



Emission characteristics of dioxins, furans and polycyclic aromatic hydrocarbons during fluidized-bed combustion of sewage sludge

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Abstract

Pre-dried sewage sludge with high sulfur content was combusted in an electrically heated lab-scale fluidized-bed incinerator. The emission characteristics of polychlorinated dibenzo-*p*-dioxins (PCDDs), polychlorinated dibenzofurans (PCDFs) and polycyclic aromatic hydrocarbons (PAHs) were studied. Coal and calcium oxide (CaO) were added during the sewage sludge combustion tests to optimize combustion conditions and control SO₂ emission. The results indicated that the flue gases emitted during mono-combustion of sewage sludge were characterized by relatively high concentrations of SO₂, NO_x and organic pollutants, due to the high sulfur, nitrogen, and volatile matter content of sewage sludge. The total 16 USEPA priority PAHs and 2,3,7,8-substituted PCDD/Fs produced from sewage sludge combustion were found to be 106.14 μg/m³ and 8955.93 pg/m³ in the flue gas, respectively. In the case of co-combustion with coal ($m_{\text{sludge}}/m_{\text{coal}} = 1:1$), the 16 PAHs and 2,3,7,8-substituted PCDD/Fs concentrations were markedly lower than those found during mono-combustion of sewage sludge. During co-combustion, a suppressant effect of CaO on PCDD/Fs formation was observed.

Key words: co-combustion; PCDD/Fs; polycyclic aromatic hydrocarbons (PAHs); sewage sludge

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Introduction

In recent years, over 9.18 million tons of dry solid sludge are produced annually in China. The construction of modern, new wastewater treatment plants will lead to about 4% increase per year in total sludge production. It has been reported that, in China, only 3.45% of sewage sludge produced was incinerated, 31.03% of sewage sludge was land-filled, and more than 13.79% of sewage sludge was still waiting for disposal (Wang *et al.*, 2006). Because of the limitations facing other sludge disposal methods, such as land-filling and agricultural utilization, it is expected that sewage sludge incineration will increase rapidly in the next decade in China.

The major advantages of using incineration as a method for sewage sludge management are: reduction of its volume, complete destruction of pathogen and organic pollutants, and energy recovery. These benefits make incineration a very important disposal method in Japan, U.S., and some EU countries (Sakai *et al.*, 1989; Ogada and Werther, 1995; Werther and Ogada, 1999). The main disadvantage of sewage sludge incineration is the formation of hazardous air pollutants. Polychlorinated dibenzo-*p*-dioxins dibenzofurans (PCDD/Fs) and polycyclic aromatic hydrocarbons (PAHs) emissions from incinerators have

caused serious public concerns because these compounds are hazardous to human health. Samaras *et al.* (2000) studied the emission characteristics of PCDD/Fs during sludge combustion, through experiments carried out in a quartz-tube furnace. PCDD/Fs emissions ranged from 0.5 to 300 ng I-TEQ/kg fuel; the high values were owing to high concentrations of chloride and copper in the sludge. Mininni *et al.* (2004) presented incineration studies of sewage sludge both in a pilot circulating fluidized bed furnace and a rotary kiln furnace. The results revealed that organic chlorine had an obvious influence on PCDD/Fs emission and that a temperature of 950°C for afterburning chamber was high enough to prevent the appearance of PCDD/Fs in the raw gas (at concentration higher than 40 ng/Nm³). Fullana *et al.* (2004) studied the formation and destruction of chlorinated pollutants in detail during sewage sludge incineration in a dual chamber reactor. They found that the ash in the post-combustion zone induced the yields of PCDD/Fs, but the presence of HCl or CuCl₂ enhanced PCDD/Fs formation. Park *et al.* (2009) gave results from sewage sludge incineration in an industrial fluidized bed incinerator. PAHs produced from sewage sludge incineration was 6.1 mg/kg on average, and PAHs concentration in raw flue gas ranged from 3.9 to 925.7 μg/Nm³.

The objective of this article was to study the emission

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characteristics of PCDD/Fs and of PAHs during sewage sludge combustion. Since sewage sludge is usually characterized by a high moisture and sulfur content, auxiliary fuel and a desulfurizing agent are usually necessary during sludge combustion. In this study, a municipal sewage sludge was incinerated in a lab-scale fluidized bed. Coal and calcium oxide (CaO) were, respectively, used as auxiliary fuel and desulfurizing agent. The effects of coal and CaO on PCDD/Fs and PAHs formation were studied.

1 Materials and methods

1.1 Materials

In the experiments, a mechanically dewatered sewage sludge was sampled from a wastewater treatment plant in Hangzhou City, China. The moisture content of the sewage sludge was 3.69 kg moisture/kg dry solid. Coal was selected as auxiliary fuel. Analytically pure CaO was used as an additive to control SO₂ emission. The proximate and ultimate analysis of sewage sludge and coal are given in Table 1. Large amounts of organically bound fuel-nitrogen, sulfur, volatile matter and ash are observed in the sludge.

Table 1 Proximate and ultimate analysis of sewage sludge and coal

Proximate analysis	Sewage sludge	Coal	Ultimate analysis	Sewage sludge	Coal
Ash (%)	52.86	37.93	C (%)	26.05	54.10
Volatile (%)	43.15	6.65	H (%)	4.19	2.48
Fixed carbon (%)	2.52	54.09	N (%)	2.83	1.22
			S (%)	3.09	0.33
			O (%)	11.1	2.61
			Cl (%)	0.030	0.006
			Heating value (MJ/kg)	10.84	20.86

1.2 Experimental facility

The schematic diagram of the experimental apparatus is shown in Fig. 1. A lab-scale electrically heated fluidized bed was used, with 1100 mm in height. The external and internal diameters of the fluidized bed incinerator are 350 mm and 60 mm, respectively. The bed contains 360 g sand. Temperatures are measured at different spots (including the air pre-heater, the dense fluidized bed and the freeboard) along the reactor, by means of thermocouples placed in shielding tubes. The pre-heater and reactor tubes were heated by three independent electric external coils. This makes it possible to control the respective temperatures of the inlet air, the dense fluidized bed and the freeboard region independently.

A screw feeder with water cooling system was used to feed semi-dried sludge particles into the fluidized bed incinerator. The feeding rate was automatically controlled by an electromagnetic speed-adjustable motor. The water cooling system makes it possible that the fuel was not pre-heated during the sludge feeding process. Silica sand with diameter of 0.3–0.6 mm was selected as bed material.

PCDD/Fs and PAHs in the exhaust gas from the incinerator were sampled by a device (Fig. 1), which was constructed according to the USEPA method 23 (1997). The fly ash in the exhaust gas was collected by a filter cartridge, heated by heating tape to maintain a constant temperature of about 200°C in the filter. The heating of the filter cartridge was applied to simulate real conditions in full-scale combustion units, where the flue gases pass through the filter devices at temperatures of about 200°C. After the filter cartridge, the gaseous sample was cooled by a condensing tube to enhance the sorbing ability of XAD-2. Finally, PCDD/Fs and PAHs were sorbed by XAD-2 resin placing after the condensing tube. The water vapor in the flue gases was collected by a flask and

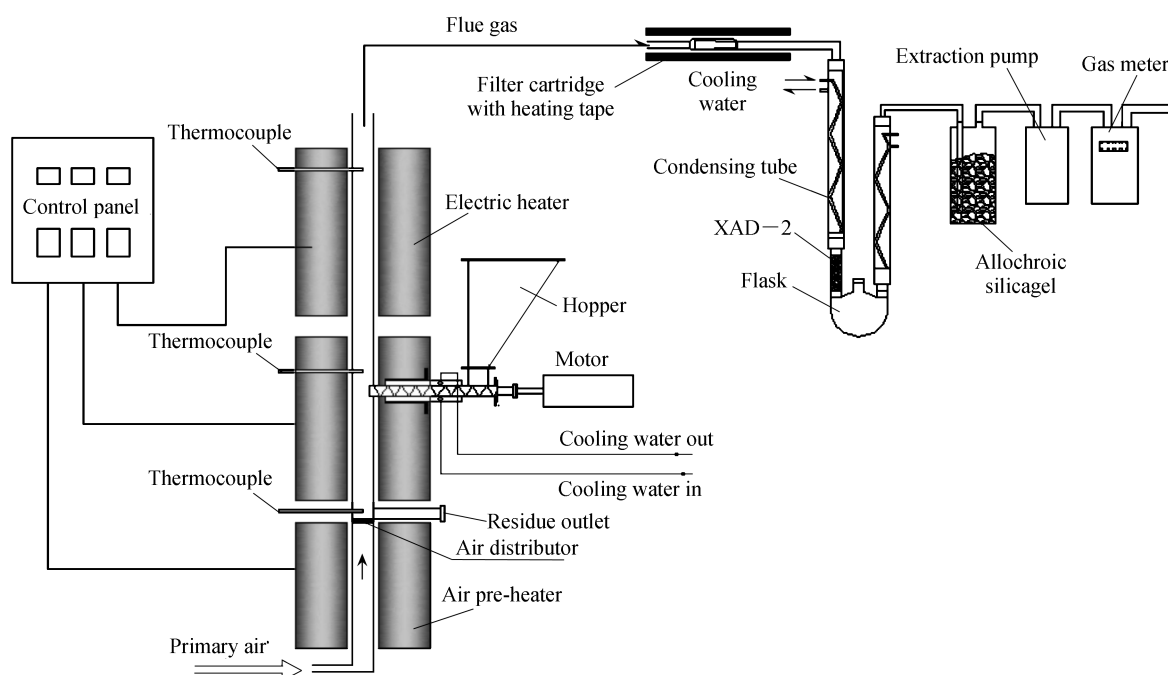


Fig. 1 Fluidized bed incinerator of sewage sludge and PCDD/Fs and PAHs sampling device.

allochroic silicagel. The humidity of the flue gases can be determined by condensation of moisture in a flask and allochroic silicagel. The total volume of dried flue gases was measured by a gas meter.

Pollutant concentrations in the flue gases were continuously measured by means of an on-line gas analyzer (Gasmeter DX-4000, Gasmeter Technologies, Finland). This analyzer is mainly composed of two units: a sampling unit for gas sampling and cleaning, and a measuring unit for their continuous analysis with a Fourier transform infrared (FT-IR) detector.

1.3 Experimental procedure

The sewage sludge was firstly dried by a Nara-type paddle dryer to a moisture content of 0.67 kg moisture/kg DS before incineration. The sludge changed from a pasty phase to a granular material (3–5 mm in diameter), due to the agitation effect of the paddles during drying process. During combustion, the temperature of the fluidized bed and freeboard were controlled at about 850°C by the electric heater as well as by the feeding rate of the dried sludge product. In addition to the mono-combustion sewage sludge experiment, co-combustion experiments were conducted using a pre-mixed fuel, containing 50% (*m/m*) of coal and 50% (*m/m*) of dried sludge. A mono-combustion experiment of coal was also carried out, for comparison with other combustion conditions. CaO was added to the fuel to control SO₂ emission; CaO was mixed homogeneously with fuel before being introduced into the incinerator.

Sample pretreatment for PCDD/Fs analysis was performed according to the USEPA Method-1613 (1994). The PCDD/Fs analyses were conducted using a high-resolution gas chromatography coupled with a high-resolution mass spectrometry (HRGC/HRMS) (JEOL JMS-800D, Japan) with a DB-5MS column (60 m × 0.25 mm × 0.25 μm). The temperature program of the capillary column was as follows: (1) 150°C hold for 1 min; (2) increased at 25°C/min to 190°C; and (3) increased at 3°C/min to 280°C, hold for 20 min. The injection volume was 1 μL by automatic splitless injection. The MS was operated at a resolution of 10000 under positive EI conditions (38 eV electron energy), and the data were obtained in the selective ion monitoring mode. All isotope standards were purchased from Wellington Laboratories, Canada.

Sample pretreatment for PAHs analysis was based on Method SY/T5119-1995. The PAHs were analyzed by HRGC (Finnigan Voyager Thermal Trace 2000, Thermo-Finnigan Company, USA) with a DB-5GC column (30 m × 0.25 mm × 0.25 μm). The temperature program of the capillary column was as follows: (1) increased from 70 to 180°C at 3°C/min; (2) increased at 10°C/min to 270°C; and (3) 270°C hold for 20 min. Helium (99.999%) was used as carrier gas, and the flow rate was 1 mL/min. The temperature of the FID detector was 280°C.

2 Results and discussions

X-ray fluorescence analysis was used to determine the content of important elements for the formation of PCDD/Fs and PAHs. The measured data (average from 2 measurements) are in g/kg DS: Cd 0.19; Cr 0.68; Cu 0.10; Fe 31.61; Ni 1.61; Pb 1.17; Zn 10.45. Five different combustion conditions were tested during the experimental program (Table 2). The inorganic and organic pollutant concentrations are, respectively, reported in Table 2 and Fig. 2. Results reported below are average values derived from 30 min of continuous measurement. Table 2 shows that mono-combustion of sewage sludge (B) was characterized by high concentrations of SO₂ and NO_x, which is due to the high sulfur and nitrogen content of sewage sludge (Table 1). During co-combustion process (A) the emission concentrations of SO₂ and HCl were markedly reduced, due to the low sulfur and chlorine content in the coal.

Figure 2 shows the organic pollutants concentrations of methane, hexane, propane, *n*-heptane, cyclohexane, benzene and aniline in flue gases. These organic pollutants were markedly higher during sewage sludge mono-combustion than those emitted during all other tests. In fact, this phenomenon was caused by the high volatile matter content of sewage sludge as has been reported by Ogada and Werther (1995).

2.1 PAHs emissions

Emission characteristics of PAHs have been widely reported during the combustion of coal and municipal solid waste (MSW) (Liu *et al.*, 2001; Ni *et al.*, 2004; Ledesma *et al.*, 2000), but few researches focused on PAHs emissions from sewage sludge combustion. Recently, Park *et al.*

Table 2 Concentration of inorganic pollutants in flue gases

Pollutant	Concentration of inorganic pollutant (mg/m ³)				
	Condition A	Condition B	Condition C	Condition D	Condition E
CO	128.5	23.1	97.3	109.4	110.2
SO ₂	2248.1	4417.1	1215.7	234.0	19.0
NO	116.3	209.1	140.2	161.1	133.0
NO ₂	0.4	1.4	1.2	0.6	1.8
N ₂ O	165.8	97.5	106.3	79.7	21.5
NH ₃	1.7	7.3	0.9	1.0	1.5
HCl	13.2	72.1	4.4	1.9	1.2
HF	0	0.1	0.4	0.2	0.1
HCN	12.2	14.3	15.4	7.9	3

A: sewage sludge co-combustion with coal ($m_{\text{sludge}}:m_{\text{coal}} = 1:1$); B: sewage sludge mono-combustion (moisture content of 0.67 kg moisture/kg DS); C: co-combustion with CaO addition ($m_{\text{sludge}}:m_{\text{coal}} = 1:1$, Ca/S = 2); D: co-combustion with CaO addition ($m_{\text{sludge}}:m_{\text{coal}} = 1:1$, Ca/S = 4); E: coal mono-combustion.

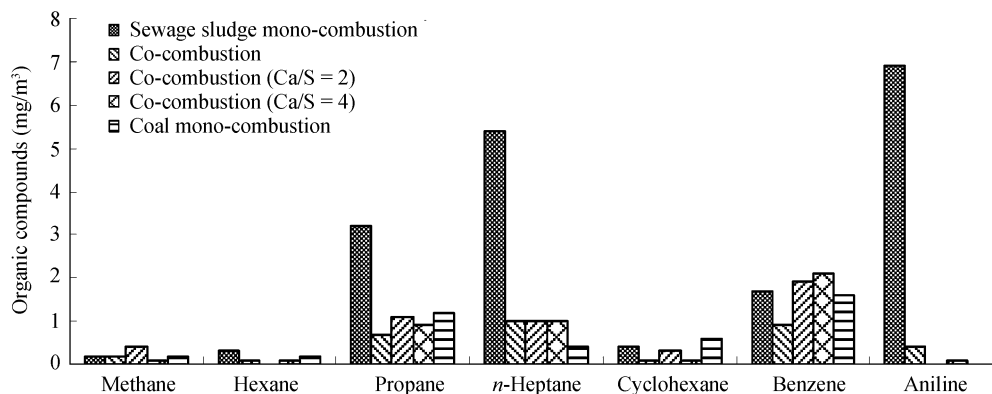


Fig. 2 Organic compound concentrations in flue gases.

(2009) reported PAHs emission from industrial fluidized bed incinerators of sludge in Korea. Their results indicated that the PAHs concentrations in raw flue gases range from 3.9 to 524 $\mu\text{g}/\text{m}^3$. In present study, PAHs emission concentrations during sewage sludge combustion are given in Table 3, which illustrates the results on 16 PAHs listed on the USEPA priority list. Table 3 reveals that the PAHs of 3-rings are dominant in the flue gas from sewage sludge mono-combustion; total PAHs concentration of 106.7 $\mu\text{g}/\text{m}^3$ was detected. This value is markedly higher than the values obtained from coal mono-combustion and sludge and coal co-combustion (A, C, D and E). The reason is likely due to the properties of the sewage sludge which is characterized by high volatile and low fixed carbon content. Our previous research on PAHs emission during combustion of different types of coals also revealed that the fuel with the higher volatile content and lower fixed carbon content led to a higher PAHs emission (Li *et al.*, 2003). The emission concentration of PAHs from the co-combustion process (A) was close to that from the coal mono-combustion. It seems that CaO has no obvious effect

on PAHs emissions during co-combustion.

The reported values for PAHs in fly ash vary with the kind of fuel and type of incineration facility examined. For fly ash emitted from medical waste combustion, values between 13.8 and 47 $\mu\text{g}/\text{g}$ have been reported (Lee *et al.*, 2002). The fly ash from coal combustion has been found to contain 1.4–1077.2 $\mu\text{g}/\text{g}$ of PAHs. Table 3 also shows the PAHs content in the fly ashes under different experimental conditions. It can be found that the PAHs of 5-rings are dominant in the fly ash from the mono-combustion of the sewage sludge, which led to the PAHs concentration of 43.0 $\mu\text{g}/\text{g}$. This is the highest value of the five experimental conditions. The PAHs content in the fly ashes are markedly decreased to 6.2 $\mu\text{g}/\text{g}$ during the co-combustion processes, because the formation PAHs of 5-rings was significantly prohibited during the co-combustion processes.

2.2 PCDD/Fs emissions

Tables 4 shows the dioxins concentrations and I-TEQ values in the flue gases and fly ashes. It was found that 2,3,7,8-substituted PCDD/Fs concentrations in the flue

Table 3 PAHs concentration in flue gases and fly ashes

PAHs	PAHs concentration in flue gases ($\mu\text{g}/\text{m}^3$)					PAHs concentration in fly ashes ($\mu\text{g}/\text{g}$)				
	A	B	C	D	E	A	B	C	D	E
Naphthalene	0.04	2.58	0.72	0.10	ND	ND	ND	ND	ND	ND
PAH of 2-rings	0.04	2.58	0.72	0.10	ND	ND	ND	ND	ND	ND
Acenaphthylene	0.48	1.84	1.11	0.02	0.38	ND	ND	ND	ND	ND
Acenaphthene	0.66	1.70	4.51	0.01	2.36	0.01	ND	0.09	ND	0.01
Fluorene	1.23	24.14	5.12	0.20	1.53	0.03	0.16	0.02	ND	ND
Phenanthrene	4.81	34.55	4.25	3.65	8.87	2.44	0.53	0.57	0.09	1.35
Anthracene	0.80	12.73	0.69	3.52	0.01	0.06	0.08	ND	ND	0.01
Fluoranthene	5.16	4.10	2.53	0.91	5.08	1.11	0.45	0.98	0.11	0.59
ΣPAHs of 3-rings	13.14	79.06	18.21	8.31	18.22	3.65	1.22	1.66	0.20	1.96
Pyrene	3.45	2.49	3.57	0.50	4.75	0.65	0.13	0.74	0.11	1.12
Benzo[a]anthracene	2.31	6.26	1.72	0.95	0.88	0.18	0.03	0.17	0.07	0.24
Chrysene	0.92	6.73	0.99	0.07	0.54	0.42	0.30	1.16	0.18	0.56
ΣPAHs of 4-rings	6.68	15.48	6.29	1.52	6.17	1.25	0.46	2.07	0.36	1.92
Benzo[b]fluoranthene	0.02	2.04	1.74	0.07	0.11	0.41	1.64	1.71	0.51	0.25
Benzo[k]fluoranthene	0.01	2.40	1.20	0.02	0.32	ND	0.62	ND	0.03	0.01
Benzo[e]pyrene	1.52	1.76	2.28	0.06	0.30	0.07	0.01	0.12	0.84	0.12
Benzo[a]pyrene	0.05	1.40	0.09	ND	0.05	0.85	0.01	ND	0.17	0.65
Indeno[1,2,3-cd]pyrene	0.98	0.23	0.81	0.81	ND	ND	38.94	3.37	ND	ND
Dibenzo[a,h]anthracene	ND	0.33	ND	0.59	0.19	ND	0.13	ND	ND	ND
ΣPAHs of 5-rings	2.58	9.56	6.12	1.55	0.97	1.33	41.35	5.2	1.55	1.03
ΣPAHs	22.44	106.68	31.34	11.48	25.36	6.23	43.03	8.93	2.11	4.91
TEQ concentration	0.67	3.88	0.99	0.83	0.48	0.96	4.30	0.66	0.33	0.77

ND: non-detectable. Conditions A–E are the same as those in Table 1.

Table 4 2,3,7,8-Substituted PCDD/Fs concentrations in flue gases and fly ashes

PCDD/Fs	PCDD/Fs concentrations in flue gases (pg/m ³)					PCDD/Fs concentration in fly ashes (pg/g)				
	A	B	C	D	E	A	B	C	D	E
2,3,7,8-TCDDg	ND	ND	0.02	0.05	ND	ND	0.02	0.02	ND	1.14
1,2,3,7,8-PCDD	ND	ND	0.25	0.03	ND	0.56	0.01	0.22	0.02	1.24
1,2,3,4,7,8-HexCDD	1.64	3.68	1.01	0.58	0.12	ND	1.99	0.01	0.02	1.50
1,2,3,6,7,8-HexCDD	2.30	18.31	2.18	4.35	2.52	5.46	1.48	6.89	2.98	0.29
1,2,3,7,8,9-HexCDD	ND	ND	5.69	0.12	3.46	9.45	1.63	15.66	4.65	8.89
1,2,3,4,6,7,8-HepCDD	77.87	178.96	32.43	25.63	0.25	17.97	24.32	14.84	7.74	10.85
OCDD	1352.39	7265.29	781.67	225.65	51.23	280.56	289.15	210.23	40.81	24.37
2,3,7,8-TCDF	ND	ND	0.12	1.23	0.34	0.01	0.01	0.03	0.12	1.12
1,2,3,7,8-PCDF	6.34	62.36	0.03	0.56	ND	2.21	0.23	0.22	ND	3.33
2,3,4,7,8-PCDF	ND	59.93	0.89	3.25	0.23	ND	2.56	0.12	0.51	0.02
1,2,3,4,7,8-HexCDF	6.33	37.61	5.18	0.89	1.12	2.45	3.36	0.02	0.03	2.56
1,2,3,6,7,8-HexCDF	4.64	146.03	10.23	1.21	ND	1.36	2.12	1.23	0.56	5.98
1,2,3,7,8,9-HexCDF	ND	15.19	ND	ND	2.85	5.28	3.25	8.88	7.72	3.44
2,3,4,6,7,8-HexCDF	ND	ND	ND	5.68	ND	ND	9.45	0.18	5.68	9.01
1,2,3,4,6,7,8-HepCDF	17.72	416.45	2.58	5.14	3.37	3.78	7.89	1.19	ND	3.31
1,2,3,4,7,8,9-HepCDF	ND	ND	ND	ND	1.16	ND	7.56	2.26	1.12	2.78
OCDF	65.23	752.11	13.95	15.65	45.53	25.58	56.66	42.62	14.39	25.48
PCDD/Fs	1534.46	8955.93	856.23	290.02	112.18	354.66	411.71	304.61	86.35	105.31
I-TEQ concentration	4.18	69.14	4.18	3.67	1.30	3.32	4.39	3.93	2.58	5.43

gases released from the mono-combustion of the sewage sludge was 8996 pg/m³. This value is 1–2 orders of magnitudes lower than the values reported from the combustion of MSW (Zhong *et al.*, 2006; Zhang *et al.*, 2008). As shown in Table 4, the highest I-TEQ value of dioxins was 69.14 I-TEQ pg/m³ from the mono-combustion of the sewage sludge. This value is close to the values reported by Werther and Ogada (1999) from large scale sewage incineration plants in Germany. Werther and Ogada (1999) suggested that the relative lower PCDD/Fs concentration (in comparison with MSW) in sewage sludge was likely due to the high concentration of sulfur in sewage sludge. During the co-combustion process (A), the concentration of the 2,3,7,8-substituted PCDD/Fs in the flue gases dramatically decreased to 1536.46 pg/m³, and the I-TEQ value of dioxins decreased to 4.18 I-TEQ pg/m³. The prohibiting effect of the coal on the PCDD/Fs formation perhaps correlates with the lower chlorine and aromatic hydrocarbons concentrations in the flue gases, as shown in Table 2 and Fig. 2, since emitted gaseous chlorine can react with aromatic hydrocarbons to produce gas phase precursors or even PCDD/Fs (Lu *et al.*, 2007).

Table 4 shows that the concentrations of the 2,3,7,8-substituted PCDD/Fs during the co-combustion processes (C) decreased from 1534.46 to 856.23 pg/m³ with the CaO addition (Ca/S = 2), and further decreased to 290 pg/m³ when the ratio of Ca/S was 4. Thus, it revealed that CaO had an inhibiting effect on PCDD/Fs formation. The result of Wey *et al.* (2008) indicated that CaO markedly inhibited the formation of HCl and chlorophenols which are important precursors for PCDD/Fs formation. Liu *et al.* (2005) revealed that CaO can significantly inhibit PCDD/Fs formation from dioxin precursors. As shown in Table 4, in spite of the obvious differences in the concentration of 2,3,7,8-substituted PCDD/Fs, the I-TEQ values of the PCDD/Fs from the co-combustion processes (A, C, D) are almost identical. This indicated that CaO mainly prohibited the formation of high chlorinated PCDD/Fs.

The PCDD/Fs concentrations in fly ash vary with the

type of fuel and type of incineration facility. The values between 56 and 1082 ng/g in fly ashes from MSW incinerators have been reported by Chang and Chung (1997), which are markedly higher than the values from the sewage sludge combustion reported in present article. As shown in Table 4, the 2,3,7,8-substituted PCDD/Fs concentration in the fly ashes from the mono-combustion of the sewage sludge is 411.71 pg/g. The lowest value is 86.35 pg/g, which is from the co-combustion process (D) with the Ca/S ratio of 4. Table 4 shows that the I-TEQ values of the PCDD/Fs in the fly ash from the mono-combustion and co-combustion processes range from 2.58 to 4.39 I-TEQ pg/g. The value from the coal mono-combustion is higher than that from the sewage sludge mono-combustion. Besides, CaO has no obvious effect on I-TEQ value during the co-combustion processes.

3 Conclusions

In this study, a sewage sludge with high sulfur content was incinerated in a lab-scale fluidized bed incinerator. The results from on-line analysis of pollutants reveal that the flue gas from the mono-combustion of the sewage sludge contains relatively high concentrations of SO₂, NO_x and organic pollutants, due to the high content of sulfur, nitrogen and volatile matter in the material. PAHs and PCDD/Fs in flue gases were sampled, based on the USEPA method. The experimental results suggested that, during mono-combustion of sewage sludge, PAHs of 3-rings are dominant in the flue gas, while PAHs of 5-rings are dominant in the fly ash. The PAHs concentrations are markedly reduced during co-combustion with coal. The PCDD/Fs concentration in the flue gas from mono-combustion was 69.14 I-TEQ pg/m³, which is markedly lower than those reported for MSW incineration. It was found that PCDD/Fs further decrease when sewage sludge is co-combusted with coal. However, further work is still needed to investigate the possible links between PAHs and PCDD/Fs formation, and the effect of sulfur content in

sewage sludge on PAHs and PCDD/Fs emissions.

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