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Evaluation of PCDD/F partitioning between vapor and solid phases in MWI flue gases with temperature variation

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Abstract

Partitioning of PCDD/Fs (polychlorinated dibenzo-*p*-dioxins and polychlorinated dibenzofuran) between vapor and solid phases in flue gas is affected by several factors including temperature variation. In this study, PCDD/F removal efficiencies achieved with activated carbon injection (ACI) and partitioning of vapor/solid phase PCDD/Fs in flue gases with temperature variation in a municipal waste incinerator (MWI) are evaluated via intensive flue gas sampling. Results indicate that most PCDD/Fs in flue gas downstream of the ACI + bag filter (BF) exist in vapor phase (over 90%) while the removal efficiencies of vapor and solid phase PCDD/Fs are 98.5–99.6% and 99.8–99.9%, respectively. The results of flue gas samplings also indicate that there is optimal operating temperature for PCDD/F removal achieved with ACI. Additionally, a pilot-scale adsorption system (PAS) is constructed in this study to evaluate the PCDD/F partitioning affected by temperature. The results of the PAS experimentation indicate that about 55% and 25% vapor phase PCDD/Fs passing through the filter cake (adsorbent) are transferred to solid phase at 150 and 200 °C, respectively. As the temperature is increased to 250 °C, filter cake (adsorbent) cannot effectively adsorb vapor phase PCDD/Fs and significant PCDD/Fs are formed via de novo synthesis.

Keywords: Dioxin; Municipal wastes incinerator; Stack sampling; Vapor pressure; Activated carbon

1. Introduction

In recent years, much concern has been focused on contamination by polychlorinated dibenzo-*p*-dioxins and polychlorinated dibenzo furans (PCDD/F) in the environment from the risk perspective due to their high toxicity. Seventeen PCDD/F congeners with chlorine substitution in 2,3,7,8 positions are most toxic to human being. Waste incineration processes including municipal waste incinerators (MWIs), medical waste incinerators and industrial waste incinerators have been identified as one of the major anthropogenic PCDD/F sources [1]. Atmospheric transport is the key factor in moving PCDD/Fs from sources to remote regions and in contaminating terrestrial and aquatic environments and the food chain. The atmospheric fate of PCDD/Fs is primarily governed by their vapor/solid partitioning. Khachatryan et al. [2] indicate that the most direct route to PCDD/F formation in the flue gases of emission sources is the vapor phase reaction. In flue gases, PCDD/Fs may exist in gaseous form (vapor phase) or be bound to particulate matter (solid phase). To meet the stringent PCDD/F emission standards, PCDD/F emission sources are generally equipped with various types of air pollutant control devices (APCDs), leading to different levels of PCDD/F control. Previous study [3] demonstrated that partitioning of PCDD/Fs between vapor and solid phases changes significantly as the flue gas passes through different APCDs. Without the real-time analyzer for instantaneous measurement of PCDD/F concentration of flue gases, PCDD/F partitioning between vapor and solid phases based on stack sampling data is highly uncertain. Limited studies have been completed so far to compare the phase distributions of PCDD/Fs and other influencing factors based on flue gas samplings.

The key parameters controlling the phase variation include the congener's vapor pressure, particle concentration and

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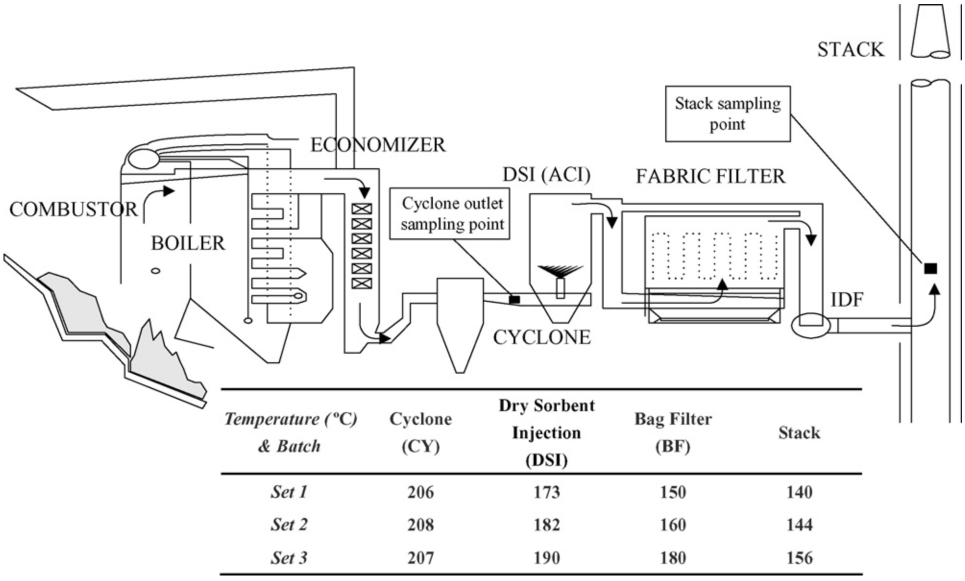
removal mechanism of the APCDs applied. The vapor pressures of seventeen 2,3,7,8-substituted PCDD/F congeners at 25 °C vary from 5×10^{-10} to 2×10^{-7} Pa [4] and decrease as chlorination level increases. The vapor pressure of the organic compound increases as the temperature increases [5]. Raccanelli et al. [6] indicated that the vapor phase PCDD/Fs at the furnace exit was actually lower than that measured at the inlet of APCDs even though the flue gas temperature at the furnace exit was higher than that at the inlet of APCDs. Hence, the temperature variation is not the only factor affecting the PCDD/F partitioning between vapor and solid phase in flue gases. That study also excludes the possibility that most vapor phase PCDD/Fs was generated by de novo synthesis. Vapor phase PCDD/Fs can be emitted from the stack by penetrating the APCDs, which are mainly designed for controlling particulate matter such as cyclone (CY), bag filter (BF) or electrostatic precipitator (EP). Vapor phase PCDD/Fs can be removed by various effective means including adsorption with carbon-based adsorbents, and catalytic destruction. Previous study [7] also indicates that activated carbon injection (ACI) technology can effectively remove vapor phase PCDD/Fs while BF is effective in removing solid phase PCDD/Fs. However, the removal efficiencies of vapor/solid phase PCDD/F congeners achieved with ACI are not always consistent due to the difference of congeners in terms of vapor pressure, and different adsorbing capacities of the activated carbons used. Hence, understanding PCDD/Fs partitioning in vapor/solid phases is important in selecting, designing and operating PCDD/F control equipment. To examine this important feature, this study was motivated to investigate the partitioning of PCDD/Fs between vapor/solid phases of flue gas. We focus on the understanding of the partitioning and removal efficiency of PCDD/Fs of MWI flue gas achieved with ACI at different flue gas temperatures. Besides, a pilot-scale adsorption system (PAS) was constructed for evaluating the effects of gas temperature on PCDD/F partitioning between vapor and solid phases.

2. Materials and methods

2.1. MWI sampling

To evaluate the performance of the APCDs at different operating temperatures for reducing vapor/solid phase PCDD/F emissions and to understand the effect of the temperature variation of APCDs on the distribution of vapor/solid phase PCDD/Fs, flue gas samples were collected simultaneously before and after APCDs in the municipal wastes incinerator (MWI) investigated. The MWI investigated was located in northern Taiwan and started to operate in 1995. It consists of three incinerating units, each with its own heat recovery system. The capacity of each incinerator is 450 t/day. This MWI was originally equipped with cyclone (CY), dry lime sorbent injection systems (DSI) and bag filters (BF) for controlling acid gas and particulate emissions. As high as 4.5 ng-TEQ/Nm^3 of PCDD/F was measured in the stack gas of this MWI back in 1998 [8]. Hence, the ACI technology was retrofitted in the MWI investigated for reducing PCDD/F emissions with AC injection rate of 50 mg/Nm^3 to meet the stringent standards of 0.1 ng-TEQ/Nm^3. In this study, 12 vapor/solid phase PCDD/F samples at different operating temperatures were collected at CY outlet and the stack of the MWI investigated. The flue gas conditions and PCDD/F sampling points of MWI investigated in this study are schematically shown in Fig. 1. The flue gas sampling was conducted with the Graseby Anderson Stack Sampling System complying with the USEPA Method 23 in this study. To avoid the error and bias caused by sampling of dioxins bound to the particulate matter, isokinetic sampling had to be conducted in order to collect representative samples. Relevant study [9] indicates that the partitioning of vapor phase PCDD/Fs decreases with the increase of filter loading during the flue gas sampling. Hence, the sampling period in our system is not over 150 min and the fiber filters are replaced one to two times in each flue gas sampling to minimize the pressure drop effect and the adsorption of PCDD/Fs onto particles.



Temperature (°C) & Batch	Cyclone (CY)	Dry Sorbent Injection (DSI)	Bag Filter (BF)	Stack
Set 1	206	173	150	140
Set 2	208	182	160	144
Set 3	207	190	180	156

Fig. 1. Sampling points and temperature variation of APCDs in the MWI investigated.

Hence, the partitioning of solid phase PCDD/Fs and highly-chlorinated PCDD/Fs would not be overestimated and the bias caused by the high particle loading on the filter is minimized. During the sampling procedure, the vapor phase sample was collected by XAD-2 resin while the solid phase portion was collected by the fiber glass filter and by rinsing of the sampling probe thereafter.

2.2. PAS experimentation

A pilot-scale adsorption system (PAS) was constructed for evaluation of possible PCDD/F transferring between vapor and solid phases during samplings with temperature variation. The PAS consists of two Graseby Anderson Stack Sampling Systems (MST 2010) as shown in Fig. 2. The simulated PCDD/F-containing gas stream (vapor phase PCDD/Fs) for PAS testing was taken from the flue gas at CY outlet of the MWI investigated. The solid phase PCDD/Fs were removed by fiber glass filter installed prior to PAS. Around 0.121–0.128 g fly ash collected earlier by CY from the MWI investigated was placed on the filter (with a filter loading about 1.54–1.63 mg/cm^2 in the filter area of 78.5 cm^2) as filter cake (adsorbent) to investigate PCDD/F transferring between vapor and solid phases and formation potential during sampling. The fly ash was first sifted with a sieve (120 mesh) and then rotated continuously for 12 h to ensure its uniformity before use. The fly ash was then extracted twice with toluene and then rinsed with nitric acid to make sure it is dioxin-free before the experiment. To investigate the influence of flue gas temperature variation on the partitioning and possible formation of vapor/solid phase PCDD/Fs, three different operating temperatures (150, 200 and 250 °C) were selected and regulated by temperature controller of PAS. The sampling period of PAS experimentation is around 40–50 min. The conditions of PAS experimentation are listed in Table 1. For the PAS experimentation, the vapor phase sample was retained by XAD-2 resin while PCDD/F retained on filter cake (adsorbent) was presumed as solid phase sample.

Table 1
The experimental conditions of PAS experimentation

	Group		
	1	2	3
CO_2 (%)	10.5–10.9		
O_2 (%)	9.9–10.2		
$H_2O_{(g)}$ (%)	14.2–15.3		
HCl (ppm)	1900–2500		
Gas flow rate (lpm)	15		
Temperature of reactor (°C)	150	200	250
Filter loading (mg/cm^2)	1.63	1.54	1.59

2.3. Sample analysis

The samples collected were spiked with known amounts of USEPA Method 23 internal standard solution. Thereafter, the XAD-2 and filter samples were Soxhlet extracted with toluene for 24 h. The toluene extract was then concentrated to about 1 ml by rotary evaporation and was replaced by 5 ml hexane for pretreatment process. Having been treated with conc. sulfuric acid,

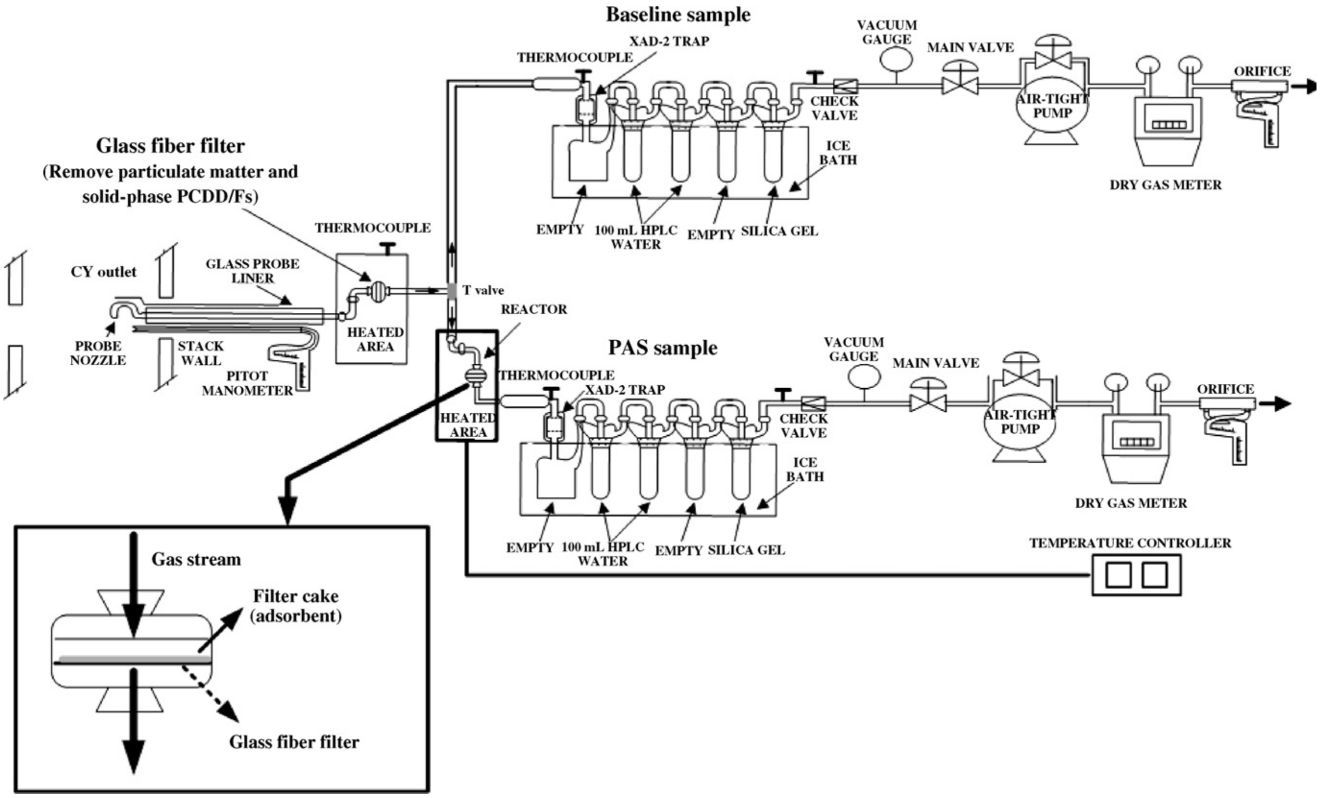


Fig. 2. Schematics of the pilot-scale adsorption system (PAS).

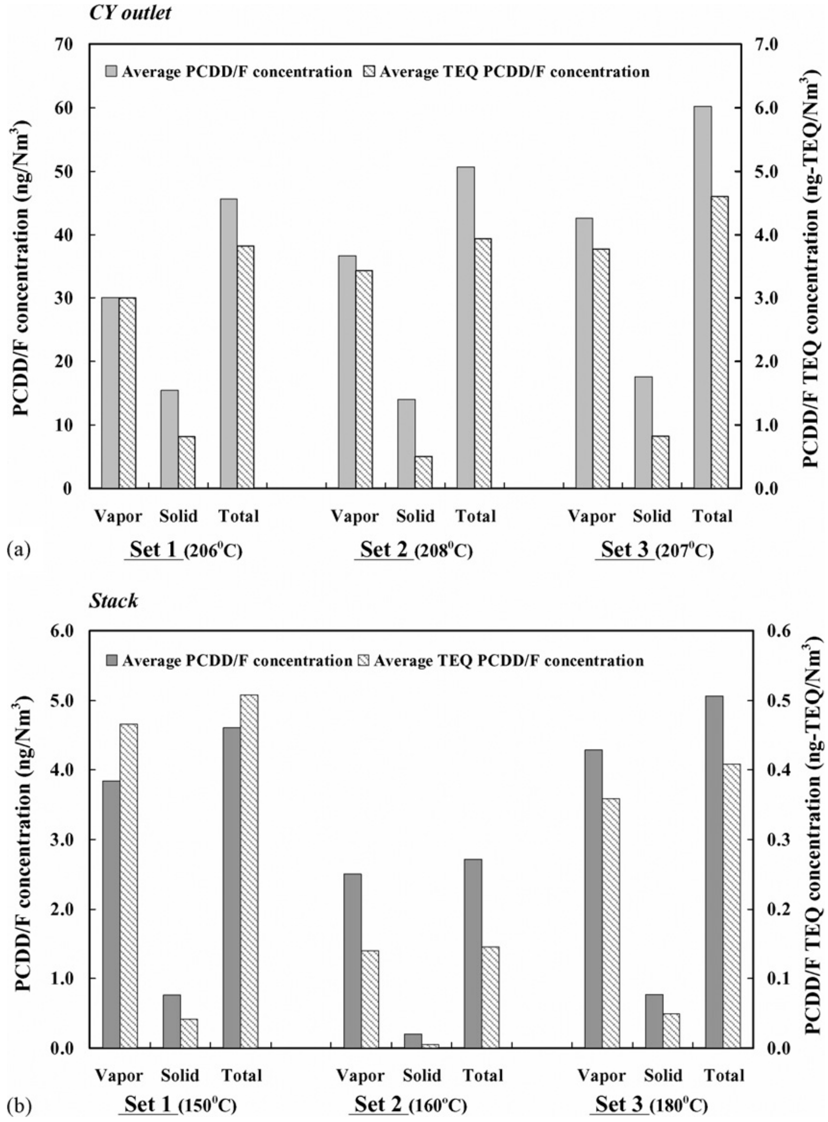


Fig. 3. Variation of PCDD/F concentrations in vapor/solid phases at (a) CY outlet and (b) stack of different temperatures in the MWI investigated.

the sample was then subjected to a series of clean-up columns including sulfuric acid silica gel column, acidic aluminum oxide column and celite/carbon column. Finally, the recovery standard solutions were spiked with known amounts of Method-23 internal standard solutions, and then analyzed for seventeen 2,3,7,8-substituted PCDD/F congeners with high resolution gas chromatography (HRGC) (Hewlett Packard 6890 plus)/high resolution mass spectrometer (HRMS) equipped with a fused silica capillary column DB-5 MS (60 m × 0.25 mm × 0.25 μm, Supelco).

3. Results

3.1. Average PCDD/F concentrations in flue gases of the MWI investigated with temperature variation

Fig. 3 shows the average PCDD/F concentrations in flue gases at two sampling points with temperature variation. Results of the flue gas samplings indicate that the PCDD/F concentrations measured are 3.82–4.61 ng-TEQ/Nm^3 at CY outlet and 0.146–0.507 ng-TEQ/Nm^3 at stack gas, respectively, with different operating temperatures. Additionally, the particulate matter (PM) concentrations measured at CY and stack ranges from 741 to 921 and 1.2 to 3.6 mg/Nm^3, respectively. PCDD/F concentration measured in stack gas was slightly higher than the PCDD/F emission limit (0.1 ng-TEQ/Nm^3) adopted for large-scale MWIs. Nevertheless, if we compare the results obtained in this study with the results obtained in 1999 [8], the ACI technology effectively reduces PCDD/F emission. Since the flue gas temperatures (sets 1–3) at CY outlet are quite close (206–208 °C); the PCDD/F concentrations measured at this sampling point do not vary significantly. Only a slight increase of PCDD/F concentration with increasing temperature is measured at CY outlet. However, the PCDD/F concentrations at stack gases are quite different as the flue gas temperature varies (138–156 °C).

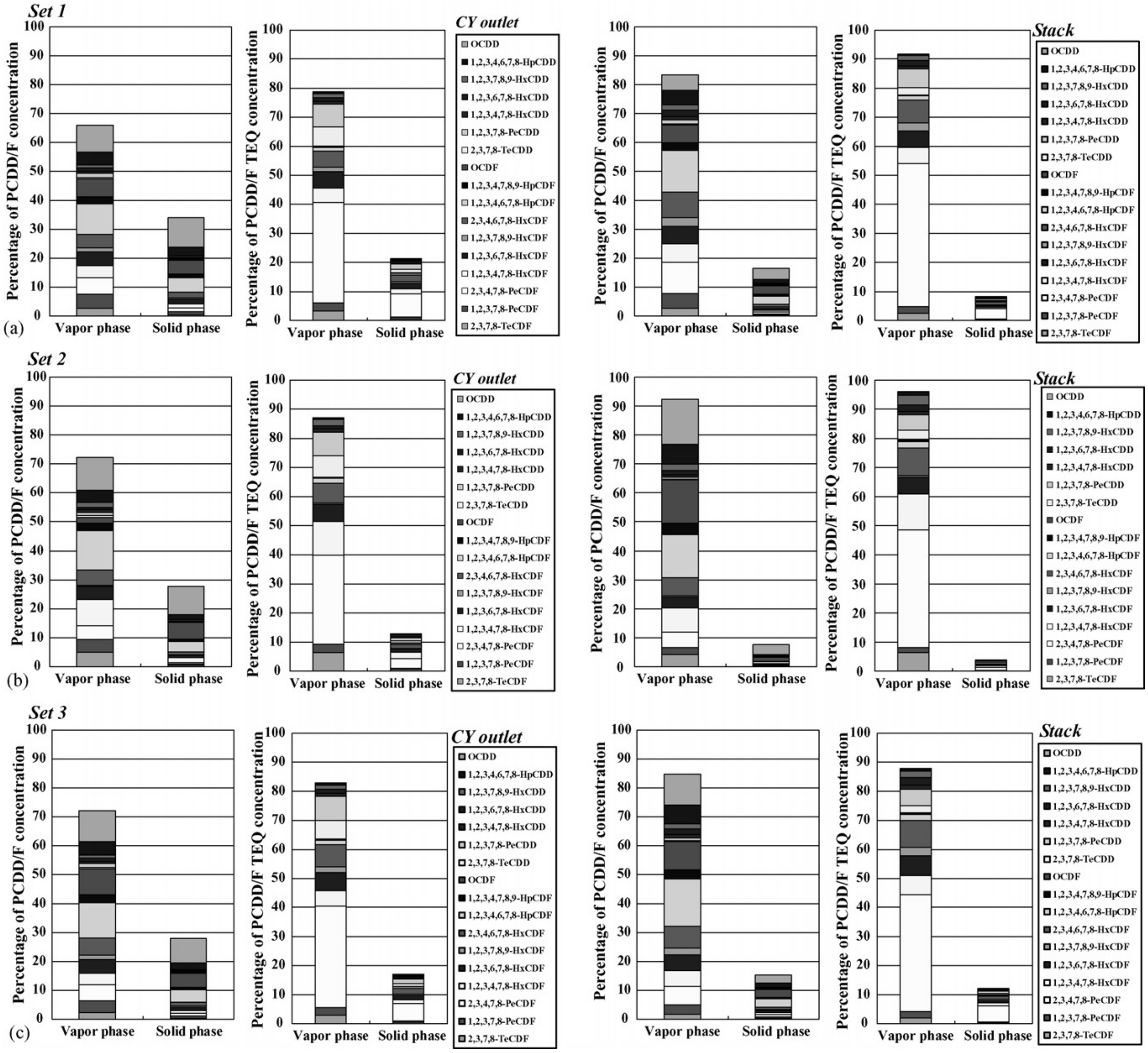


Fig. 4. Partitioning of PCDD/Fs in vapor/solid phases at cyclone outlet and stack gas of the MWI investigated with temperature variation.

3.2. Vapor/solid phase PCDD/F removal efficiencies with temperature variation

Fig. 4 shows the distribution of vapor/solid phase PCDD/Fs in the flue gases at different sampling points in the MWI investigated. PCDD/F congeners mostly distribute in vapor phase (about 75% of the total PCDD/Fs) at CY outlet. In stack gas, over 85% of PCDD/F congeners exist in vapor phase. In addition, Fig. 4 also indicates that 2,3,4,7,8-PeCDF is the major contributor of the TEQ concentrations in the vapor phase at all sampling points. Fig. 5 indicates the vapor/solid phase PCDD/F removal efficiencies achieved with ACI + BF with temperature variation. Overall, the solid phase PCDD/F removal efficiencies observed with temperature variation are higher than that in vapor phase. The vapor and solid phase PCDD/F removal efficiencies achieved with ACI + BF at set 2 (operating tempera-

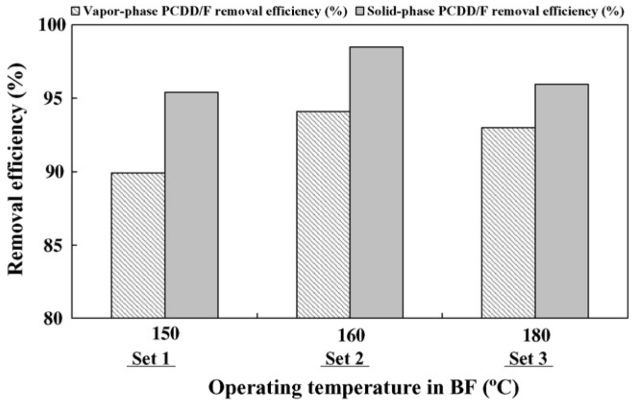


Fig. 5. Vapor/solid phase PCDD/F removal efficiencies achieved with ACI + BF with temperature variation in the MWI investigated.

ture: 160 °C) are the highest of three BF operating temperatures investigated. The PCDD/F concentration emitted from stack of set 2 is also the lowest among those three operating temperatures. The results obtained demonstrate a positive correlation between the solid phase PCDD/F and particle concentrations in flue gas. Based on the studies completed in other countries, certain correlation between particle and PCDD/F concentration has been established [10].

3.3. Partitioning of PCDD/Fs between vapor/solid phase with temperature variation

In order to evaluate the partitioning of seventeen 2,3,7,8-substituted PCDD/F congeners between vapor and solid phases, the vapor-solid distribution coefficient for each congener was calculated from the Eq. (1):

$$\varphi = \log\left(\frac{C_\mathrm{v}}{C_\mathrm{s}}\right) \quad (1)$$

φ is the coefficient of vapor/solid phase PCDD/F partition; C_v the concentration of PCDD/F congener exists in vapor phase (ng/Nm^3); C_s the concentration of PCDD/F congener adsorbed on particles (ng/Nm^3).

φ is used to represent the coefficient of vapor/solid phase PCDD/F congeners partition in logarithm. When the φ value is >0, it indicates that over 50% PCDD/F congeners are distributed in vapor phase. Previous study indicates [10] that vaporization is the major mechanism that causes solid phase PCDD/Fs to transfer into vapor phase (especially for the congeners of lowly chlorinated level, like TCDD). Hence, the coefficient of semi-volatile compounds (like PCDD/Fs) adsorbed to particles was mainly affected by the vapor pressures of those compounds. Furthermore, the vapor pressures of PCDD/F congeners increase as the gas temperature increases. Eitzer and Hites [11] correlated saturation vapor pressure (P_L^0) of PCDD/Fs with gas chromatographic retention indexes (GC-RI) on a non-polar (DB-5) GC column using p,p'-DDT as a reference standard. The correlation has been modified using RI (retention index) developed by Donnelly et al. [12] and Hale et al. [13]:

$$\log P_\mathrm{L}^0 = \frac{-1.34(\mathrm{RI})}{T} + 1.67\times10^{-3}(\mathrm{RI}) - \frac{1320}{T} + 8.087 \quad (2)$$

P_L^0 is the saturation vapor pressure of the organic compound (Pa); RI the retention index (from 2338 to 3196 for each seventeen 2,3,7,8-substituted PCDD/F congener); T the temperature (K).

To compare the partitioning of PCDD/Fs between vapor and solid phases in flue gases with temperature variation, data collected are further analyzed. Figs. 6 and 7 show the trend between the $\log(C_\mathrm{v}/C_\mathrm{s})$ versus $\log P_\mathrm{L}^0$ of each PCDD/F congener in flue gases downstream the CY and ACI + BF, respectively. Each dot represents the $\log(C_\mathrm{v}/C_\mathrm{s})$ versus $\log P_\mathrm{L}^0$ of each seventeen 2,3,7,8-substituted PCDD/F congener in the flue gas operating at different temperatures. In Fig. 6, the values of $\log(C_\mathrm{v}/C_\mathrm{s})$ are mostly >0 in flue gases downstream the particle control devices (CY). As the flue gases pass through the ACI + BF (Fig. 7), PCDD/Fs still mostly exist in vapor phase (over 85%), and the $\log(C_\mathrm{v}/C_\mathrm{s})$ values are all >0 and higher than that observed at the CY outlet.

3.4. Formation and transferring between vapor/solid phase PCDD/Fs by PAS experimentation

In PAS experimentation, the glass fiber filter installed prior to the PCDD/F adsorbent effectively removes particulate matter and the solid phase PCDD/Fs. Therefore, the results of PAS experimentation are obtained under the condition without the interference of particulate matter. The recovery of the vapor phase PCDD/Fs in PAS experimentation represents the percentage of vapor phase PCDD/Fs that pass through the filter cake (adsorbent) compared to that approaching to the adsorbent. Fig. 8 indicates the recovery of the vapor phase PCDD/Fs in PAS experimentation with the temperature variation. About 55% and 25% vapor phase PCDD/Fs were transferred to solid phase at Group 1 (150 °C) and Group 2 (200 °C), respectively, as flue gas passed through the adsorbent (filter cake). Relevant study [9] indicates that when the filter loading was over 1.5 mg/cm^2 during

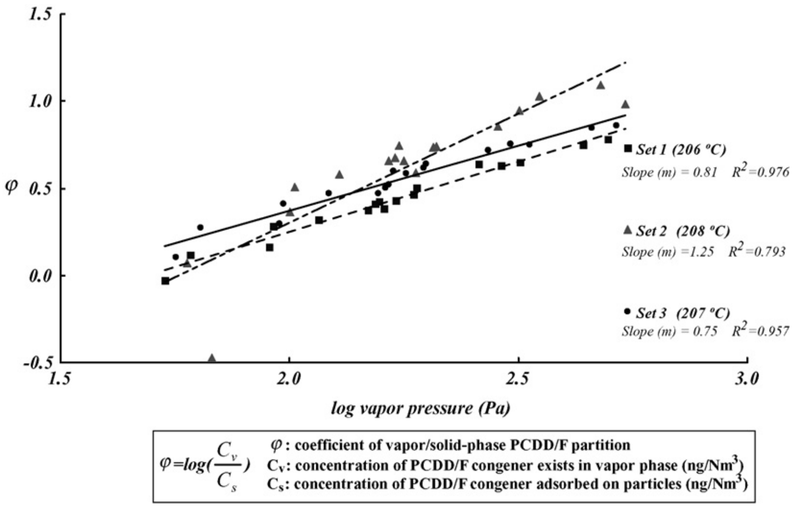


Fig. 6. The coefficient of vapor/solid phase PCDD/F partitioning at CY outlet.

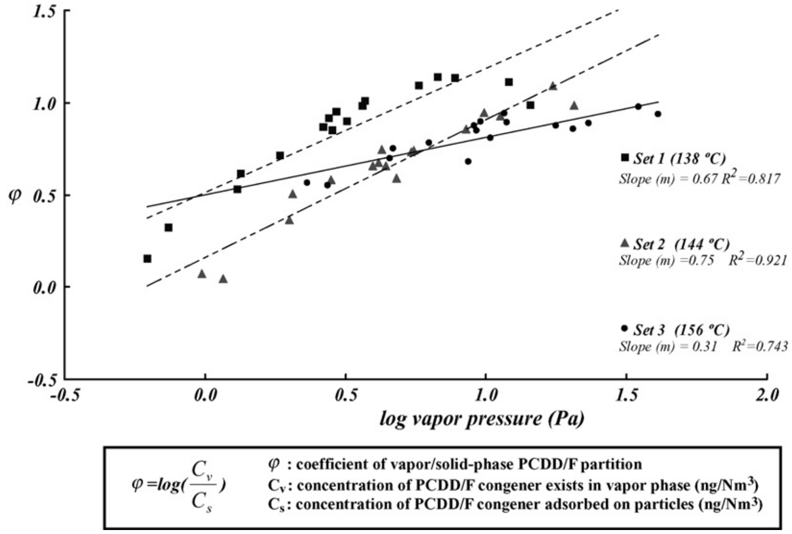


Fig. 7. The coefficient of vapor/solid phase PCDD/F partitioning in stack gas.

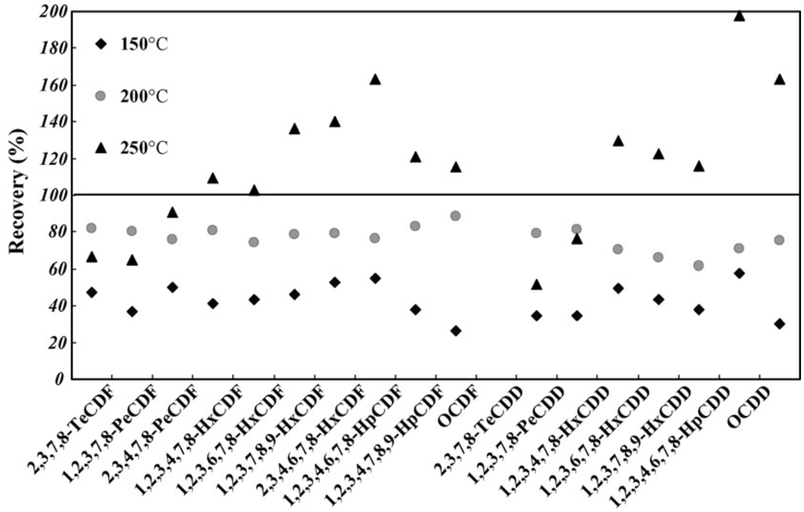


Fig. 8. Vapor phase PCDD/F recovery in PAS experimentation with temperature variation.

the flue gas sampling, the partitioning of vapor phase PCDD/Fs decreased by 60% at 120 °C. It might be attributed to the fact that the adsorbent applied acts like filter cake made by CY ash with the surface area of 60 m^2/g and has the tendency to adsorb vapor phase PCDD/Fs. In general, the adsorption capacity of adsorbent decreases with increasing temperature of the system in PAS experimentation. Fig. 9 shows that the vapor/solid phase PCDD/F concentrations increase (about four times) as the gas stream passed through the PCDD/F adsorbent at 250 °C.

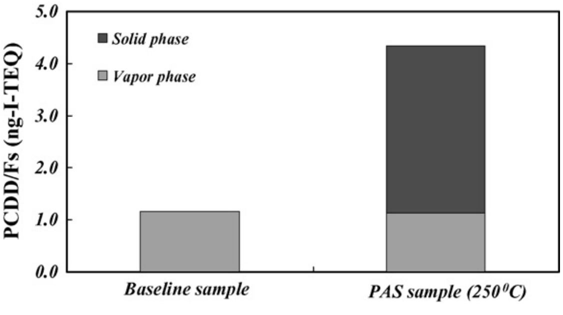


Fig. 9. Transferring and formation of vapor/solid phase PCDD/Fs in PAS experimentation at 250 °C.

4. Discussion

Based on the flue gas sampling results, the PCDD/F concentrations measured at stack gases are quite different as the flue gas temperature varies (138–156 °C). Fig. 3 also indicates that there is an optimal operating temperature (set 2) for PCDD/F removal with ACI. In addition, Evereart and Baeyens [14] indicate that de novo synthesis taking place between 200 and 400 °C results in the PCDF/PCDD ratio greater than 1. In the MWI investigated, the temperature of flue gas at CY outlet was over 200 °C which was within the de novo synthesis temperature window and the PCDF/PCDD ratio in flue gas is 1.99. Hence, relatively high PCDD/F concentrations measured at CY outlet in this study were more likely attributed to the de novo synthesis. Operating temperature of BF also affects the vapor/solid phase PCDD/F removal efficiencies. In set 1, the temperature in flue gas decreases from CY outlet to stack (CY: 206 °C → BF: 150 °C, set 1). As the level of temperature variation in flue gases is intense ($\Delta T_{\%}$: −27.2%), the vapor phase PCDD/Fs likely to condense on particle surface in flue gas. Therefore, the activated carbon injected in DSI could not effectively remove vapor phase PCDD/Fs (vapor phase PCDD/F removal efficiency is below 90%) in flue gas as the BF temperature is maintained at 150 °C. As the level of temperature variation ($\Delta T_{\%}$: −12.9%) gets smooth (CY: 207 °C → BF: 180 °C, set 3), the partitioning of PCDD/Fs in flue gas does not vary significantly. Therefore, the activated carbon injected could remove vapor phase PCDD/Fs in flue gas with a reasonable efficiency (vapor phase PCDD/F removal efficiency is over 90%). Compared with the values of $\log(C_\mathrm{v}/C_\mathrm{s})$ observed in Figs. 6 and 7, $\log(C_\mathrm{v}/C_\mathrm{s})$ generally increases with increasing vapor pressure of each PCDD/F congener. Because the solid phase PCDD/Fs are removed by CY to some extent and vapor phase PCDD/Fs are mostly formed via de novo synthesis, PCDD/F congeners measured at CY outlet are mostly distributed in vapor phase (over 70%). On the other hand, ACI + BF could effectively remove both vapor/solid phase PCDD/Fs in flue gases, and the slopes (m: 0.31–0.75) of $\log(C_\mathrm{v}/C_\mathrm{s})$ versus $\log P_\mathrm{L}^0$ shown in Fig. 7 are all smaller than the cases shown in Fig. 6 equipped with the particle control devices such as CY (m: 0.75–1.25). Previous study [7] indicated that the m value would be the largest at the flue gas with significant PCDD/F formation and without effective removal. Hence, the m value can represent the characteristics of different APCDs applied upstream of the flue gas. The results of PAS experimentation indicate that as the PAS temperature is increased to 250 °C, the recovery of vapor phase PCDD/Fs is over 100%. It might be attributed to two causes; one is that the PCDD/F adsorbent cannot effectively adsorb vapor phase PCDD/Fs as the system temperature is over 250 °C. The second possible cause is that significant amounts of PCDD/Fs are formed via de novo synthesis at 250 °C with high HCl concentration in gas stream (Table 1) and may eventually vaporize from the PCDD/F adsorbent to exist as vapor phase. Additionally, high amount of PCDD/Fs were generated at 250 °C, confirming the hypothesis of de novo synthesis, which results in the increase of the recovery of vapor phase PCDD/Fs to over 100%.

5. Conclusions

This study aims to investigate the partitioning of PCDD/Fs between vapor and solid phases with temperature variation in an MWI and PAS experimentation, respectively. ACI technology can effectively remove vapor phase PCDD/Fs while BF is effective in removing solid phase PCDD/Fs as indicated in the MWI sampling results. Removal mechanism of solid phase PCDD/Fs relies on filtration mechanism of the bag filters. The solid phase PCDD/F and particle removal efficiencies achieved with bag filters are fairly close. The results of MWI sampling also indicate that there is optimal operating temperature (set 2) for PCDD/F removal with ACI. MWI sampling results also indicate that the slopes (m) of $\log(C_\mathrm{v}/C_\mathrm{s})$ versus $\log P_\mathrm{L}^0$ of PCDD/F congeners can represent the PCDD/F removal and formation characteristics between vapor/solid phases. In addition, the results of PAS experimentation indicate that the temperature variation (150–250 °C) significantly affects the partitioning of PCDD/Fs between vapor and solid phases. Transfer of vapor phase PCDD/Fs to solid phase increases as the chlorination level of PCDD/Fs congener increases at Group 1 (150 °C) and Group 2 (200 °C). As the temperature is increased to 250 °C (Group 3), de novo synthesis significantly affects the partitioning of PCDD/Fs between vapor/solid phases.

Acknowledgements

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