

Laboratory-scale study of the suppression of PCDD/F emission during coal and MSW co-incineration

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Abstract

The experimental test of co-incinerating Chinese raw municipal solid waste (MSW) and coal in a laboratory-scale tubular reactor was first reported in present study, and the emission of normal gas components and the effects of the S/Cl molar ratio or coal mixing percentages on polychlorinated dibenzo-*p*-dioxins and dibenzofurans (PCDDs/Fs) emission were investigated and discussed. The results indicated that OCDD was the only PCDD homologues since others like TCDD–HpCDD was hardly detected, while as the categories of PCDF homologues were comparatively much more general. The amount of PCDD was much larger than that of PCDF in all operating conditions. Since $\sum\text{PCDF}/\sum\text{PCDD}\ll 1$, the dominant role of the precursor formation was proven in our experimental conduction. With increasing the coal addition to MSW (from 0 to 16%), PCDD and PCDF were reduced considerably. Coal and MSW may suppress the PCDD/F emissions efficiently (over 95%) during the MSW incineration process. The PCDD/F suppression results of the present study could be helpful guidance to the industrial application of Chinese MSW and auxiliary coal co-incineration processes. The PCDD/F stack emission data of two industrial incinerators using co-incineration technology in China seem to have a great positive reduction of PCDDs/Fs.

Key words: co-incineration; PCDDs/Fs; suppression; S/Cl molar ratio

Introduction

The mechanism of PCDD/F formation during combustion processes is basically similar, whereas the predominant factors that affect the formation of PCDDs/Fs differ with the nature of the fuel, e.g. municipal solid waste (MSW) or coal. Due to the relatively complex components of MSW, its high water content and low heating value, etc., it is very difficult to operate a MSW incinerator for Chinese MSW in an auto-thermal manner. Moreover, MSW often contains chlorine sources, which upon combustion may result in a significant production of extremely toxic compounds like PCDDs/Fs (Khachatryan and Dellinger, 2003). Now in China the PCDD/F stack emission limit value for MSW incinerators is 1 ng I-TEQ/Nm³ (11% O₂; I-TEQ (International Toxicity Equivalence Quantity)), while in European Union is much more strict as 0.1 ng I-TEQ/Nm³ (11% O₂). By contrast, the coal combustion is comparatively easier, and its chlorine content is less important, thus implying that the formation of PCDDs/Fs is less promoted in combustion gas. The PCDD/F emission

during coal combustion is approximately 2–3 orders of magnitudes lower than that of burning MSW. The PCDD/F levels in stack gas of coal-fired utility boilers were examined in some countries, and measured as e.g. 0.001–0.04 ng I-TEQ/Nm³ (Germany), 0.02 ng I-TEQ/Nm³ (0.35 ng I-TEQ/kg coal, Holland), 0.076 ng I-TEQ/kg coal (USA), etc. (USEPA, 2001; Hunsinger *et al.*, 1998; Riggs *et al.*, 1995).

In order to sustain a stable combustion of low heating value of Chinese MSW (always in the range of 4000–6000 kJ/kg), auxiliary fuel is generally needed. Chinese coal, being abundantly and cheaply available, is the indicated auxiliary fuel. The co-firing of MSW and coal has gradually become an effective method to reduce the PCDD/F emission from municipal solid waste incinerators (MSWIs), as was proven when coal had been fed to a MSW or RDF incinerator (Stieglitz *et al.*, 1991; Lindbauer *et al.*, 1992; Raghunathan *et al.*, 1997; Lu Sheng-yong *et al.*, 2003).

The addition of certain compounds, such as S- and N-containing compounds had been proposed for their inhibition ability of PCDD/F formation during co-combustion or post combustion processes (Lindbauer *et al.*, 1992; Ogawa *et al.*, 1996; Buekens and Huang 1998; Sama-

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ras *et al.*, 2001; Ruokojarvi *et al.*, 2001). S-containing compounds can be available by adding directly as SO₂ gas or adding S-rich coal or feeding elemental sulfur. Experimental results indicated that the effect of S-rich coal on the formation of PCDDs/Fs was more efficient than adding SO₂ gas (Ogawa *et al.*, 1996; Buekens and Huang 1998).

The medium-scale combustion experiment of natural gas demonstrates that the quantity of PCDDs/Fs produced is very high in the presence of HCl but without SO₂ (S/Cl = 0) (Raghunathan *et al.*, 1996). It decreases considerably when the molar ratio of S/Cl is 0.64, however, without a further effect with increasing of the S/Cl-ratio.

The incinerator working practice of co-firing coal and MSW demonstrates that the formation of PCDDs/Fs in the low temperature zone is related to the molar ratio of S/Cl in the fuel. The typical ratio in municipal solid waste incinerator (MSWI) is 0.2, while in coal combustion, such ratio is at least one order of magnitude higher. When the ratio is up to 10, PCDD/F formation in the low temperature zone is suppressed by approximately 90% (Lutho *et al.*, 1998). Co-firing coal and MSW have been investigated that an S/Cl ratio between 1 and 5 might significantly decrease the emission of PCDDs/Fs (Lindbauer *et al.*, 1992).

Griffin (1986) first postulated that the relative high sulfur content of coal was the reason of reducing PCDD/F emission in the coal combustion. The suppression of PCDD/F formation during coal and MSW co-combustion is attributed to the sulfur oxides. The Cl₂ formed from Deacon reaction is considered as the main source of chlorine, which chlorinated the aromatic compounds through substitutive reactions (Griffin, 1986). SO₂ in combustion gas could react with Cl₂ to consume chlorine agents (Cl*) which plays an important role in the formation of PCDDs/Fs. The reactions between sulfur and metal catalysts present in the fly-ash (i.e., CuO), change the reactivity of catalyst, thus suppress the ability of forming Cl₂ by the Deacon reaction via catalyst. In the process of combustion, sulfur oxides may reduce the synthesis of PCDDs/Fs through the formation of sulfate-phenol precursors (Gullett *et al.*, 1992).

The effect of sulfur containing compounds on the suppression of PCDDs/Fs is not due to SO₂, but to SO₃, which deactivates the catalytic reaction of the fly-ash surface (Lindbauer *et al.*, 1994; Bobik *et al.*, 1996; Huang and Buekens, 1999). Some research groups also find promotion effects to PCDD/F formation using elemental sulfur as reactants (Stieglitz *et al.*, 1991; Bechtler, 1998; Yan *et al.*, 2003).

In this article, co-firing coal and Chinese raw MSW in a laboratory-scale tubular furnace reactor was first conducted and reported to support the mechanism of PCDD/F suppression during coal and MSW co-combustion. The PCDD/F suppression results from adding different weight percentage of coal to the combustion process of Chinese raw MSW will be illustrated through analysis and comparison.

1 Experimental

1.1 Materials

Raw Chinese MSW was sampled and the typical physical characteristics shown in Table 1.

Table 1 Composition of raw MSW (wt%)

| Kitchen waste | Residue | Paper | Textile | Wood and bamboo | Plastic | Others |
|---------------|---------|-------|---------|-----------------|---------|--------|
| 56 | 5 | 11.3 | 7.4 | 5.5 | 13 | 1.8 |

The raw MSW was air-dried at 100°C for 12 h, and ground to powder (<125 μm) for further analysis and experiments. Primary and ultimate analysis was carried out for the air-dried raw MSW powder as shown in Table 2.

1.2 Experiment methods

The laboratory-scale horizontal tubular furnace of Fig.1 was used for the experiments. The control temperature range for this tubular furnace is 200–1200°C (±2°C). The laboratory reactor consists of a quartz tube with I.D. of 16 mm and a length of 400 mm, which is placed centrally into the electric heating furnace for about 250 mm of its total length. The residence time of the combustion gas in the furnace is averagely estimated to be 1.0 s with air flow 300 ml/min. Prior to be vented to the atmosphere, the combustion gas is adsorbed on XAD-2 resin and absorbed in two toluene (Made in Tedia, USA. HPLC) impinges placed in an ice-water bath. The gas phase PCDDs/Fs is then captured by XAD-2 and toluene. The XAD-2, toluene and quartz pipe rinses are mixed for further analysis of gas phase PCDDs/Fs or together with solid phase PCDDs/Fs.

The experiment contains five operation conditions. The first run is blank; the second to the fifth (LA1-LA4) are combustions of MSW with added coal (0, 5%, 16% and 44%). The experimental operating conditions and parameters are shown in Table 3.

Each experiment from the second to the fifth run burned 1.5 g MSW in consecutive batches of 0.3 g each with 6 min combustion time. In the first 0.3 g MSW burning, the combustion gas was sampled by a 100-ml syringe and analyzed for CO, CO₂, CH₄ and H₂ by gas chromatograph. Normal gas emission in the first minute of each first combustion batch of 0.3 g fuel is shown in Table 4.

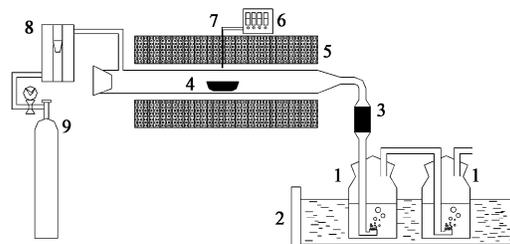


Fig. 1 Horizontal tubular furnace and associated equipment. (1) absorbent (toluene, AC); (2) ice-water bath; (3) adsorbant resin (XAD-2); (4) tubular reactor; (5) electric furnace; (6) temperature controller; (7) thermocouple; (8) gas flow meter; (9) air supply.

Table 2 Primary and ultimate analysis of raw MSW and coal with air dry basis

| Fuel | Primary analysis (wt%) | | | | Ultimate analysis (wt%) | | | | | | Heating value (kJ/kg) |
|-------------------|------------------------|-------|-------|-------|-------------------------|------|------|-------|-------------------|------|-----------------------|
| | M | A | V | FC | C | H | N | O | S | Cl | |
| MSW | 3.07 | 46.70 | 41.57 | 8.66 | 27.36 | 3.30 | 1.80 | 16.94 | 0.22 ^a | 0.61 | 10854 |
| Coal ^b | 2.13 | 29.72 | 19.50 | 48.65 | 58.19 | 3.68 | 1.01 | 4.76 | 0.40 ^a | 0.11 | 23729 |

^a The data are the global sulfur content; ^b the coal used is an anthracite. M: moisture; A: ash; V: volatile compounds; FC: fixed carbon; C: carbon; H: hydrogen; N: nitrogen; O: oxygen; S: sulfur; and Cl: chlorine.

Table 3 Design of operating conditions and parameters in combustion tests

| Number | Reaction temperature (°C) | Excess air coefficient (α) | Air flow (ml/min) | S/Cl molar ratio | Ratio of coal added (wt%) |
|--------------|---------------------------|-------------------------------------|-------------------|------------------|---------------------------|
| Raw MSW | – | – | – | – | – |
| Blank sample | 850 | – | 300 | 0 | 0 |
| LA1 | 850 | 1.5 | 220 | 0.40 | 0 |
| LA2 | 850 | 1.5 | 240 | 0.43 | 5 |
| LA3 | 850 | 1.5 | 280 | 0.52 | 16 |
| LA4 | 850 | 1.5 | 450 | 0.85 | 44 |

LA1-LA4 are the second to the fifth combustions of MSW with adoled coal (0, 5%, 16%, and 44%).

Table 4 Conventional gas emissions in each operation condition

| Number | CO ₂ (%) | CH ₄ (%) | CO (%) | H ₂ (%) |
|--------------------------------|---------------------|---------------------|--------|--------------------|
| LA1-1 (the 1st min combustion) | 14.86 | 4.00 | 9.62 | 0.95 |
| LA2-1 (the 1st min combustion) | 15.47 | 4.40 | 8.97 | 2.30 |
| LA3-1 (the 1st min combustion) | 15.31 | 3.40 | 7.66 | 3.01 |
| LA4-1 (the 1st min combustion) | 14.38 | 2.02 | 5.49 | NA |

NA: stands for data not available.

Due to the limitation of the experimental set-up and fuel characteristics, fuel was not fed continuously and was not always equally well prepared and homogenized. Data at late stages of the combustion are therefore less reliable. Further work is needed to improve the experiment method to make the results more reliable.

Before next four burning steps, the reaction tube was thereafter completely rinsed by toluene, dried by nitrogen flow (99.9%) and baked on 850°C for 15 min. During the second to the fifth 0.3 g MSW burning, the combustion gas was sampled for analysis of PCDD/F concentrations by HRGC/LRMS (Voyager Trace 2000, Thermal Finnigan, USA). The burning interval took approximately 0.5–1 min, which was needed to retrieve the previous matrix and insert the next into the reaction area with subsequent air-proof closure of the quartz tube. During this change-over interval, PCDD/F sampling stopped automatically. The PCDD/F sample was the sum of four burnings, while the ultimate PCDD/F formation data for per unit MSW was the average of it. No duplicated experiment run was done.

The distribution of PCDDs/Fs in gas and residue in sample boat of LA1 and LA3 was measured in the experiment. As the analysis results showed, PCDDs/Fs in residue were not detected since they were below the instrument detection limit (1 pg/ μ l).

1.3 Sample analysis method

The sample analysis following the common applicable method is described below. Each sample was spiked by 10 μ l isotope internal standards consisting five ¹³C isotopes (¹³C-2,3,7,8-TCDD, ¹³C-2,3,7,8-TCDF, ¹³C-1,2,3,6,7,8-HxCDD, ¹³C-1,2,3,4,6,7,8-HpCDF, ¹³C-OCDD) and then

Soxhlet extracted by 250 ml toluene for 24 h. The extract is condensed to 1–2 ml by rotary evaporation. The condensed solution has to be purified by multistage chromatography columns, i.e., acidic/alkaline silica gel column, acidic alumina column and another small acidic alumina column. The cleaned-up eluant was condensed to 1–2 ml, then transferred into a centrifugal tube and gently blown to dry by high purity nitrogen. Thereafter, 10 μ l recovery standard (¹³C-1,2,3,4-TCDD; ¹³C-1,2,3,7,8,9-HxCDD) was added to the dry centrifugal tube and subject to sonication for 15 min. ¹³C-1,2,3,4-TCDD was used to measure the amount of Te-PeCDD/Fs, ¹³C-1,2,3,7,8,9-HxCDD was used for Hx-OCDD/Fs.

All analyses were performed in a low resolution Finnigan Voyager mass spectrometer (LRMS) with a trace 2000 high resolution gas chromatograph (HRGC). Chromatographic separations are carried out on a 60-m DB-5 silica-fused capillary column with internal diameter of 0.25 mm and a stationary phase film thickness of 0.25 μ m. The temperature program for the GC oven is: initial temperature 100°C, held for 2 min; 100–200°C at 25 °C/min; 200–280°C at 3°C/min; 280°C held for 20 min. The carrier gas is helium (99.999%) at 1 ml/min. Splitless sample injection was used. The mass spectroscopy was applied at the following conditions: electron impact ionization 70 eV; electron multiplier voltage 420 V; ion source temperature 220°C; interface temperature 250°C; selected ion monitoring mode (SIM). All the isotope standards were purchased from Wellington Laboratories, Canada, or Cambridge Isotope Laboratory, USA. The toxicity of the sample was presented in terms of MSW weight, using TEF-values according to the USEPA method 8280B. In this article, the recovery efficiencies for five ¹³C isotope internal standards are in the range of 66%–100%.

2 Results and discussion

2.1 PCDD/F formation in co-combustion tests

Fig.2a reveals the distributions of PCDDs homologues in the emissions. In the four experimental conditions,

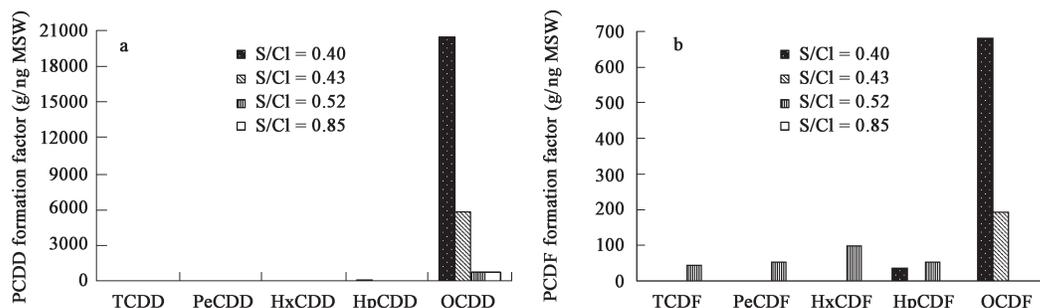


Fig. 2 Distribution of PCDD (a) and PCDF (b) homologues of co-combustion emissions.

the dominant PCDDs were OCDD. Small amount of HpCDD was detected in raw MSW combustion and TCDD–HxCDD were hardly present under all conditions. Fig.2b shows the distributions of PCDF homologues in the emissions. The amount and categories of furans differ greatly: OCDF was dominant with small amount of TCDFs and HpCDFs when burning raw MSW. When the molar ratio of S/Cl was 0.43, only OCDF was detected. When the ratio was 0.52, TCDF–HpCDF were present and without OCDF. PCDFs were almost undetected when it rose to 0.85. PCDDs always exceeded PCDFs under all the conditions. As far as the concentrations of Fig.2 are concerned, $\sum PCDF/\sum PCDD \ll 1$ stressed the importance of precursor formation rather than the de novo synthesis in our experimental conductions, which has been widely accepted by worldwide researchers as mentioned by Everaert and Baeyens (2002). This result differs from experiments using RDF, coal and elemental sulfur mixtures conducted by Palladas *et al.* (2004). PCDFs were the predominant homologues in their results. The rapid quench of the reactor and its quick removal during our experiments do not favor de novo synthesis, hence leading to the very small PCDF/PCDD-ratio. This is also an accepted fact.

Fig.3 reveals the relation between the molar ratio of S/Cl in the fuel and the total PCDD/F formation: PCDDs/Fs decreased significantly with the increase of coal/MSW-ratio (range from 0 to 16%); however, when the addition exceeded 16%, only small extra reduction of PCDDs/Fs was noticed.

Fig.4 shows that the I-TEQ emission declined clearly with increasing of S/Cl molar ratio in mixed fuel from 0.4 to 0.5. And only a slight decrease appeared when S/Cl molar ratio was over 0.5. It could be concluded that one critical ratio of auxiliary coal existed with S/Cl molar ratio around 0.5, and even though ratio of auxiliary coal

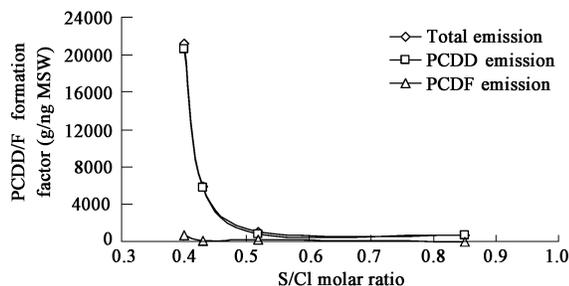


Fig. 3 Relation between the S/Cl molar ratio and total PCDD/F formation.

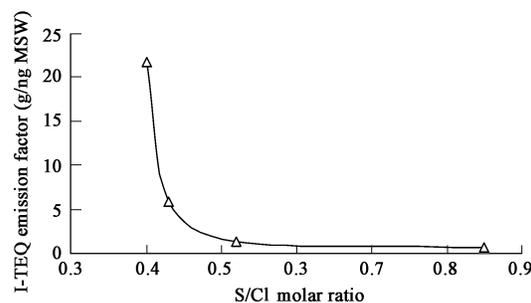


Fig. 4 Relation between the S/Cl molar ratio of fuel and PCDD/F I-TEQ emissions.

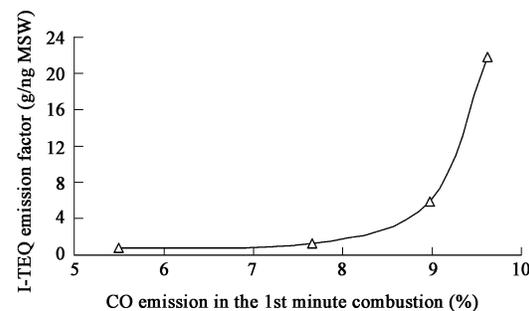


Fig. 5 Relation between the CO emission at the beginning of combustion and the PCDD/F I-TEQ emissions.

exceeded this value much, no great decrease appeared. It also could be found in Fig.4 that the relationship between I-TEQ emission and S/Cl molar ratio in the fuel followed one of non-linear function.

Fig.5 shows the relations between the CO emission in the beginning of combustion and that of PCDD/F I-TEQ emission per unit MSW. The more CO emitted at the beginning stage of the combustion, the more PCDDs/Fs was yield. The same possible dependency of PCDDs/Fs with CO in large-scale MSWIs was reviewed and evaluated by Everaert and Baeyens (2002).

Table 5 PCDD/F suppression effect with the coal addition ratio

| Coal addition ratio (wt%) | 0 | 5 | 16 | 44 |
|---------------------------|---|------|------|------|
| PCDD (%) | - | 72.0 | 96.1 | 96.6 |
| PCDF (%) | - | 82.5 | 66.1 | 100 |
| PCDD/F (%) | - | 72.4 | 95.2 | 96.0 |
| I-TEQ (%) | - | 73.0 | 94.2 | 96.0 |

Table 6 MSW composition analysis in two 300 t/d industrial MSW incinerators

| Physical composition (%) | | | | | | | | | |
|--------------------------|--------|-------|------|---------------|-----------------------|-------|---------|--------|------|
| Plastics | Rubber | Paper | Wood | Kitchen waste | Metal | Glass | Textile | Others | |
| 17.2 | 0.4 | 10.6 | 4.2 | 38.4 | 0.4 | 1.6 | 7.7 | 19.5 | |
| Primary analysis (%) | | | | | Ultimate analysis (%) | | | | |
| M | A | V | FC | Qnet (kJ/kg) | C | H | N | S | O |
| 52.16 | 10.54 | 26.56 | 2.86 | 6238 | 18.01 | 2.15 | 0.58 | 0.08 | 8.36 |

The data are the global sulfur content. M: moisture; A: ash; V: volatile compounds; FC: fixed carbon; C: carbon; H: hydrogen; N: nitrogen; O: oxygen; S: sulfur.

2.2 Suppression of PCDD/F formation during combustion

Table 5 lists the efficiency by which the coal addition suppresses the PCDD/F formation of MSW incineration, increasing from 70%–80% to over 95%.

Recent work achieved efficiency up to 85% when coal, RDF and sulfur were mixed at a weight percentage of 72/18/10 (Palladas *et al.*, 2004). In their experiments, S/Cl molar ratio was great higher than that in this study. Results of Table 5 indicate that: (1) coal and MSW may effectively suppress the PCDD/F formation during the MSW incineration process; (2) the addition of coal around 20% could result in a notable suppression efficiency up to 95%, while only minor increasing happened when the coal addition percentage rising to 44%.

2.3 PCDD/F stacks gas emission from industrial incinerators in China

Fluidized bed and grate incinerators are the most widely applied technologies in China to deal with raw MSW. Due to complex components and low caloric value of Chinese MSW (approximately 4000–6000 kJ/kg), co-firing MSW and auxiliary coal in fluidized bed is applied to maintain stable, complete burning and lower secondary pollution. PCDD/F stack gas emission data from two industrial incinerators using differential density circulating fluidized bed technology from Zhejiang University are presented here. The composition analysis of the feeding MSW in these two incinerators refers to Table 6. Table 7 lists the PCDD/F stack emission test results from these two 300 t/d industrial MSW incinerators.

The air pollutants control devices are semi-dry system plus bag filter and no activated carbon injection applied temporarily. It can be seen that the PCDD/F stack emission are far below the National Limitation 1 ng I-TEQ/Nm³, and also meet the European Union Limitation 0.1 ng I-TEQ/Nm³. From the industrial application data, it could partly demonstrate the significant suppression of PCDD/F emission by MSW and coal co-incineration technology.

Table 7 PCDD/F stack emission from two 300 t/d industrial MSW incinerators

| Incinerator | Fuel ratio (coal/MSW) | PCDD/F stack emission (ng I-TEQ/Nm ³ , 11% O ₂) |
|------------------|-----------------------|--|
| Qiaosi (300 t/d) | 19/81 | 0.06; 0.012; 0.0068 (three test results) |
| Qiaosi (300 t/d) | 22/78 | 0.0082; 0.0025 (two test results) |

3 Conclusions

The co-firing coal and MSW experiments in a tubular reactor are concluded that: (1) OCDD was the only PCDD homologues; (2) the amount of PCDD was much larger than that of PCDF under all operating conditions. Since $\sum PCDF/\sum PCDD \ll 1$, the dominant role of the precursor formation was proven in our experimental conductions; (3) with increasing the coal addition to MSW (from 0 to 16%), PCDD and PCDF were reduced considerably; (4) coal and MSW co-firing may suppress the PCDD/F formation efficiently (over 95% efficiency with 16% of mixed coal). In the stage of fuel combustion, less CO emission may lead to low PCDD/F formation.

The PCDD/F stack emission data from two industrial co-firing fluidized bed incinerators in China partly demonstrate suppressing effect of the auxiliary coal on PCDD/F formation and emission.

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