024 $\pm$ 0.022
1,2,3,4,6,7,8-HpCDF	0.004	0.123	0.022	0.030 $\pm$ 0.029
1,2,3,4,7,8,9-HpCDF	0.002	0.082	0.008	0.014 $\pm$ 0.018
OCDF	0.000	0.024	0.003	0.005 $\pm$ 0.005
PCDDs	0.103	1.157	0.291	0.330 $\pm$ 0.195
PCDFs	0.092	2.27	0.386	0.667 $\pm$ 0.555
<b>I-TEQ</b>	<b>0.195</b>	<b>2.87</b>	<b>0.755</b>	<b>0.997 <math>\pm</math> 0.667</b>
<b>Total PCDD/Fs (pg/g dry weight)</b>				
TCDDs	0.925	12.4	2.32	3.04 $\pm$ 2.09
PeCDDs	1.16	14.3	3.46	3.92 $\pm$ 2.33
HxCDDs	2.64	15.0	4.90	5.36 $\pm$ 2.39
HpCDDs	3.08	19.3	7.68	7.87 $\pm$ 4.14
OCDD	6.85	29.3	19.4	18.30 $\pm$ 5.16
TCDFs	1.10	19.6	3.81	4.58 $\pm$ 3.37
PeCDFs	1.24	20.2	4.21	4.87 $\pm$ 3.50
HxCDFs	0.785	17.2	3.53	3.99 $\pm$ 3.10
HpCDFs	0.530	11.6	2.78	3.09 $\pm$ 2.02
OCDF	0.331	23.6	3.18	4.55 $\pm$ 5.09
Total PCDDs	15.1	83.3	37.3	38.5 $\pm$ 13.3
Total PCDFs	4.31	92.2	17.1	21.1 $\pm$ 15.5
<b><math>\Sigma</math>PCDD/F</b>	<b>19.4</b>	<b>175</b>	<b>54.9</b>	<b>59.6 <math>\pm</math> 27.8</b>

Korea (1.3-5.5 pg TEQ/g dry weight; Moon et al., 2002b). Concentrations of PCDD/F in marine sediments from some Industrialized bays (Ok et al., 1999) and Masan Bay (Im et al., 2002) in Korea ranged from 2 to 32 pg TEQ/g dry weight and from 1 to 76 TEQ/g dry weight, respectively. Concentrations of PCDD/F in some Korean river sediments ranged from 0.07 to 23.9 pg TEQ/g dry weight (Ryoo et al., 2005). Consequently, the PCDD/F concentrations measured in our study were relatively lower than those reported at several Korean coastal sites.

#### Homologue and congener profiles of PCDD/Fs

The profiles of homologue and toxic congeners of PCDD/Fs in sediments from Mokpo coastal waters are shown in Fig. 4. Octachlorinated dibenzo-p-

dioxin (OCDD) was the predominant homologue group, while profiles of PCDF homologues were generally similar to each other (Fig. 4a). The OCDD homologue group was dominant in the marine sediments that acted as sinks for PCDD/Fs (Zook and Rappe, 1994). This was generally due to the lower solubility of OCDD in water (Muir et al., 1992) and the lower photodegradation potential in the environment (Baker and Hites, 2000). Of the toxic 2, 3, 7, 8-substituted congeners of PCDD/Fs in all locations surveyed, the contribution of total PCDFs to the total TEQ concentrations was higher than that of PCDDs (Fig. 4b). The congeners 1,2,3,7,8-PeCDD, 1,2,3,4,7,8-HxCDF, 2,3,4,7,8-PeCDF were the major contributors to TEQ concentrations, accounting for about 50% of the total TEQs. This pattern is similar to those

previously reported (Sakurai et al., 1996; Im et al., 2002).

### MDS ordination and pollution sources of PCDD/Fs

The PCDD/F contamination pattern in sediments contaminated exclusively by atmospheric deposition was dominated by OCDD, while sediments contaminated directly by industrial discharges contained complex patterns of PCDD/F congeners (Zook and Rappe, 1994). Baker and Hites (2000) suggested that the photochemical synthesis of OCDD from pentachlorophenol (PCP) in aerosols was the most significant source of OCDD in the environment. Therefore, we performed two-dimensional non-parametric multidimensional scaling (MDS) ordination using PRIMER for Windows (PRIMER Version 5.2.9, Plymouth, UK), to further characterize the spatial variability and potential sources of PCDD/F contamination in marine sediments.

The 10 homologue groups of PCDD/Fs in collected sediment samples from tetra- to octa-CDD/Fs were subjected to MDS ordination. This multivariate statistical technique has been used to determine the spatial variability of chemical composition in sediments (Fletcher et al., 2001; Moon et al., 2007). The results of non-parametric MDS ordination of the PCDD/F concentrations at individual sampling locations, using the Bray Curtis similarities calculated from squareroot transformed data, are shown in Fig. 5. The plot had a stress of 0.06, with values less than 0.1 representing a good ordination with little chance for misinterpretation (Clarke and Warwick, 1994). Four clusters were identified on the variable plot, each containing data grouped according to their homologue patterns of PCDD/Fs.

The first cluster (Group A) included most of the sampling locations in Mokpo coastal waters compared to other clusters. Although each of the sampling locations had different potential sources of PCDD/Fs, their homologue distributions were relatively similar, with small standard deviations. At all sampling locations, homologue profiles of PCDD/Fs were characterized by increasing contribution of the PCDD homologues with similar contributions of each homologue group for PCDFs (Fig. 6). These patterns are similar to those in atmospheric deposition samples in Korea (Moon et al., 2005) and in sediments from Tokyo Bay in Japan, which were contaminated by the deposition of combustion emissions (Yamashita et al., 2000; Ogura et al., 2001). Therefore, our result suggests that the major source of PCDD/Fs in Mokpo coastal waters was mainly

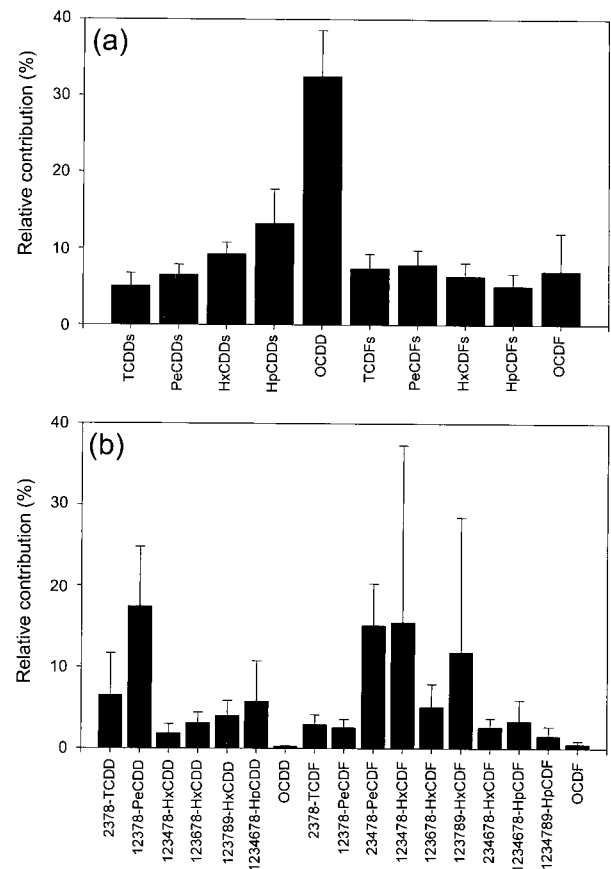


Fig. 4. Average normalized profiles of homologues (a) and toxic 2, 3, 7, 8-substituted congeners (b) of PCDD/Fs in marine sediments from Mokpo coast in Korea. Vertical lines represent standard deviations. Each homologue group and toxic 2, 3, 7, 8-substituted congeners were normalized to the total and TEQ concentrations of PCDD/Fs, respectively.

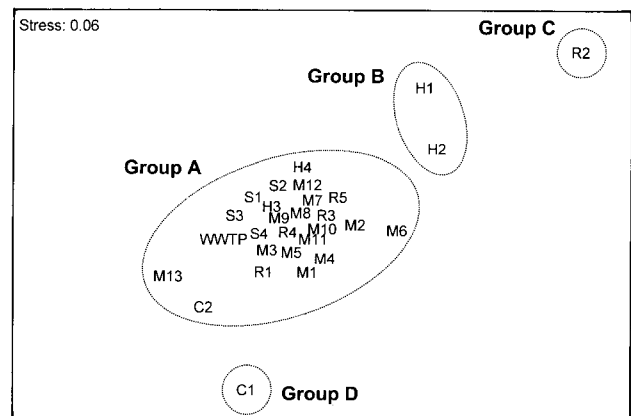


Fig. 5. Non-parametric multidimensional scaling (MDS) ordination plots of PCDD/F homologue distribution in marine sediments from Mokpo coastal water in Korea.

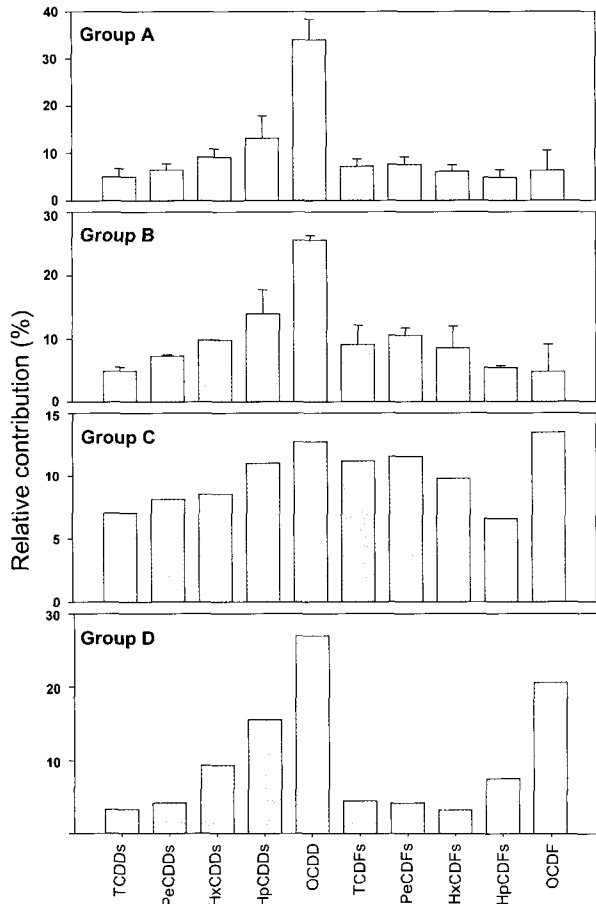


Fig. 6. Comparison of homologue profiles of PCDD/Fs for each group clustered by MDS ordination results. Results for each homologue profiles were normalized to the total concentration of PCDD/Fs in the sediments. Vertical lines represent standard deviations.

associated with combustion processes from industrial complexes.

The second group, Group B, was clustered by the harbor sediments such as Stations H1 and H2. The sediments from Group B were characterized by an increasing contribution of PCDFs in comparison with those of Group A. It is difficult to explain the difference in the homologue distributions for PCDD/Fs between Group A and B. However, Group B had relatively higher contributions for the less chlorinated dioxins such as tetra- and penta-CDD/Fs than those in Group A, suggesting existence of a specific source associated with combustion process by low temperature in this coastal zone such as shipyards.

The third cluster (Group C) represented Station R2 (Samhyang River), which is encompassed by a

number of households and industrial complexes in Mokpo. The homologue profile of PCDD/Fs in the sediment from Station R2 was characterized by a similar contribution from each homologue group, suggesting the influence of local and on-going sources, causing a lack of various degradation processes in the environment. The complex profile of PCDD/F homologues in the sediment from Station R2 may be attributed to domestic and industrial discharges (Zook and Rappe, 1994).

The sediment of Group D (Station C1) was dominated by more highly chlorinated homologues such as OCDD and OCDF. In particular, the high contribution of OCDF in the sediments is associated with the input of PCP to the marine environment (Ogura et al., 2001; Masunaga et al., 2003). Therefore, it can be suggested that the source of Station C1 is associated with the industrial combustion process and with additional sources such as PCP-related source.

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