Estimation and characterization of unintentionally produced persistent organic pollutant emission from converter steelmaking processes

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Abstract Unintentionally produced persistent organic pollutants (UP-POPs) including polychlorinated dibenzo-*p*-dioxins, and dibenzofurans (PCDD/Fs), polychlorinated biphenyls (PCBs), and polychlorinated naphthalenes (PCNs) were characterized and quantified in stack gas and fly ash from the second ventilation systems in five typical converters in five different steelmaking plants. The 2378-substituted PCDD/Fs (2378-PCDD/Fs) and dioxin-like PCB (dl-PCBs) toxic equivalents (TEQs) were 1.84–10.3 pg WHO-TEQ Nm⁻³ in the stack gas and 5.59–87.6 pg WHO-TEQ g⁻¹ in the fly ash, and the PCN TEQs were 0.06–0.56 pg TEQ Nm⁻³ in the stack gas and 0.03–0.08 pg TEQ g⁻¹ in the fly ash. The concentrations of UP-POPs in the present study were generally lower than those in other metallurgical processes, such as electric arc furnaces, iron ore sintering, and secondary metallurgical processes. Adding scrap metal might increase UP-POP emissions, indicating that raw material composition was a key influence on emissions. HxCDF, HpCDF, OCDF, HpCDD, and OCDD were the dominant PCDD/Fs in the stack gas and fly ash. TeCB and PeCB were dominant in the stack gas, but HxCB provided more to the total PCB concentrations in the fly ash. The lower chlorinated PCNs were dominant in all of the samples. The 2378-PCDD/F, dl-PCB, and PCN emission factors in stack gases from the steelmaking converter processes (per ton of steel produced) were 1.88–2.89, 0.14–0.76, and 229–759 µg t⁻¹, respectively.

Keywords UP-POPs · PCDD/Fs · PCBs · PCNs · Emission factor · Converter steelmaking

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Introduction

Polychlorinated dibenzo-*p*-dioxins and dibenzofurans (PCDD/Fs), polychlorinated biphenyls (PCBs), and polychlorinated naphthalenes (PCNs) are highly toxic, persistent in the environment, bioaccumulated through food webs, and can be transported on a global scale. PCDD/Fs, PCBs, and PCNs can be unintentionally produced in and emitted from thermal processes (such as incineration, electric arc furnaces (EAFs), iron ore sintering (SNT), and secondary metal smelting, so they are sometimes called unintentionally produced persistent organic pollutants (UP-POPs). The emission of UP-POPs from thermal industrial processes is currently of great interest and concern.

Metallurgical industries have been shown to be important sources of PCDD/F emissions to the environment, and it has been reported that they are the most important PCDD/F sources (Anderson and Fisher 2002; Buekens et al. 2001). SNT and EAFs, used in integrated steelmaking processes, are thought to be two of the main global UP-POP sources (Aries et al. 2006; Liu et al. 2012a, b; Tian et al. 2012; Yu et al. 2012). UP-POP emissions from SNT have been found to be comparable to those from coking, but lower than those from thermal wire reclamation and copper secondary metallurgical processes (Hu et al. 2013a, b; Liu et al. 2009, 2012b; Nie et al. 2012a, b).

Compared with the number of studies of emissions from EAFs and SNT, there have been few studies of PCDD/F and PCB emissions from converter processes in steelmaking. No studies of PCN emissions from converter steelmaking plants have yet been carried out. The effects of certain factors that might be related to UP-POP emissions, such as the technologies applied, the converter scales, air pollution control technologies, and raw materials, were not throughout investigated in previous studies of PCDD/F and PCB emissions during converter steelmaking (Fisher et al. 1997; Grochowalski et al.

2006; Jackson et al. 2012). Therefore, more case studies are needed so that UP-POP emissions during converter steelmaking processes can be systematically and scientifically evaluated.

About half of the total global crude steel production is performed in China, where there are more than 600 converters of different types and sizes. It is, therefore, important to study the converter steelmaking processes used in China to improve our knowledge of UP-POP emissions from these plants. In this study, UP-POP emissions from five converters in five different steelmaking plants in northern China were studied. The relationships between different UP-POP concentrations in the samples were examined to identify similarities between different chemicals. The main factors that influenced UP-POP emissions were assessed, and the UP-POP emissions from the converter steelmaking processes were characterized. The results will be useful for developing and implementing control measures for UP-POP emissions from converter steelmaking processes.

Materials and methods

Sampling procedures

Converter steelmaking processes normally have three stages: desulfurization, decarburization, and secondary metallurgical processes. Molten iron, scrap steel, iron alloys, and other materials are used as the primary raw materials. The molten iron is converted into liquid steel in the converter, and this process does not require extra energy resources, simply using the heat (>1,200 °C) already present and produced by chemical reactions involving the molten iron itself to make the steel. Typically, an average of 10–30 % of the converter charge is scrap steel, which is used as a kind of coolant.

Most of the gases and particles are generated during the blowing, charging, and tapping steps in the steelmaking process. A secondary ventilation system is installed directly above the converters, and this usually consists of a roof-mounted canopy hood designed to capture the particles.

However, not all of the particles can be captured. So there is a potential risk for workers to be exposed to particulates and chemicals.

Top- and bottom-blown converters are the most popular techniques for steelmaking. In this study, one top-blown converter (C5) and four top- and bottom-blown converters (C1–C4) from five different steelmaking plants were investigated. The sizes of the converters studied ranged from 50 to 300 t (per batch). A more modern steelmaking technique was used in converter C1, which produces a higher-quality steel, used less energy, and emitted less common pollutants. This modern technique involved the molten iron being desulfurized in a desulfurization station, and dephosphorized and desiliconized in a converter, to decrease its sulfur, phosphorus, and silicon contents, before being decarburized. The molten iron pretreatment in the other four converters only involved a desulfurization step in a desulfurization station and a dephosphorization step in a ladle or a torpedo car before decarburization.

A total of 15 stack gas and five fly ash samples were collected from the secondary ventilation systems of the five converters. Secondary ventilation systems primarily capture gases and particles produced during the charging and tapping processes. The air pollution control devices (APCDs) used in the converters studied were bag filters. The stack gas sampling points were located downstream of the APCDs. The temperature of the stack gas after bag filters were in the range of 50–70 °C. The stack gas samples were collected using an automatic isokinetic sampling system (Isostack Basic; TCR TECORA, Milan, Italy). Three stack gas samples were collected from each plant to investigate UP-POP emissions. Details of the converter steelmaking plants are shown in Table 1.

Preparation and analysis of samples

PCDD/Fs, PCBs, and PCNs were quantified using isotope dilution high-resolution gas chromatography-high-resolution mass spectrometry (MS) methods. The PCDD/Fs and PCBs were analyzed using method EN 1948 (PCDD/Fs) and EPA 1668B (PCBs). The stack gas samples were spiked with

Table 1 Basic information on the converter steelmaking plants studied

	C1	C2	C3	C4	C5
Converter type	TB ^a	TB ^a	TB ^a	TB ^a	T ^b
Converter scale (tons)	300	120	120	120	50
Percentage of scrap steel added (%)	20	16	15	12	10
Air pollution control device	Bag filters	Bag filters	Bag filters	Bag filters	Bag filters
Average stack gas flow rate (Newton cubic meter per hour)	324,279	1,175,206	994,669	543,129	199,229
Fly ash production rate (tons per year)	2580	3192	1440	732	516

^a Top- and bottom-blown converter

^b Top-blown converter

Table 2 UP-POP concentrations in stack gas and fly ash from the converter steelmaking processes

Compound	C1		C2		C3		C4		C5	
	Gas (n=3)	Ash	Gas (n=3)	Ash	Gas (n=3)	Ash	Gas (n=3)	Ash	Gas (n=3)	Ash
Σ 2378-chlorinated PCDD/Fs (picogram Newton per cubic meter or picogram per gram)	76.0–104	2.26	44.7–123	2.87	62.9–163	0.14	75.1–159	0.14	81.2–124	0.24
Σ dl-PCBs (picogram Newton per cubic meter or picogram per gram)	529–747	0.005	52.7–224	0.07	44.7–91.9	0.18	57.0–199	0.09	35.8–66.4	0.07
Σ PCNs (nanogram Newton per cubic meter or nanogram per gram)	11.2–40.8	2.94	18.0–44.3	2.00	1.49–5.64	2.71	3.38–9.29	2.52	5.43–11.1	1.80
WHO-TEQ (picogram TEQ Newton per cubic meter or picogram TEQ per gram)	5.47–10.3	87.6	1.84–4.64	57.8	6.07–10.0	5.59	3.07–5.37	8.83	3.37–4.36	13.3
TEQ of PCNs (picogram TEQ Newton per cubic meter or picogram TEQ per gram)	0.41–0.56	0.08	0.19–0.22	0.03	0.07–0.26	0.04	0.08–0.27	0.06	0.06–0.23	0.07

$^{13}\text{C}_{12}$ -labeled internal standards and then extracted with toluene for about 24 h in a Soxhlet apparatus. After eluting the extracts through multilayer silica gel columns and activated carbon columns, they were concentrated to about 20 μl . Before injection, $^{13}\text{C}_{12}$ -labeled injection standards were added so that the recoveries of the internal standards could be calculated. The fly ash samples were spiked with $^{13}\text{C}_{12}$ -labeled internal standards and pretreated with 1 mol l^{-1} HCl before being extracted. The extraction and cleanup methods were the same for the fly ash samples as for the stack gas samples. The same extraction and cleanup procedures were used for the PCNs as were used for the PCBs, for both the stack gas and fly ash samples.

PCDD/Fs, PCBs, and PCNs were analyzed in the extracts using an Agilent 6890 gas chromatograph coupled with a Waters Autospec Ultima high-resolution MS. A DB-5 MS fused-silica column (60 m $\times$ 0.25 mm i.d. $\times$ 0.25 μm) was used to separate the analytes. The MS was calibrated using perfluorokerosene, and it was operated at $\geq 10,000$ resolution with an electron impact ionization source set at an electron energy of 38 eV.

Quality control and quality assurance

The recoveries of the surrogate sampling standards in the stack gas samples were 75–105 %, and they met the analytical method requirements. The $^{13}\text{C}_{12}$ -labeled PCDD/F, PCB, and PCN internal standard recoveries were 45–119 %, 50–126 %, and 53–121 %, respectively, proving the validity of the analytical methods used. A target compound was identified if the GC retention time of the peak of interest matched the corresponding $^{13}\text{C}_{12}$ -labeled standard, the signal-to-noise ratio was greater than 3:1, and the isotopic ratio between the quantitation and confirmation ions was within ± 15 % of the theoretical value. One blank sample was included in each batch of samples analyzed, and only OCDD and OCDF concentrations were a little higher than detection

limits, but were lower than the 10 % of the concentration in the stack gas and fly ash.

The UP-POP concentrations in the stack gas were converted to “dry standard conditions” (273 K and 101.3 kPa) and the 2005 World Health Organization (WHO) authorized toxic equivalence factors (TEFs) that were used to calculate the PCDD/F and dl-PCB toxic equivalents (TEQs). The PCN TEQ was defined as the sum of the products of the concentration of each PCN congener multiplied by the potency factors (RPF) of PCN congeners relative to 2378-tetrachlorodibenzo-

Fig. 1 Relationships between the percentage of scrap steel added and the unintentionally produced persistent organic pollutant concentrations in the stack gas

p-dioxin (Noma et al. 2004). For congeners found at concentrations below the limit of detection (LOD), a value of half of the LOD was used to calculate the total concentrations and the TEQs.

Results and discussion

UP-POP concentrations emitted by the converter steelmaking processes

The PCDD/F, PCB, and PCN concentrations and their TEQs are summarized in Table 2. The 2378-substituted PCDD/F

concentrations in the stack gas were 0.045–0.16 ng Nm⁻³ (0.77–12.9 pg WHO-TEQ Nm⁻³), which were similar to the concentrations (1–13 and 17.7 pg I-TEQ Nm⁻³) found in other converter steelmaking plant stack gas (Fisher et al. 1997; Grochowalski et al. 2006). The PCDD/F concentrations in the fly ash were 0.14–2.26 ng g⁻¹ (5.52–87.6 pg WHO-TEQ g⁻¹), which were much lower than those found in EAFs (Chiu et al. 2011; Jager 1993; Sofilic et al. 2012).

The dl-PCB concentrations were 0.036–0.75 ng Nm⁻³ (0.03–2.85 pg WHO-TEQ Nm⁻³) in the stack gas and 0.005–0.18 ng g⁻¹ (0.03–1.02 pg WHO-TEQ g⁻¹) in the fly ash. These results are similar to the dl-PCB concentrations found in basic oxygen furnaces and EAFs (Grochowalski

Fig. 2 PCDD/F, dl-PCB, and PCN fingerprints for the (a) stack gas and (b) fly ash from the converter steelmaking processes

et al. 2006) in Poland, but much lower than dl-PCBs emissions found in SNT plants in the UK, which were 42–111 pg WHO-TEQ Nm^{-3} (Aries et al. 2006).

The average PCN concentrations in the stack gas and fly ash were 20.9 ng Nm^{-3} (range $3.38\text{--}44.3 \text{ ng Nm}^{-3}$) and 2.39 ng g^{-1} (range $1.80\text{--}2.94 \text{ ng g}^{-1}$), respectively. The mean PCN TEQs in the stack gas and fly ash were $0.23 \text{ pg TEQ Nm}^{-3}$ ($0.06\text{--}0.56 \text{ pg TEQ Nm}^{-3}$) and $0.06 \text{ pg TEQ g}^{-1}$ ($0.03\text{--}0.08 \text{ pg TEQ g}^{-1}$), respectively. PCN emissions from converter steelmaking processes have not been reported before. The PCN concentrations were much lower than those have been found in stack gases produced by other thermal processes, such as secondary metal smelting ($41.3\text{--}8,021 \text{ ng Nm}^{-3}$), SNT ($3\text{--}983 \text{ ng Nm}^{-3}$), thermal wire reclamation ($2,045\text{--}14,490 \text{ ng Nm}^{-3}$), and magnesium metallurgical ($2,470\text{--}93,400 \text{ ng Nm}^{-3}$) processes (Ba et al. 2010; Liu et al. 2010, 2012b; Nie et al. 2011, 2012b).

The UP-POP emissions from plant C1 were in the same range as those from plants C2–C5. This indicates that the modern steelmaking techniques used in plant C1, to produce high-quality steel, to save energy, and to reduce emissions of common pollutants, were not likely to help decrease UP-POP emissions. We also assessed other factors affecting UP-POP emissions and found that the amount of scrap metal added might affect the UP-POP concentrations in the stack gas. The preliminary results in Fig. 1 shows the increase in the UP-POP concentrations in the stack gas with increasing amount of scrap steel added, especially for 2378-PCDD/Fs and dl-PCBs. Further study with much more case plants is needed for establishing the correlations between raw materials and UP-POP emissions.

Some scrap steel may contain oil, surface oxidation, and chlorinated organic chemicals. The preliminary heating of the iron and scrap steel takes place in the presence of plastics,

Fig. 3 PCDD/F, dl-PCB and PCN homolog profiles in the (a) stack gas and (b) fly ash from the converter steelmaking processes

coatings, paint, seals, and nonferrous metals (Grochowalski et al. 2006). Therefore, the addition of scrap steel might be an important source of chlorine that may lead to the formation of UP-POPs. The pretreatment of scrap metal to remove these unwanted impurities should be thus helpful in decreasing the UP-POP emissions from converter steelmaking processes.

Characteristics of the UP-POPs emissions

The homolog profiles for the 17 2378-substituted PCDD/Fs (2378-PCDD/Fs), 12 dl-PCBs, and 18 dl-PCNs (i.e., PCN congeners with RPFs) were used to form fingerprints of the PCDD/F, dl-PCB, and PCN emissions from the converter steelmaking plants. The PCDD/F, dl-PCB, and dl-PCN concentrations were normalized to the total 2378-PCDD/F, total dl-PCB, and total PCN concentrations, respectively, to illustrate the PCDD/F, dl-PCB, and PCN distributions in the stack gas and fly ash from the converter steelmaking plants.

The 2378-PCDD/F, dl-PCB, and dl-PCN fingerprints in the stack gas and fly ash are shown in Fig. 2. Similar 2378-PCDD/F fingerprints were found in all five converter steelmaking plants. 1234678-HpCDF, 1234678-HpCDD, OCDD and OCDF were the main PCDD/F congeners in the stack gas and fly ash. CB-77, 105, and 118 were the main dl-PCB congeners in the stack gas and fly ash, CB-118 being the most abundant. Lower chlorinated PCN congeners, such as CN-1, CN-2, and CN-5/7, were the main PCNs in the stack gas. Higher chlorinated PCN congeners, such as CN-73, CN-66/

67, and CN-79, contributed a larger proportion of the total PCNs in the fly ash. It would have been, at least partly, caused by the fraction of semivolatile compounds adsorbed to particles being higher for less volatile congeners (Lohmann and Jones 1998). The 2378-PCDD/F, dl-PCB, and dl-PCN fingerprints in the converter steelmaking process were very similar to fingerprints found in other thermal processes including SNT, secondary copper smelting, and EAF processes (Hu et al. 2013a; Liu et al. 2012a, b).

The PCDD/F, dl-PCB, and PCN homolog profiles in the stack gas and fly ash from the converter steelmaking processes are shown in Fig. 3. The higher chlorinated PCDD/F homologs, such as the HxCDF, HpCDF, OCDF, HpCDD and OCDD, were the main PCDD/F homologs in all of the stack gas and fly ash. The lower chlorinated PCBs, such as the TeCB and PeCB, were the dominant PCBs in the stack gas, but the higher chlorinated PCBs (e.g., the HxCB) were found at higher proportions of the total PCB concentrations in the fly ash. The lower chlorinated PCNs (MoCN, DiCN, TiCN, and TeCN) were dominant in all 15 of the stack gas, accounting for more than 80 % of the total PCN concentrations. However, the PeCN were found at much higher concentrations in the stack gas from plant C1 than in the stack gas from the other plants. The TiCN, TeCN, and PeCN were the main homologs found in the fly ash, accounting for more than 75 % of the total PCN concentrations.

Based on the target compound concentrations, principal component analysis (PCA) was performed and the results are shown

Fig. 4 Plot of the first and second principal components of the target compound concentrations

in Fig. 4. The PCA analysis showed that the PCN concentrations were closely related to the PCB concentrations in the stack gas. Most of the PCB and PCN homolog concentrations are in the upper right part of Fig. 4, while most of the PCDDs and PCDFs are located in the upper left part of the figure.

Figure 5 shows that the sum of the tetra- to octa-chlorinated PCB concentrations generally correlated well with the sum of the tetra- to octa-chlorinated PCN concentrations ($r^2=0.74$). This shows that the PCBs might have similar formation pathways to the PCNs in the converter steelmaking processes.

Estimating UP-POPs emissions from the converter steelmaking processes

Emission factors are generally used to estimate the total emissions of a given pollutant from a given source, based on limited data. Emission factors need to be determined to estimate UP-POP emission inventories and to assist regulatory agencies in developing appropriate control strategies so that overall emissions can be caused to decrease. Emission factors and amounts emitted were calculated for each of the chemicals analyzed in this study using the equations we have presented previously (Liu et al. 2012b; Nie et al. 2011, 2012a).

The 2378-PCDD/F, dl-PCB, and PCN stack gas emission factors (per ton of steel produced) for the converter steelmaking processes were 1.88–2.89 $\mu\text{g t}^{-1}$ (5.38–50.7 ng WHO-TEQ_{PCDD/Fs} t^{-1}), 0.14–0.76 $\mu\text{g t}^{-1}$ (0.26–3.45 ng WHO-TEQ_{dl-PCBs} t^{-1}), and 229–759 $\mu\text{g t}^{-1}$ (0.32–1.54 ng TEQ t^{-1}), respectively. The PCDD/F emission factors were comparable to emission factors that have been reported previously (Grochowalski et al. 2006), but much lower than emission factors proposed by the UNEP toolkit (UNEP 2012, Stockholm Convention) for converter steelmaking with clean raw materials (0.1 $\mu\text{g TEQ t}^{-1}$). The 2378-PCDD/F, dl-PCB, and PCN fly ash emission factors were 0.11–6.70, 0.06–0.18, and 2.19–18.9 $\mu\text{g t}^{-1}$, respectively. Nearly all of the fly ash produced

in the plants studied was recycled and used as a raw material in the sintering process, so fly ash emissions were not included when we calculated the annual emissions of UP-POPs from the converter steelmaking processes. Based on the annual crude steel output in 2012 (at least 5.67×10^8 t), our preliminary estimates of the annual atmospheric emissions of PCDD/Fs, PCBs, and PCNs from converter steelmaking through second ventilation systems in China are 30.5, 1.17, and 0.55 g TEQ, respectively. The National Implementation Plan for the Stockholm Convention on Persistent Organic Pollutants (NIP) of China identified that the total PCDD/F released to the air was 5.0 kg TEQ years⁻¹, about 50 % of which was from ferrous and nonferrous metal production in 2004 (NIP of China 2007). This shows that converter steelmaking processes are probably not a key priority UP-POP source in China.

Conclusions

The emissions of PCDD/Fs, PCBs, and PCNs from five converter steelmaking plants in China were investigated. Different converter steelmaking processes were studied, and we found that a modern technique (used in plant C1) that causes lower concentrations of common pollutants to be emitted than older techniques do might not actually help to decrease UP-POP emissions. We also found that there was no significant difference in UP-POP emission from different types of converters. We found that the amount of scrap steel included in the converter steelmaking process might influence the UP-POP concentrations emitted, the UP-POP emission concentration increasing as the amount of scrap steel added increased. The amounts of UP-POPs emitted from the converter steelmaking processes were found to be much lower than the amounts emitted from iron ore sintering, electric arc furnaces, and coking processes. This suggests that the converter steelmaking processes are probably not a key priority UP-POP source.

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Fig. 5 Correlation between the sum of the tetra- to octa-chlorinated naphthalene concentrations and the sum of the tetra- to octa-chlorinated biphenyl concentrations

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