



Gas/Particle Partitioning of Dioxins in Exhaust Gases from Automobiles

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ABSTRACT

This study investigates distributions of polychlorinated dibenzo-*p*-dioxin and polychlorinated dibenzofuran (PCDD/F) congeners in the exhaust gases of gasoline- and diesel-fueled vehicles. 6 sport utility vehicles (SUVs), 6 diesel passenger vehicles (DPVs), and 3 heavy duty diesel vehicle (HDDV) were examined using chassis dynamometer tests for measuring vehicular dioxin emissions. The mean PCDD/F I-TEQ emission factors were 0.101, 0.0688 and 0.912 ng I-TEQ/km for the SUVs, DPVs and HDDV, respectively. Highly chlorinated congeners dominated both gaseous and particulate phase PCDD/Fs. The major contributors of gas-phase PCDD/F I-TEQ for the SUVs, DPVs, and HDDV were 2,3,4,7,8-PeCDF, 2,3,7,8-TeCDD, and 2,3,4,7,8-PeCDF, respectively; however, 2,3,4,7,8-PeCDF was the major contributor in particulate-phase PCDD/F I-TEQ of these vehicles. The particulate-phase PCDD/Fs was responsible for 78.0, 90.3 and 71.1% of total PCDD/Fs for the SUVs, DPVs, and HDDV, respectively. Therefore, the control of particulate matter is more critical than that of gaseous pollutants for reducing PCDD/F emissions from automobiles.

Keywords: PCDD/Fs; Chassis dynamometer test; Sport utility vehicle; Diesel vehicle; Gas/Particle phase partitioning.

INTRODUCTION

In recent years, polychlorinated dibenzo-*p*-dioxins and polychlorinated dibenzofurans (PCDD/Fs) has been extensively concerned due to their acute toxicity and associated adverse health effects. PCDD/F congeners with chlorine substitution in 2,3,7,8 positions are most toxic to human and the PCDD/Fs can be formed during any incomplete combustion process (Bumb *et al.*, 1980). In the past, many studies focused on the PCDD/F emissions from various sources, such as municipal solid waste incinerators (MSWIs), metallurgical activities, crematories and temples (Wang *et al.*, 2003a-b; Lee *et al.*, 2004; Li *et al.*, 2007; Wang and Chang-Chien, 2007; Hu *et al.*, 2009a-c). However, mobile sources are almost as important as stationary sources in PCDD/F emission contribution in many countries.

The emission of PCDD/Fs from automotive has been investigated in several studies (Geueke *et al.*, 1999;

Joumard *et al.*, 2000; US EPA, 2001; Kim *et al.*, 2003; Weilenmann *et al.*, 2005; Dyke *et al.*, 2007). For example, Ryan and Gullett (2000; 2002) reported that the value was 0.023 ± 0.022 ng TEQ/km for a HDDV with a high mileage engine. Geueke *et al.* (1999) found that the PCDD/F emission concentrations ranged within 0.93–45 and 1.2–218 pg/Nm³, for unleaded gasoline cars and diesel trucks, respectively. Some researchers also estimated the PCDD/F emissions of vehicles in tunnels (Oehme *et al.*, 1991; Wevers *et al.*, 1992). Oehme *et al.* (1991) observed that the PCDD/F emission factors of gasoline and diesel vehicles were 0.028–0.52 and 0.72–9.5 ng I-TEQ/km, respectively. Ryan and Gullett (2000) indicated that tunnel studies relied on indirect emission measurements and generalized assumptions (e.g., average fuel consumption and/or mileage) that can introduce uncertainties in the derivation of emission estimates. In Taiwan, the relative contribution fraction of PCDD/Fs from mobile sources has gradually raised since the execution of more stringent PCDD/F emission standards for stationary sources. However, limited data are available to assess the magnitude of automobile PCDD/F emissions.

In the exhaust of vehicles, PCDD/Fs may be gaseous or bound to particulate matter (PM). Precisely understanding

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the PCDD/Fs partitioning in vapor/solid phases is important for developing the PCDD/F control technologies of vehicles and modeling the transportation of PCDD/Fs in environment. Atmospheric fate of PCDD/Fs is primarily governed by their partitioning between particle and gas phases that influences the deposition, chemical reaction, and long range transport of PCDD/Fs. The vapor/solid partitioning of PCDD/Fs is also important for their movement from sources to aquatic environments and the food chain. Therefore, understanding PCDD/Fs partitioning between gas and particulate phases is necessary to conduct their dispersion modeling and risk assessments (Oh *et al.*, 2001; Rice *et al.*, 2008; Armitage *et al.*, 2009; Novak *et al.*, 2009). So far, few study reported the PCDD/F partitionings between vapor/solid phases in vehicle exhausts. To examine this important feature, this study was motivated to investigate the partitioning of PCDD/Fs between vapor/solid phases in the exhausts of gasoline- and diesel-fueled vehicles. In this study, 6 SUVs, 6 DPVs, and 3 HDDV were tested in accordance with regulated driving cycles to estimate emission concentrations, emission factors, and gas/particulate phase distributions of PCDD/Fs in the vehicle tailpipe exhausts. This study provides essential information for developing control strategies, dispersion modeling, and risk assessments of vehicular PCDD/F emissions.

MATERIALS AND METHODS

Vehicles

This study took into account the sale volumes, mileages, and engine years of tested SUVs, DPVs, and HDDV before conducting experiments. Therefore, we tested six 2002–2005 top five sale SUVs, six 2005–2006 top three DPVs, and three 1994–1999 best sale HDDV, compliant to the Phase II HDDV emission regulations. The mileages of tested SUVs and DPVs ranged from 20000 to 62500 km and 12000 to 70000 km, respectively. The basic information concerning the tested vehicles is listed in Table 1.

Test Procedures

The dynamometer (SCHENCK 500/GS112) used in this

study was provided from the Environmental Protection & Energy Test Lab of Automotive Research & Test Center, which is fully accredited by Taiwan EPA for executing emission tests of gasoline/diesel and alternative fuel vehicles, and middle/heavy duty diesel engines. For the simulation of real on-road operation statuses, SUVs were tested according to the driving pattern FTP-75 (a US federal test procedure), comprising cold start (0–505 sec), transient (505–1369 sec), and hot start (1369–1874 sec) phases with a whole cycle mileage of 17.77 km. The maximum, minimum, and mean velocities of the three phases were 91.2, 0, and 34.1 km/h, respectively. We also adopted the new European driving cycle (NEDC) consisting of four repeated ECE-15 driving cycles (Economic Commission for Europe) and an Extra-Urban driving cycle (EUDC) in tests. The NEDC represents the typical usage of a car in Europe, and it is also used to assess the air pollutant emission levels of light duty diesel vehicles in Taiwan. The HDDV was tested for PCDD/Fs on the exhaust emission certification procedures of the US Federal Test Procedure (FTP-75) (US Code of Federal Regulations, 1993).

The FTP cycle sequence includes tests of expressway, congested urban, and uncongested urban driving patterns. For the vehicle exhaust gas emission measurements, the FTP-75, NEDC, and FTP transient cycle test procedures are also the current Taiwan standard emission driving patterns used for SUVs, DPVs, and HDDV, respectively. Thus, they were employed in this study.

Sampling

All tested engines were fueled with the same batch commercial diesel fuel or unleaded gasoline in order to disable the interference resulted from fuel variations. The sampling procedures followed the USEPA modified Method 23. The sampling train adopted in this study is comparable with that specified by the USEPA modified Method 5. Gas-phase samples were collected by XAD-2 resins while particulate-phase ones were simultaneously sampled using fiberglass filters. All the exhaust gas samples were collected isokinetically under the aforesaid test cycles and implemented for a complete test period.

Table 1. Basic information concerning the tested vehicles.

Vehicle model	Fuel	Volume (cm ³)	Mileage (km)	Model year	Driving Cycle
SUV					
A (n = 2)	unleaded gasoline	1998	33009–62506	2003, 2005	FTP-75
B (n = 2)		1998	35400–62415	2002, 2003	
C (n = 2)		2261	20205–46325	2004, 2005	
DPV					
D (n = 2)	diesel	1896	8020–24547	2005, 2006	NEDC
E (n = 2)		1991	12487–32104	2005	
F (n = 2)		2497	25574–70275	2005	
HDDV					
G (n = 2)	diesel	2835	7533–12478	1994, 1999	US-Transient
H (n = 1)		2835	22136	1999	

Analyses of PCDD/Fs

All vehicle exhaust flue gas samplings and chemical analyses in this study were carried out by an accredited laboratory (Super Micro Mass Research and Technology Center, Cheng Shiu University) for PCDD/F-associated samplings and analyses in Taiwan. The protocols of quality assurance/control (QA/QC) for PCDD/F analyses are strictly adopted. Immediately before an analysis, a standard solution was added to a sample to ensure the recovery of the analysis process. Prior to sampling, XAD-2 resin grains were spiked with PCDD/F surrogate standards prelabeled with isotopes, including $^{37}\text{Cl}_4$ -2,3,7,8-TCDD, $^{13}\text{C}_{12}$ -1,2,3,4,7,8-HxCDD, $^{13}\text{C}_{12}$ -2,3,4,7,8-PeCDF, $^{13}\text{C}_{12}$ -1,2,3,4,7,8-HxCDF, and $^{13}\text{C}_{12}$ -1,2,3,4,7,8,9-HpCDF. The recoveries of the spiked PCDD/F surrogate standards were 94–111%, 105–111%, and 98–115% for SUVs, DPVs, and HDDV, respectively. The MDLs ranged from 0.000542–0.0636, 0.000545–0.0242, and 0.000635–0.0691 ng/Nm³ for SUVs, DPVs, and HDDV, respectively. All the samples met the recovery criteria within 70–130%, as well as a relative standard deviation (RSD) less than 15%, revealing no PCDD/F breakthrough in sampling. The details were similar to that given in our previous work (Wang *et al.*, 2003).

High-resolution gas chromatograph/high-resolution mass spectrometer (HRGC/HRMS) was used for PCDD/Fs analyses. The HRGC (Hewlett-Packard 6970 Series gas chromatograph, CA) was equipped with a DB-5 fused silica capillary column (L = 60 m, i.d. = 0.25 mm, film thickness = 0.25 μm) (J&W Scientific, CA) with a splitless injection. The oven temperature program was set according to the following: beginning at 150°C (held for 1 min), followed at 30 °C/min to 220°C (held for 5 min), then at 1.5 °C/min to 240°C (held for 5 min), and finally at 1.5 °C/min to 310°C (held for 20 min). The HRMS (Micromass Autospec Ultima, Manchester, UK) mass spectrometer was equipped with a positive electron impact (EI+) source. The analysis mode of the selected ion monitoring (SIM) was used with resolving power at 10000. The electron energy and source temperature were specified at 35 eV and 250°C, respectively.

RESULTS AND DISCUSSION

Average PCDD/F Concentrations and Emission Factors in Exhaust Gases

The mean PCDD/F I-TEQ concentrations in exhaust gases of the SUVs, DPVs, and HDDV are listed in Table 2, which were 0.0544 (RSD = 70.3%), 0.0337 (88.6%), and 0.0724 ng I-TEQ/Nm³ (56.7%), respectively. The total PCDD/F I-TEQ concentrations of SUVs and HDDV were 1.6 and 2.1 times higher than that of DPVs. The PCDD/PCDF I-TEQ ratios were all less than 1, indicating that the toxicity from PCDFs dominated the total PCDD/F toxicity in the exhaust gases of vehicles.

The PCDD/F I-TEQ concentrations obtained in this study were higher than those of HDDVs (1.1–9.7 pg I-TEQ/Nm³) reported by Geueke *et al.* (1999) and a light duty diesel engine (6.4–14.5 pg I-TEQ/Nm³) (Kim *et al.*, 2003). Hagenmaier *et al.* (1990) observed that the PCDD/F I-TEQ concentration was 141.5 pg I-TEQ/Nm³ for passenger cars fueled with leaded gasoline.

The fuel consumption had not been measured in this study, so the PCDD/F emission factors of the vehicles obtained in this study were only reported on the basis of mileage. For the tested SUVs, DPVs, and HDDV, the calculated mean PCDD/F emission factors were 2.92, 0.884 and 13.6 ng/km, respectively, with corresponding mean I-TEQ emission factors of 0.101, 0.0688 and 0.912 ng I-TEQ/km, respectively. Thus, the HDDV emitted more PCDD/Fs than the other tested vehicles. The PCDD/F I-TEQ emission factors of SUVs and DPVs in this study were greater than those of unleaded gasoline vehicles and a diesel car (0.0007–0.005 and 0.0024 ng I-TEQ/km, respectively) reported by Hagenmaier *et al.* (1990) and that of a diesel truck in tunnel sampling (0.172 ng I-TEQ/km) performed by Gertler *et al.* (1996). The PCDD/F I-TEQ emission factor of HDDV in this study is comparable to the data (0.663–1.3 ng I-TEQ/km) of HDDVs in dynamometer tests obtained by Lew *et al.* (1993). The parameters such as the models, mileages, and engine years had been evaluated their relation with PCDD/Fs by conducting principal component analysis and spearman

Table 2. Mean PCDD/F concentrations and emission factors of vehicles.

	SUVs		DPVs		HDDV	
	mean (n = 6)	RSD%	mean (n = 6)	RSD %	mean (n = 3)	RSD %
Concentrations						
PCDDs ng/Nm ³	0.612	82.6	0.103	56.2	0.451	49.4
PCDFs ng/Nm ³	0.633	74.2	0.221	54.1	0.654	46.1
PCDDs/PCDFs ratio	0.96	15.3	0.52	64.0	0.61	28.2
Total PCDD/Fs (ng/Nm ³)	1.25	86.4	0.324	38.5	1.11	44.5
PCDDs/PCDFs (I-TEQ) ratio	0.23	45.9	0.11	37.4	0.432	29.4
Total I-TEQ (ng I-TEQ/Nm ³)	0.0544	70.3	0.0337	88.6	0.0724	56.7
Emission factors						
PCDDs ng/km	1.78	82.1	0.243	47.3	5.73	46.1
PCDFs ng/km	1.14	80.4	0.521	29.5	7.84	39.3
Total PCDD/Fs ng/km	2.92	78.5	0.884	18.9	13.6	34.2
Total I-TEQ I-TEQ/km	0.101	63.7	0.0688	68.7	0.912	47.7

correlation. However no relation had been found between them, maybe because there were some more influential factors, such as maintenance condition, replacement period of lubricant, and the types of catalyst convertors, that had not been considered in this study.

Congener Profiles of Gas/Particulate Phase PCDD/Fs in Vehicular Exhaust Gases

Fig. 1 shows the congener profiles of gas/particulate phase 17 PCDD/Fs used to characterize the fingerprints of PCDD/F emissions from the tested vehicles. The fraction (%)

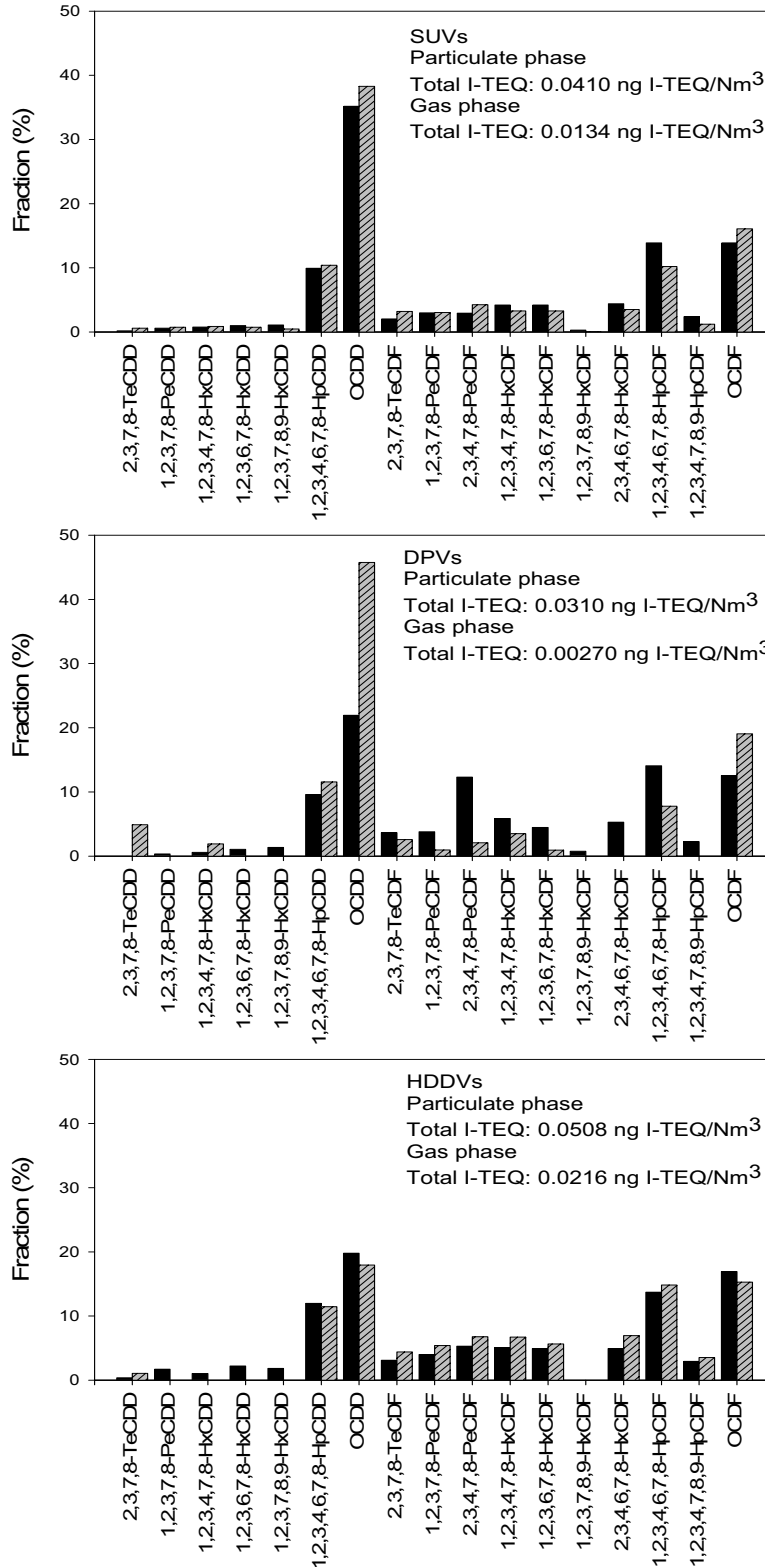


Fig. 1. Congener profiles of gas/particulate phase PCDD/Fs in the exhaust gases of vehicle.

of a gaseous (or particulate) phase PCDD/F congener represents its concentration normalized by the total concentration of gaseous (or particulate) phase PCDD/Fs. The gaseous and particulate phase PCDD/Fs exhibited similar congener profiles, and the highly chlorinated congeners dominated both gaseous phase and particulate phase PCDD/Fs. The dominant three PCDD/F congeners of SUVs were OCDD (gaseous and particulate phase fractions (G and P PFs) = 38.5 and 35.0%, respectively), OCDF (G and P PFs = 16.7 and 14.1%, respectively), and 1,2,3,4,6,7,8-HpCDF (G and P PFs = 9.8 and 14.7%, respectively). A similar trend of PCDD/F congener profiles was observed for the DPVs that had G and P PFs of 42.4 and 20.4% for the OCDD, 17.2 and 15.4% for the OCDF, and 7.1 and 16.3% for the 1,2,3,4,6,7,8-HpCDF. For the HDDV, the dominant four congeners were OCDD (G and P PFs = 18.0 and 21.9%), OCDF (G and P PFs = 15.0 and 14.3%), 1,2,3,4,6,7,8-HpCDF (G and P PFs = 15.3 and 14.2%), and 1,2,3,4,6,7,8-HpCDD (G and P PFs = 12.6 and 10.7%). The results reveal that the differences in fuel and engine types did not result in obvious variation of the PCDD/F congener profiles. The PCDD/F congener profiles of vehicles obtained in this study were similar to those reported by previously study (Gertler *et al.*, 1996; Ryan and Gullett, 2000). These vehicular PCDD/F congener profiles are different from those of metallurgical processes (Wang *et al.*, 2010), which 2,3,7,8-TeCDF, 1,2,3,7,8-PeCDF, and 2,3,4,7,8-PeCDF are the more prominent congeners in the stack flue gases.

Partitioning of Gas/Particulate Phase PCDD/Fs

Table 3 shows the PCDD/F gas/particulate phase distributions in the exhaust gases of tested vehicles. The percentage of a gaseous (or particulate) phase PCDD/F congener is equal to its concentration $\times 100\%$ divided by the total (gas + particulate) PCDD/F concentration. The particulate-phase PCDD/Fs accounted for 78.0, 90.3 and 71.1% of total PCDD/Fs for the SUVs, DPVs and HDDV, respectively. These vehicles emitted more particulate-phase PCDFs than particulate-phase PCDDs. For the SUVs, the top PCDD/F contributor was particulate-phase OCDD (27.3%), and the highly chlorinated (hexa- to octa-) PCDD/Fs was responsible for more than 90.1% of total PCDD/Fs. Similarly, the particulate-phase OCDD was the major contributor of PCDD/Fs for the DPVs and HDDV, and the highly chlorinated PCDD/Fs accounting for 83.6 and 84.2% of total PCDD/Fs, respectively. Note that the PCDD/Fs were almost dominated by particulate-phase PCDD/Fs except for 2,3,7,8-TeCDF.

For the SUVs, 2,3,4,7,8-PeCDF was the major contributor of PCDD/F I-TEQ concentrations in both gas- (8.6%) and particulate-phase (28.8%) PCDD/Fs, while 2,3,4,6,7,8-HxCDF, 1,2,3,4,7,8-HxCDF, and 1,2,3,6,7,8-HxCDF also had noticeable contributions in particulate PCDD/F I-TEQ, (6.9, 7.3, and 7.3%, respectively) (Fig. 2). Dissimilarly, for the DPVs, the major contributors to gas- and particulate-phase PCDD/F I-TEQ were 2,3,7,8-TeCDF (9.8%) and 2,3,4,7,8-PeCDF (56.3%), respectively. Similar results were observed for the HDDV, with 2,3,4,7,8-PeCDF

Table 3. Percentages of gas/particulate (G/P) phase PCDD/F concentrations in the exhaust gases of vehicles.

PCDD/Fs	SUVs (n = 6)		DPVs (n = 6)		HDDV (n = 3)	
	G (%)	P (%)	G (%)	P (%)	G (%)	P (%)
2,3,7,8-TeCDF	0.13	0.15	0.28	NA	0.29	0.27
1,2,3,7,8-PeCDF	0.16	0.47	0.09	0.26	0.22	1.26
1,2,3,4,7,8-HxCDF	0.18	0.56	0.12	0.47	0.13	0.76
1,2,3,6,7,8-HxCDF	0.18	0.71	0.10	0.85	0.11	1.80
1,2,3,7,8,9-HxCDF	0.12	0.85	NA	1.06	NA	1.35
1,2,3,4,6,7,8-HpCDF	2.22	7.24	0.94	8.47	3.63	7.58
OCDD	8.46	27.3	4.13	18.4	5.19	15.6
2,3,7,8-TeCDF	0.70	1.65	0.23	2.47	1.25	2.29
1,2,3,7,8-PeCDF	0.69	2.46	0.09	3.02	1.52	2.94
2,3,4,7,8-PeCDF	0.88	2.61	0.19	9.81	1.92	3.88
1,2,3,4,7,8-HxCDF	0.74	2.99	0.31	5.09	1.50	3.73
1,2,3,6,7,8-HxCDF	0.76	3.08	0.08	4.18	1.60	3.62
1,2,3,7,8,9-HxCDF	0.01	0.24	0.01	0.60	NA	NA
2,3,4,6,7,8-HxCDF	0.66	3.42	0.58	4.38	1.96	3.61
1,2,3,4,6,7,8-HpCDF	2.16	11.48	0.69	14.76	4.42	10.1
1,2,3,4,7,8,9-HpCDF	0.26	1.79	0.23	2.58	0.78	2.14
OCDF	3.67	11.0	1.68	13.88	4.34	10.2
Σ PCDDs	11.4	37.3	5.66	29.5	9.58	28.6
Σ PCDFs	10.5	40.7	4.08	60.8	19.3	42.5
Total PCDD/Fs	22.0	78.0	9.74	90.3	28.9	71.1
Total I-TEQ	24.6	75.4	8.0	92.0	29.9	70.1

NA: not available

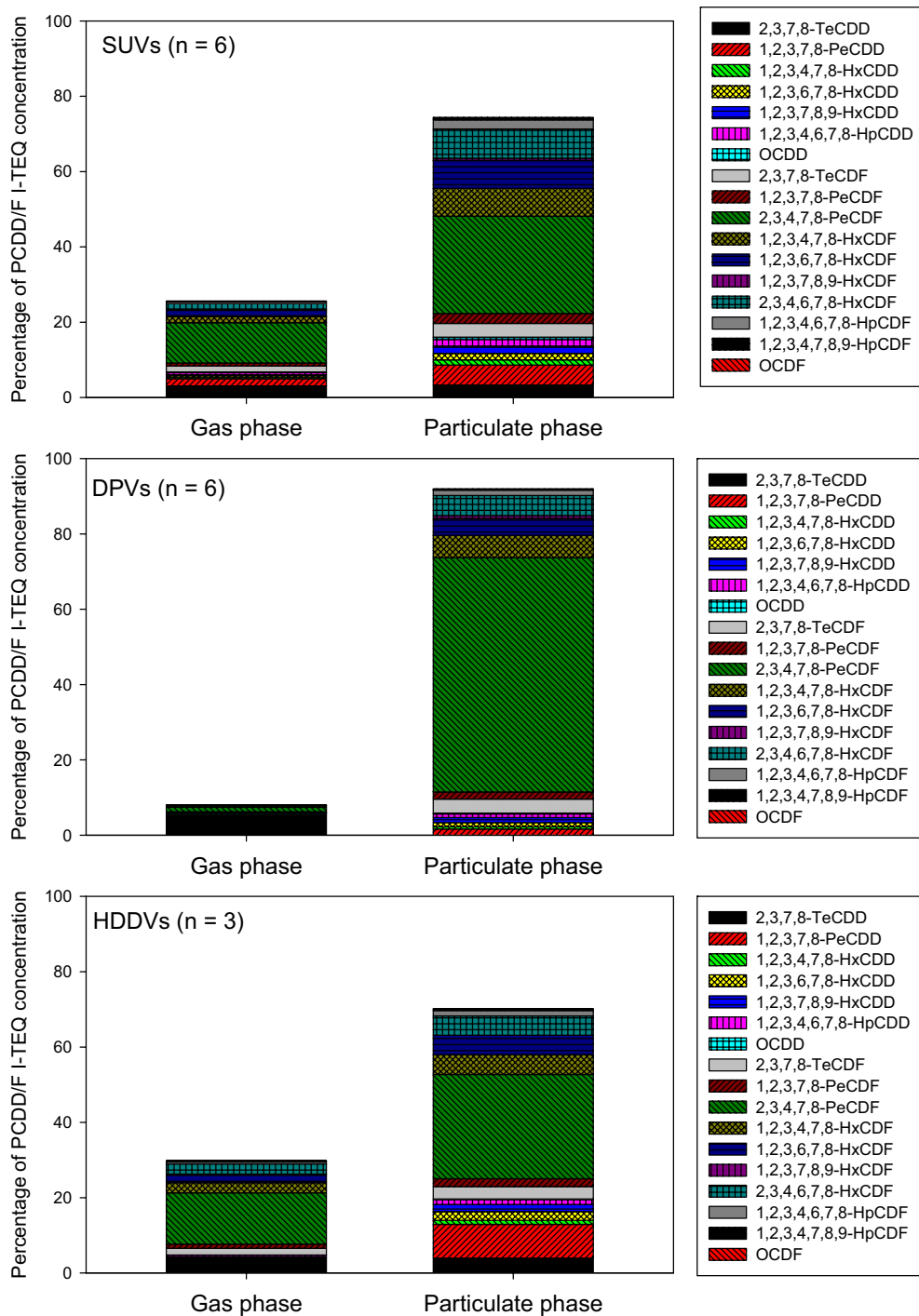


Fig. 2. Partitioning of gas/particulate phase PCDD/F I-TEQs in the exhaust gases of vehicles.

(14.3%) and 2,3,7,8-TeCDD (4.3%) as the major contributors in gas-phase I-TEQ, and 2,3,4,7,8-PeCDF (28.0%), 1,2,3,7,8-PeCDD (9.2%), 1,2,3,4,7,8-HxCDF (5.6%), 1,2,3,6,7,8-HxCDF (5.0%), and 2,3,4,6,7,8-HxCDF (5.3%) in the particulate phase. The findings demonstrate that the particulate-phase PCDD/Fs contribute more to the total I-TEQ emission than the gas-phase ones. Accordingly, it is possible to effectively lower PCDD/F emission from automobiles if more PM can be removed in vehicular exhaust gases.

CONCLUSIONS

In this study, 6 SUVs, 6 DPVs and 3 HDDV were tested (in accordance with standard driving cycles) for vehicular gas- and particle-phase PCDD/F emissions. The DPVs emitted lower PCDD/F concentrations than the other tested vehicles. Highly chlorinated congeners dominated gas- and particulate-phase PCDD/Fs in the exhausts of vehicles. Particulate-phase PCDD/Fs accounted for 78.0, 90.3 and 71.1% of total PCDD/Fs for the SUVs, DPVs and HDDV,

respectively; furthermore, the major contributor of PCDD/Fs was the particulate-phase OCDD. The particulate-phase PCDD/Fs also contributed more PCDD/F I-TEQ emissions than the gas-phase ones in the exhaust gases.

For the SUVs, DPVs, and HDDV, the gas-phase PCDD/F I-TEQ were mainly contributed by 2,3,4,7,8-PeCDF (8.6%), 2,3,7,8-TeCDD (9.8%), and 2,3,4,7,8-PeCDF (14.3%), respectively, while for the particulate-phase PCDD/F I-TEQ, 2,3,4,7,8-PeCDF was the major contributor for these three types of vehicles, and contributed 28.8%, 56.3% and 28.0%, respectively. Most congeners were dominated by particulate-phase PCDD/Fs except for 2,3,7,8-TeCDD. Therefore, particle reduction should be more favored than gas removal in exhausts to lower PCDD/F emission from automobiles.

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