



Study on the Emission Characteristics of PCDD/Fs and Other Multiple Pollutants in Flue Gas from Sintering Plant in China

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Abstract: In China, sintering flue gas has become the most important source of air pollution rather than burning coal-fired flue gas. The emission characteristics and removal efficiencies of various gaseous pollutants were studied on a 180 m² sintering equipped with a semi-dry oxidative desulfurization and denitrification system in Tangshang, Hebei province. The emission concentrations of particulate matter, fluoride, NO_x and SO₂ were measured in the flue gas while polychlorinated dibenzo-p-dioxins and dibenzofurans (PCDD/Fs) were tested in both flue gas and dust ash at 100% and 60% load conditions. The collections and analyses of pollutants were used by smoke sampler, TESTO350 and dioxin sampler according to national standards of China. The results shown that the average emission concentration of particulate matter, fluoride, NO_x and SO₂ in the inlet of the flue gas purification system were 47.4 mg/m³, 0.12 mg/m³, 150 mg/m³ and 521 mg/m³ while the emission concentrations in the outlet were 1.2 mg/m³, 0.07 mg/m³, 24 mg/m³ and 20 mg/m³, respectively. Through the flue gas purification system, the PCDD/Fs concentrations were reduced from 23.12 ng/m³ to 1.14 ng/m³ at 100% load and from 12.25 ng/m³ to 0.84 ng/m³ at 60% load. The PCDD/Fs concentrations in the dust ash were about 178.56~197.21 ng/kg. The desulfurization and denitrification system significant reduced effect on the emission of particulate matter, fluoride, NO_x, SO₂ and PCDD/Fs with the value 97.5%, 41.7%, 84.0%, 96.2% and 95.1% at 100% load, respectively. The removal affects increases along the chlorination degree of PCDD/Fs. Meanwhile, the different load influenced the emission concentrations and removal rates of PCDD/Fs in the flue gas, the removal effects increases as the load increases. Although the desulfurization and denitrification system has good removal effects on various pollutants, the dioxins were enriched in the dust ash so as to cause secondary pollution.

Keywords: sintering flue gas, PCDD/Fs, gaseous pollutants, oxidative desulfurization and denitrification, removal effect

1. Introduction

Nowadays, steel industry has become an important pillar industry of the national economy. With the increase of steel production capacity. The problem of air pollution in China's steel industry become increasingly seriously. The flue gas of the sintering machine head is the main source of atmospheric pollutants in the steel plant. The pollutants emitted by the sintering machine head mainly include particulate matter, SO₂, NO_x, fluoride, and dioxins [1], especially dioxin emissions [2] account for more than one-third of the entire plant, and the sintering head flue gas is also the most important source of dioxin emissions in China except for waste incineration. At present, in China, it has a comprehensive study on the emission characteristics of atmospheric pollutants from coal-fired power plants and waste incineration plant [3-6], but the research on the emission characteristics of sintering heads has just started [4,7]. Compared with foreign studies [8,9], there is a lack of basic data on the emission characteristics of various pollutants in steel plants, as well as monitoring results under different operating loads [8-25].

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Sintering is currently the focus of China's iron and steel enterprises pollution emission. In order to improve the atmospheric environment, China's environmental protection department has set the most stringent ultra-low emission standards for the sintering flue gas, which were 10 mg/m³ for particulates, 35 mg/m³ for SO₂, 50 mg/m³ for NO_x, 3.0 mg/m³ for fluoride and 0.5 ng-TEQ/m³ for dioxins. And then many steel companies have installed a large number of new flue gas purification devices. In the treatment process, a variety of comprehensive dedusting, desulfurization, denitrification and dioxins removal technologies have been applied, which are including catalytic oxidation absorption method, medium temperature selective catalytic reduction(SCR) method, activated carbon/coke adsorption method, etc. However, the researches on the impact of purification devices on the emission characteristics of various pollutants is limited, which are not conducive to mastering the actual total contaminant emissions. In particular, a catalytic oxidation absorption technology of circulating fluidized bed has been applied to the treatment of steel sintering flue gas in Hebei province. The impact of this technology on the emission of various pollutants and the removal efficiency of the flue gas remains to be further studied.

At the same time, the sintered dioxin emissions problem has always been the focal point of attention of researchers. Dioxins as known as Polychlorinated dibenzo-p-dioxins and dibenzofurans(PCDD/Fs) listed by the Stockholm Convention(UNEP 2002)are a family of pollutants whose most toxic congener(2,3,7,8-TCDD) has been classified as carcinogenic for humans by the International Agency for the Research on Cancer[3,4,24]. In China, dioxin emission guidelines for iron ore sintering were first implemented in 2012. It is estimated that there are about 10.2 kg/TEQ of dioxin emissions per year from various sources, in China [26]. In order to control PCDD/Fs emissions,various end-of-pipe treatment technologies have been developed [27], such as catalytic decomposition using SCR catalysts [28,29], installing a fixed-bed adsorption system [30] and as well as spraying of powdered activated carbon [31] into flue gas and bag filter system with activated carbon adsorption [32-36] which the removal efficiency could reach to 95%, and even higher. But the researches on dioxin in China mainly focused on the emission concentration after sintering machine bellows and electrostatic precipitator(ESP). Conversely, the impact of new ultra-low emission purification devices on the emission of dioxin in flue gas and the distribution of dioxin in by-products of purification equipment are lacking.

Based on the multi-polluting purification device for sintering flue gas by oxidation absorption method, this study investigated the removal effects and total emissions of particulate matter, SO₂, NO_x and fluoride, as well as the distribution characteristics and purification effects of dioxins in the flue gas under two distinct load conditions. Furthermore, the dioxins in the by-products of the flue gas purification equipment was also tested to prove the basic reference for the harmless treatment of dust ash.

2. Materials and methods

2.1. Selecting a template

The experiments were carried out in a 180 m² sintering machine in Tangshang, Hebei province. In order to remove various harmful pollutants such as NO_x, SO₂, particulate matter,fluoride, PCDD/Fs, etc. from the sintering flue gas, the oxidative desulfurization and denitrificationtechnology were applied. The sampling points were shown in Figure 1. This sampling system mainly consists of three part, the sintering line unit, the oxidative desulfurization and denitrification unit(flue gas treatment unit) and gas emission unit. The sintering line unit includes sintering strand, ESP and fan. The oxidative desulfurization and denitrification unit includes catalytic oxidation device, circulating fluidized bed(CFB), bag filter and ash warehouse. The gas emission unit includes booster fan and stack.

Firstly, the flue gas enters the oxidative desulfurization and denitrification unit after through the preliminary treatment of the ESP. The catalytic oxidation device oxidized NO becomes NO₂ when the flue gas passing through. In the CFB system, the semi-dry technique was used. Meanwhile, the slaked

limes were injected into the CFB to remove target NO_2 and SO_2 . The slaked lime can be recycled by the bag filter and reused in the CFB, while the ashes were collected in the ash warehouse. Finally, the cleaned flue gas by the bag filter is discharged through the stack.

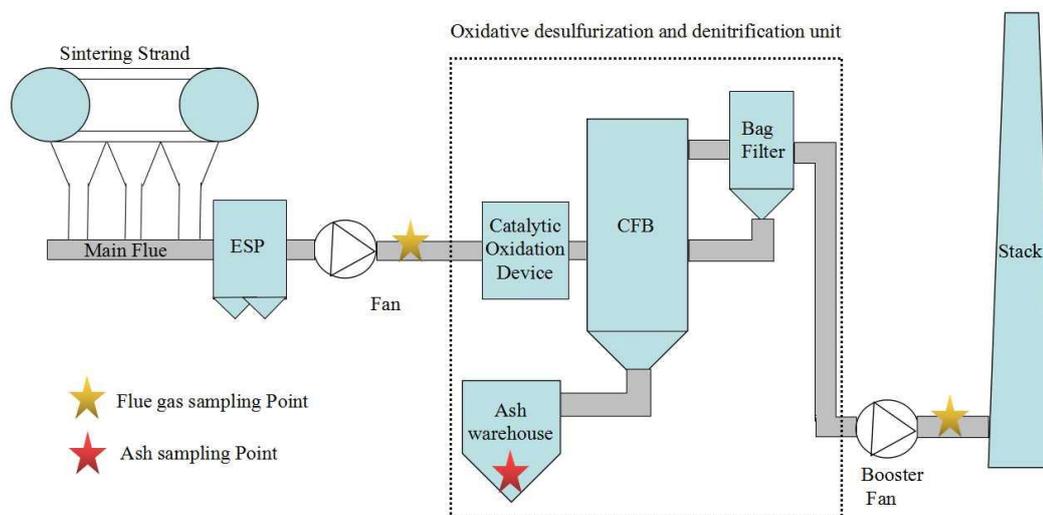


Figure 1. The sampling diagram

In this study we explored the effect of the oxidative desulfurization and denitrification unit on the removal of particulate matter, fluoride, NO_x , SO_2 and 2,3,7,8-substituted PCDD/Fs in flue gas and the 2,3,7,8-substituted PCDD/Fs content in the ash. The sampling points were defined before catalytic oxidation device as inlet and before stack as outlet. All targeted pollutants were both tested in the inlet and outlet at full load working condition, while PCDD/Fs were not only tested at 100% load but also at 60% load.

Table 1. Sampling parameters of flue gas.

Parameter	W1 Inlet	W1 Outlet	W2 Inlet	W2 Outlet
Temperature ($^{\circ}\text{C}$)	120	80	120	81
Moisture content (%)	8.8	9.3	8.8	9.5
Standard dry flow (m^3/h)	594,940	497,890	351,700	490,350
Oxygen content (%)	16.0	16.4	16.0	16.3
Flow velocity (m/s)	12.4	15.3	11.2	15.4

Working condition W1: 100% load.

Working condition W2: 60% load.

The smoke sampling parameters in both inlet and outlet were shown in **Table 1**. In the inlet, the standard dry flow of W1 is about $600,000 \text{ m}^3/\text{h}$, while the standard dry flow of W2 is about $350,000 \text{ m}^3/\text{h}$. Due to the presence of the booster fan, the outlet gas flow of 2 working conditions were all about $500,000 \text{ m}^3/\text{h}$.

2.2. Sampling and Quantification Methods

2.2.1. Particulate matter

The sampling of particulate matter is based on National Environmental Protection Standard of the People's Republic of China -- HJ836-2017 (i.e. Stationary source emission -- Determination of mass concentration of particulate matter at low concentration -- Manual gravimetric method). Before sampling, the front elbow, sealing aluminum ring and the steel support should be cleaned to remove the particles that may be adsorbed. At the same time, quartz filter should be baked for 1 h at $180 \text{ }^{\circ}\text{C}$. After cooling, the filter, steel plate and elbow are assembled together with a sealed aluminum ring.

2.2.2. Fluoride

The sampling of fluoride is based on National Environmental Protection Standard of the People's Republic of China -- HJ/T67-2001 (i.e. Stationary source emission -- Determination of fluoride -- Ion selective electrode method). The collected fluoride is stored in a polyethylene bottle. A sample of unknown concentration fluoride was titrated with hydrochloric acid using bromocresol green as indicator.

2.2.3. NO_x and SO₂

The sampling of NO_x and SO₂ are based on National Environmental Protection Standard of the People's Republic of China -- HJ 693-2014 (i.e. Stationary source emission -- Determination of nitrogen oxides--Fixed potential by electrolysis method) and HJ 57-2017 (i.e. Stationary source emission -- Determination of sulfur dioxide--Fixed potential by electrolysis method). Both NO_x and SO₂ were measured using an automatic tester (Testo 350). Place the front end of the analyzer sampling tube on the sampling point and ensure no air leakage.

2.2.4. PCDD/Fs

The sampling of 2,3,7,8-substituted PCDD/Fs is based on National Environmental Protection Standard of the People's Republic of China -- HJ/T77-2001 (i.e. Determination of polychlorinated dibenzo-*p*-dioxins and polychlorinated dibenzo-*p*-furans by isotope dilution HRGC/HRMS).

Each sample was spiked with ¹³C₁₂-labelled internal standards and then Soxhlet extracted by toluene. The extracts were purified with a series cleanup silica gel column, include sulfuric acid gel column and multi-layer silica gel column [37]. Samples were analyzed by DSF High Resolution Gas Chromatography-Mass Spectrometry. The total toxic equivalents (TEQ) concentration was calculated using international toxic equivalency factors (I-TEFs) [38-40].

3. Results and discussions

3.1. Removals of Particles, Fluoride, Nox, and SO₂

The emission concentrations of particulate matter, fluoride, NO_x and SO₂ were investigated in this study. The various substances in the flue gas were significantly reduced when going through the oxidative desulfurization and denitrification unit. The concentrations of particulate matter, fluoride, NO_x and SO₂ in both inlet and outlet and the removal rates (Figure 2).

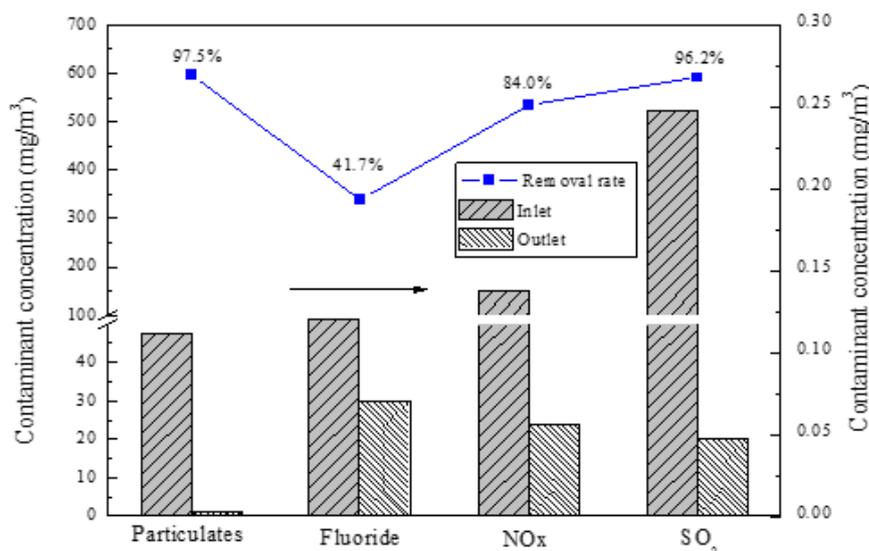


Figure 2. Concentrations and removal rates of particulate, fluoride, NO_x and SO₂ in inlet and outlet



In the inlet, the average emission values of particulate matter, fluoride, NO_x and SO₂ in the flue gas were 47.4 mg/m³, 0.12 mg/m³, 150 mg/m³ and 521 mg/m³, respectively. Meanwhile the outlet emission concentrations of those pollutants were tested as 1.2 mg/m³ (particulate matter), 0.07 mg/m³ (fluoride), 24 mg/m³ (NO_x) and 20 mg/m³ (SO₂). Obviously, the oxidative desulfurization and denitrification unit had excellent removal effects on particulate matter and SO₂ with the removal rates up to 97.5% and 96.2%, respectively. Meanwhile, the fluoride removal rate was the weakest among these pollutants removal rate with the value of 41.7%. The emissions of particulate matter, fluoride, NO_x and SO₂ were all lower than the latest ultra-low emission standards in China. And the total mass flow emission rates of particulate matter, fluoride, NO_x and SO₂ in the stack were 0.6 kg/h, 34.8 g/h, 12.0 kg/h and 10.0 kg/h, respectively.

3.2. Removals of PCDD/Fs

The flue gas treatment unit not only decreasing the concentrations of particulate matter, fluoride, NO_x and SO₂ but also had a significant removal effect on PCDD/Fs at the same time. The total emission concentrations of 2,3,7,8-substituted PCDD/Fs and I-TEQ values at 2 working conditions were shown in Tables 2 and 3.

Table 2. Concentrations and removal rates of 2,3,7,8-substituted PCDD/Fs in flue gas and ash at 2 working conditions in inlet and outlet.

	W1 Inlet	W1 Outlet	W2 Inlet	W2 Outlet	Ash1	Ash2
	ng/m ³				ng/kg	
∑PCDDs	2.69	0.10	1.67	0.10	449.93	327.29
∑PCDFs	20.43	1.04	10.58	0.74	1986.00	1854.10
TEQ-∑PCDDs	0.21	0.02	0.10	0.02	19.14	14.06
TEQ-∑PCDFs	2.23	0.15	1.03	0.09	178.07	164.50
∑PCDD/Fs	23.12	1.14	12.25	0.84	2435.93	2181.39
∑TEQ	2.44	0.17	1.13	0.11	197.21	178.56
Rate of PCDD/Fs						
PCDDs/∑PCDD/Fs	0.12	0.09	0.14	0.12	0.18	0.15
PCDFs/∑PCDD/Fs	0.88	0.91	0.86	0.88	0.82	0.85
PCDFs/PCDDs	7.58	10.02	6.34	7.17	4.41	5.67
TEQ-PCDDs/∑PCDD/Fs	0.09	0.12	0.09	0.14	0.10	0.08
TEQ-PCDFs/∑PCDD/Fs	0.91	0.88	0.91	0.86	0.90	0.92
TEQ-PCDFs/PCDDs	10.45	7.36	9.94	6.38	9.30	11.70
Removal rate (%)						
PCDDs	96.16		93.77			
PCDFs	94.93		92.97			
TEQ-PCDDs	90.43		85.10			
TEQ-PCDFs	93.26		90.43			
Total-PCDD/Fs	95.07		93.08			

Table 3. Total emissions and removal rates of PCDD/Fs in inlet and outlet.

Sampling	Total emissions (g/h)	Removal rate (%)
W1 Inlet	13.76	95.87
W1 Outlet	0.57	
W2 Inlet	7.29	94.26
W2 Outlet	0.42	

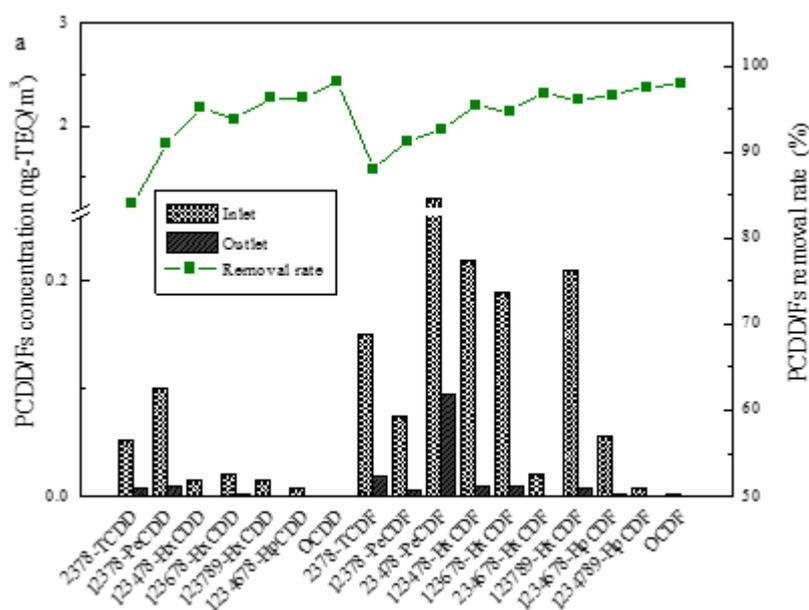
At full load, the mean emission values of 2,3,7,8-substituted PCDD/Fs in both inlet and outlet flue gas were 23.12 and 1.14 ng/m³, respectively. And the total TEQ values of 2,3,7,8-substituted PCDD/Fs were 2.44 and 0.17 ng·TEQ/m³. Due to the immaturity of current emission reduction technologies, the concentrations of PCDD/Fs produced during the steel sintering process were higher than the PCDD/Fs concentrations (0.0054–0.1961 ng·TEQ/m³) in flue gas from municipal solid waste with typical fluidized bed incinerators [41]. Conversely, the concentration of PCDD/Fs emitted during the steel sintering process was similar with the concentration of medical waste incinerators (MWIs), which the

total TEQs of PCDD/Fs in stack gas from MWIs were 0.031 to 3.463 ng·TEQ/m³ [42]. The oxidative desulfurization and denitrification unit showed a remarkable removal efficiency on 2,3,7,8-substituted PCDD/Fs with the removal rate of 95.07%, which mainly removed 96.16% of PCDDs. Meanwhile the total PCDD/Fs emission concentration was 13.76 g/h in the inlet and 0.57 g/h in the outlet, and the removal rate of total PCDD/Fs was 95.87%.

Compared with the full load, the emission values of 2,3,7,8-substituted PCDD/Fs at 60% load were slightly reduced to 12.25 ng/m³(concentration), 1.13 ng·TEQ/m³(TEQ) in the inlet and 0.84 ng/m³(concentration), 0.11 ng·TEQ/m³(TEQ) in the outlet. In addition, the emission value of 2,3,7,8-substituted PCDD/Fs decreased significantly with removal rate of 93.08% which was lightly lower than at full load. The total PCDD/Fs emissions in both inlet and outlet were 7.29 g/h and 0.42 g/h with removal rates of 94.26%, which were all lower than at full load. No matter W1 and W2, the PCDD/Fs emitted from the stack were all below the China emission standard of 0.5 ng·TEQ/m³.

Furthermore, the formation of PCDD/Fs in the flue gas at the steel sintering process was widely recognized as a typical *de novo* synthesis pathway [39,40]. Both inlet and outlet at 2 working conditions, PCDFs dominated among the total amounts of PCDD/Fs, which accounted for 86 to 91 % of the total yield. The rates of PCDFs to PCDDs were about 6.34 to 10.02(>1), which conformed to the characteristics of the *de novo* synthesis pathway (Table 2). Additional, distinct loads had a certain effect on the concentrations of PCDD/Fs. The full load had greater the concentrations of PCDD/Fs.

Figure 3 showed the concentrations and removal rates of 2,3,7,8-substituted PCDD/Fs homologues in the inlet and outlet at 2 working conditions. Obviously shown in Figures 3a and 3b, 1,2,3,7,8-PeCDD was the main component in PCDDs while 2,3,4,7,8-PeCDF was the main product in PCDFs in both inlet and outlet at W1. In addition, among the homologues of PCDD/Fs, OCDD and OCDF had the highest removal rates with 98.18% and 98.05%, respectively. Conversely, 2,3,7,8-TCDD was the worst removal rate at 83.96%. At W2, PCDD/Fs distribution showed a same trend in both inlet and outlet. OCDD and OCDF had the highest removal rate with the values of 96.63% and 97.83%. And also 2,3,7,8-TCDD showed the worst removal rate at 76.15%.



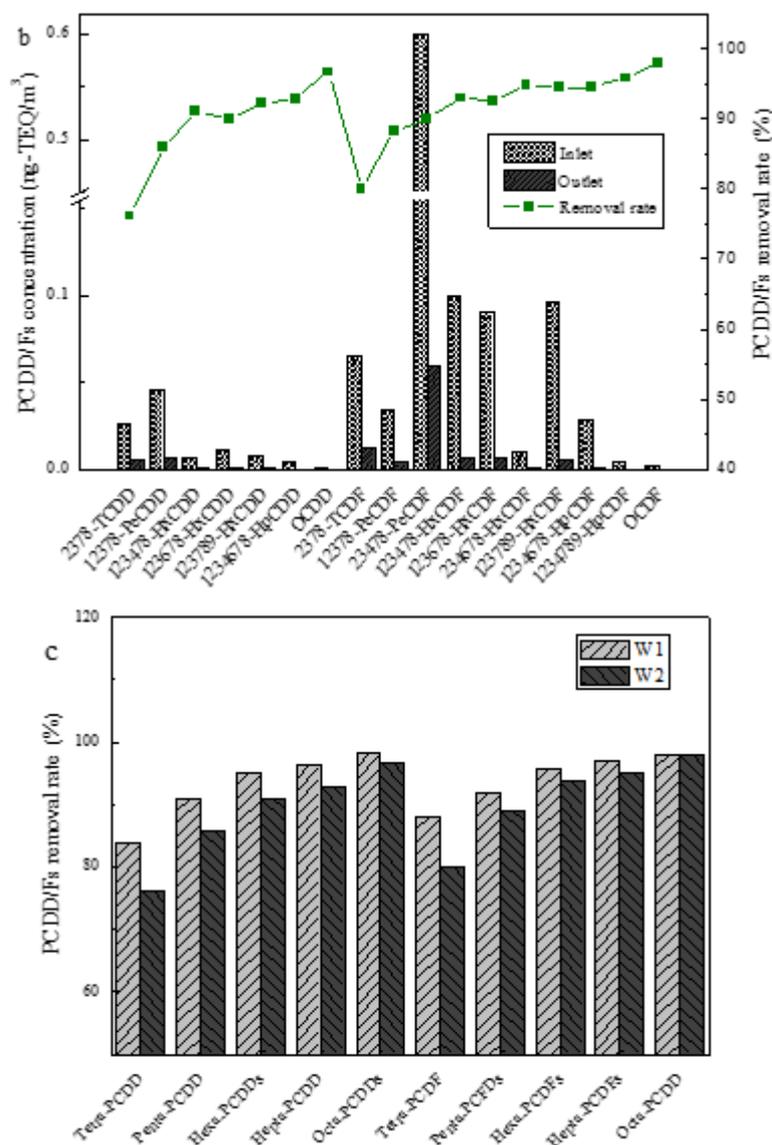


Figure 3. PCDD/Fs concentration and removal rate at W1(a) and at W2(b), PCDD/Fs homologues removal rate at W1 and W2(c).

Figure 3c summarized the removal rates of 2,3,7,8-substituted PCDD/Fs homologues at 2 working conditions. In general, compared with 60% load, the oxidative desulfurization and denitrification unit had a better effect on removing PCDD/Fs at full load. Moreover, the removal effect increased as the chlorination of PCDD/Fs increased at 2 working conditions. The removal rates were sorted as follows: Octa-PCDD/Fs > Hepta-PCDD/Fs > Hexa-PCDD/Fs > Penta-PCDD/Fs > Tetra-PCDD/Fs, which showed that the oxidative desulfurization and denitrification unit was more efficient to remove high chlorinated PCDD/Fs rather than low chlorinated PCDD/Fs. It is possible that another reason may lead to the result that slaked lime acted as both adsorbent and catalyst promotes dechlorination of PCDD/Fs.

3.3. Homologue Distributions of PCDD/Fs

The homologue distributions of PCDD/Fs were often referred to as its 'fingerprint' or 'signature', which was used to indicate the emission characteristics of PCDD/Fs [41,42]. Figure 4 shown the homologue distributions of PCDD/Fs 2,3,7,8-substituted were obtained from the flue gas at 2 working conditions.

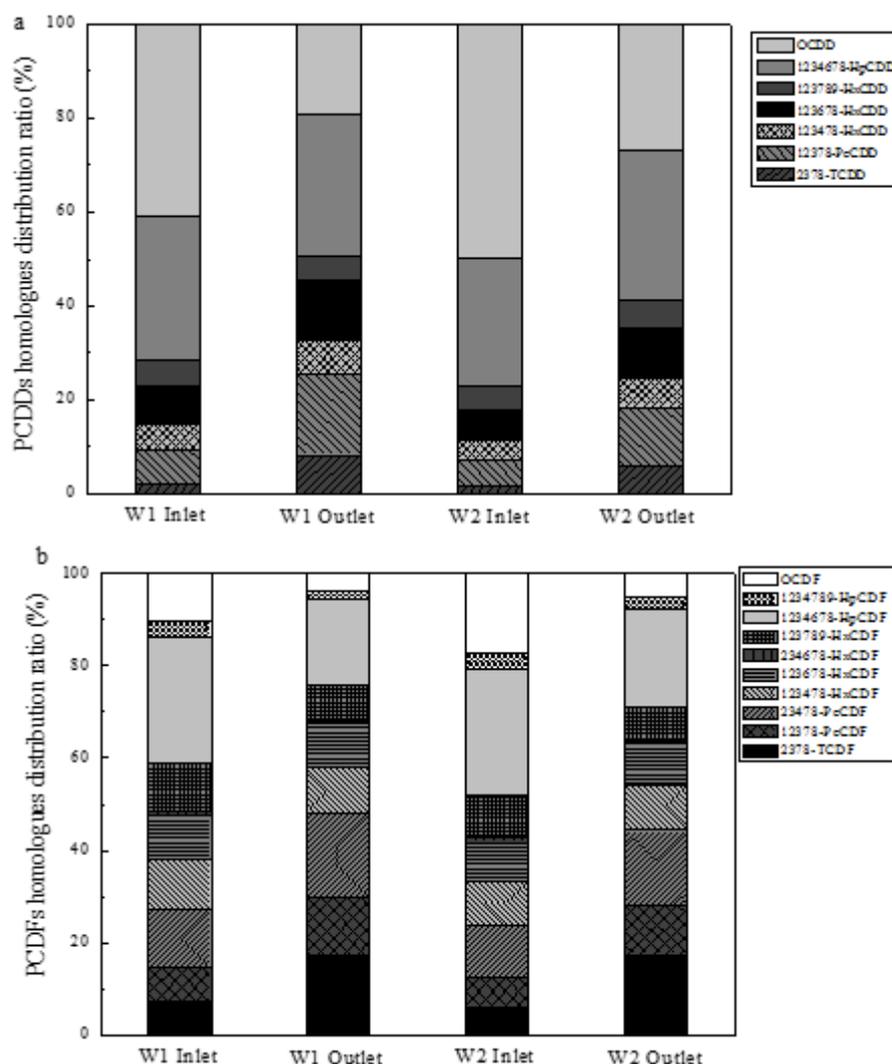


Figure 4. PCDDs(a) and PCDFs(b) homologues distribution ratio in flue gas at W1 and W2.

Whether W1 or W2 in the inlet, OCDD was the main component of PCDDs, accounting for about 40.85%(W1) and 49.70%(W2). Secondly, 1,2,3,4,6,7,8-HpCDD accounted for 30.82%(W1) and 27.54%(W2), while the ratios of other chlorine substituted PCDDs were below 10%, especially 2,3,7,8-TCDD only accounted for 1.97%(W1) and 1.56%(W2). For PCDFs in the inlet, 1,2,3,4,6,7,8-HpCDF dominated at 27.41% at 2 working conditions. In contrast, the ratios of 1,2,3,4,7,8,9-HpCDF accounted 1.03%(W1) and 0.95%(W2), which was the least proportion of PCDFs.

In the outlet, PCDDs homologous distribution in full load and 60% load showed similar characteristics. Compared with the inlet, 1,2,3,4,6,7,8-HpCDD became the main product rather than OCDD, OCDD rate ranked the second which was slightly lower than 1,2,3,4,6,7,8-HpCDD. The ratio of 1,2,3,7,8-PeCDD was more than doubled than in the inlet while 2,3,7,8-TCDD was still the least product. For PCDFs, the proportion of 2,3,7,8-TCDF was doubled approximately. Additionally, the rest chlorine substituted PCDFs were average distributed compared with inlet distribution. Furthermore, the 2 working conditions do not influence the distribution characteristics, which showed that the flow rate has no significant effect on the homologues distribution ratios of PCDD/Fs.

3.4. Distributions of Dioxins in Dust Ash

In order to figure out the comprehensive distribution characteristics of PCDD/Fs, the dust ash was also collected and analyzed in this study. The concentrations of PCDD/Fs in the dust ash at W1 and W2

were 2435.93 ng/kg(concentration), 197.21 ng·TEQ/m³(TEQ) and 2181.39 ng/kg(concentration), 178.56 ng·TEQ/m³(TEQ), respectively (Table 2). PCDD/Fs concentrations were far lower than that the average concentration of PCDD/Fs augmented from 73.47 ng/g in boiler ash to 95.49 ng/g in filter ashat waste incineration process. However, the concentrations of PCDFs were significantly higher than that of PCDDs. PCDFs were still the main products in the dust ash, the generation amounts of PCDFs were 4.41 to 5.67 times that of PCDDs while TEQ concentrations of PCDFs were 9.30 to 11.7 times that of PCDDs.

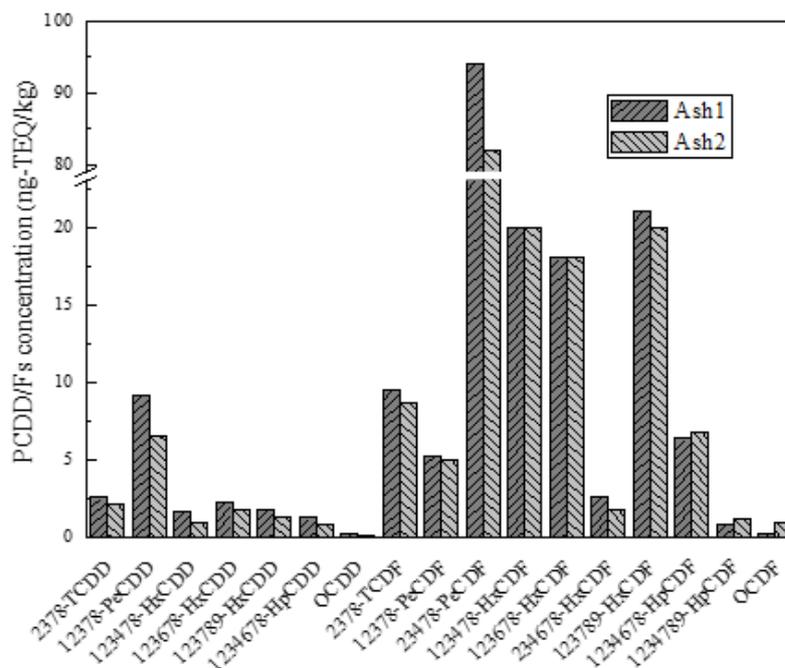
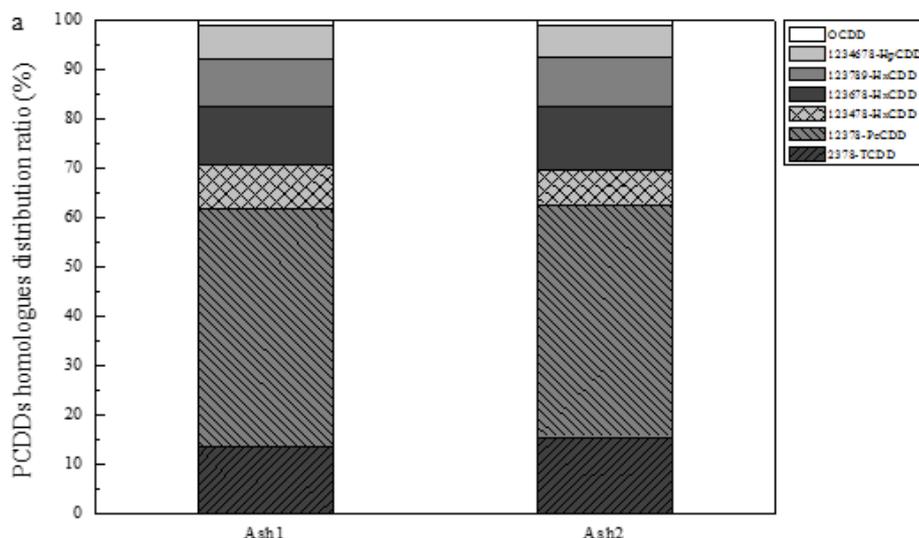


Figure 5. PCDD/Fs homologues concentration in dust ashatW1 and W2.

Figure 5 reveals the PCDD/Fshomologues concentrations in the dust ashes atW1 and W2. In both cases, the homologues distribution patterns of PCDD/Fs were almost the same. 1,2,3,7,8-PeCDD was the main chlorinated product of PCDDs with the ratios of 48.07%(W1) and 46.94%(W2), which were also consistent with the homologues distribution characteristics in the flue gas. At the same time, 2,3,4,7,8-PeCDF was the highest content in total PCDFs. More comprehensive contents were shown in Figure 6. The results showed that the different loads have no significant effect on the homologues distributions of PCDD/Fs [40-50].



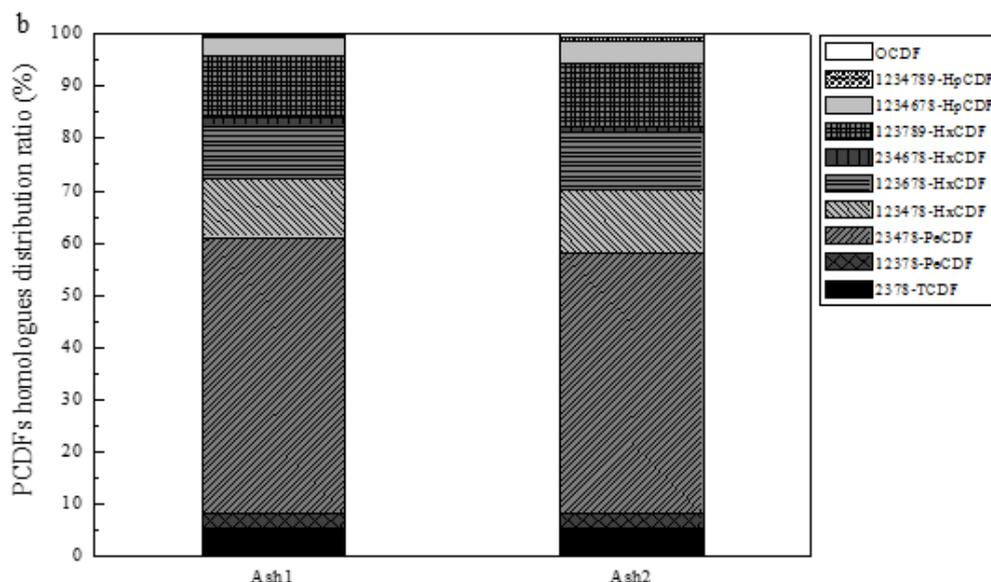


Figure 6. PCDDs(a) and PCDFs(b) homologues concentration rate in dust ash at W1 and W2.

4. Conclusions

This study focused on the removal effects and emission distribution characteristics of particulate matter, SO_2 , NO_x , fluoride and dioxins in the sintering flue gas with an oxidative desulfurization and denitrification system. The results showed:

(1) The emission concentrations of particulate matter, fluoride, NO_x , SO_2 and PCDD/Fs were reduced dramatically to 1.2 mg/m^3 , 0.07 mg/m^3 , 24 mg/m^3 , 20 mg/m^3 and $0.17 \text{ ng} \cdot \text{TEQ/m}^3$ (100% load condition), $0.11 \text{ ng} \cdot \text{TEQ/m}^3$ (60% load condition), respectively, which were all under the ultra-low emission standards in China.

(2) The oxidation purification device of sintering flue gas had remarkable removal effects of various pollutants. The removal rates of particulate matter, SO_2 , NO_x , fluoride and dioxins are 97.5%, 96.3%, 84.0% and 41.7% respectively, especially, the removal rates of PCDD/Fs were up to 95.1% (100% load) and 93.1% (60% load).

(3) The sintering flue gas purification device tended to remove high chlorinated PCDD/Fs, the removal rates were sorted as follows: Octa-PCDD/Fs > Hepta-PCDD/Fs > Hexa-PCDD/Fs > Penta-PCDD/Fs > Tetra-PCDD/Fs.

(4) The different load conditions cannot change the homologous distributions of PCDD/Fs but only impacted the emission concentrations and removal rates of PCDD/Fs in the flue gas, which indicated that the 100% load condition resulted higher PCDD/Fs concentrations as well as the removal effects comparing with 60% load condition.

However, PCDD/Fs in the desulfurization and denitrification by-products were significantly concentrated in the dust ashes with $197.21 \text{ ng} \cdot \text{TEQ/m}^3$ and $178.56 \text{ ng} \cdot \text{TEQ/m}^3$. In the subsequent disposal of the desulfurization and denitrification by-products, the secondary pollution of dioxins should be investigated. At the same time, we should pay attention to the enrichments and distributions of various pollutants in the purification device and the impact on equipment operation under long-term production. Further studies are needed to investigate the formation potential and removal technology of PCDD/Fs at China sintering plants.

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