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Determination of polychlorinated dibenzo-*p*-dioxins and dibenzo-furans in solid residues from wood combustion by HRGC/HRMS

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Abstract

PCDD/PCDF were determined in solid samples from wood combustion. The samples included grate ashes, bottom ashes, furnace ashes as well as fly and cyclone ashes. The solid waste samples were classified into bottom and fly ash from native wood and bottom and fly ash from waste wood. For each of the four classes concentration distribution patterns from individual congeners, the sums of PCDD/PCDF and the international toxicity equivalents (I-TEQ) values are given. The I-TEQ levels of fly ash from waste wood burning can be approximately up to two thousand times higher than the values from fly ashes of natural wood. The I-TEQ levels in bottom ashes from waste wood combustion systems are as low as the corresponding ashes from the combustion of native wood. Grate ash samples from waste wood combustion systems with low carbon burnout show high levels of PCDD/PCDF. © 2000 Elsevier Science Ltd. All rights reserved.

Keywords: Polychlorinated dibenzo-*p*-dioxins (PCDD); Polychlorinated dibenzo-furans (PCDF); Solid waste; Ash; Wood combustion; Electrofilter; Fly ash; Incineration

ABR: $\sum$ sum of all quantifiable congeners. The limit of detection ($\leqslant$) is assumed to be 3 times the peak-to-peak noise of the mass trace 10 times the peak width near the expected peak; I-TEQ international toxicity equivalent value (Safe, 1992); u_c combined measurement uncertainty (Anon, 1993b); MWI municipal waste incinerator

1. Introduction

This investigation represents a part of a project directed by the Swiss authorities Bundesamt für Umwelt, Wald und Landschaft (BUWAL), Bern, with the aim of conducting an actual survey concerning the quantities of solid wastes and the concentrations of polychlorinated dibenzodioxins and dibenzofurans (PCDD and PCDF) contained therein. The solid wastes investigated were by-products originating from various processes of the Swiss technological environment. The solid wastes were slags, ashes, filter dusts and filter cakes from the different processing steps of municipal and hazardous waste in-

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cinerators and wood combustion facilities, solid wastes from metal industry, slags from battery recycling, solid waste from the paper industry, ash from cremation and tissue incineration from hospitals, ash from open fires and sludge from road dust samplers. The data were acquired from more than 100 samples, including approximately 30 samples from many types of wood combustion, which provide the basis for the future calculation of the corresponding PCDD/PCDF flow in these wastes. The samples investigated and presented in this paper originated mainly from the different furnace parts of burner systems for wood combustion in which natural, residual or waste wood was incinerated. The samples were electrofilter ashes, fly ashes, bottom ashes and grate ashes.

The main primordial sources for the unintended formation of PCDD/PCDF are thermal and chemical industrial processes (Fiedler and Van den Berg, 1996; Ballschmitter and Bacher, 1996; Oehme, 1998). Wood burning is always accompanied by the unwanted production of PCDD/PCDF. They can be found in the solid ashes and in the waste gas stream. The amount of PCDD/PCDF that will be formed is dependent on the quality of wood burned.

In Switzerland, the annual wood burning per capita amounts to about 320 kg (Neue Zürcher Zeitung, 1996). 380 000 t of waste wood are produced annually. A more intensive use of wood to produce energy is a component of the present Swiss energy policy. Potentially, about 6% of the heat energy production of Switzerland could be supplied by wood combustion (VHe, 1991; Noger et al., 1996). In 1993 the combustion of 2.1×10^6 m^3 of wood resulted in an estimated amount of 24 000 t of ash. An approximate mass fraction of 1% remains as ash from wood burning. This compares to a mass fraction of 5–6% of ash remaining from the burning of *Miscanthus*, (family Graminae). This plant is discussed as a possible alternative renewable energy source.

The solid residues from wood combustion can either be used in industry as supplementary material such as concrete production or filler material, in agriculture as fertilisers or must be disposed of in waste dumps. Research work is in progress to get more information about the type of chemicals and their concentrations which are contained in such residues (Noger et al., 1996). Dioxins and some heavy metals are present in nearly all types of ashes from wood burning and have most relevance to the decisions on how to efficiently optimise the safe disposal of the residues (Oehme and Müller, 1995; Noger et al., 1996; BUWAL, 1993; Hasler and Nussbaumer, 1994, Pohlandt and Marutzky, 1994).

The combustion of wood in Switzerland is mainly accomplished by using both log wood boilers and automatic wood chip furnaces. Manually fed log wood boilers are more widely used than automatic units. The primary fuel for log wood boilers is uncontaminated native wood. Burning of waste wood is not permitted. Table 1 summarises the number and types of actual Swiss wood burning systems. Mainly small-scale combustion systems are operated.

The combustion of waste wood requires a more advanced ash handling system and furnace technology (Nussbaumer et al., 1998). Furthermore, it is prohibited to burn waste wood in furnaces with a capacity below 350 kW. Waste wood furnaces are primarily used as process heat units. Fuel gas cleaning systems are required such as an electrostatic precipitator or fabric filters to limit the heavy metal and particulate emissions. Table 2 shows the estimated amount and classification of wood burned in different types of combustion systems. The quantity of wood used in Switzerland for energy production annually amounts to approximately 2.1 million m^3 of wood resulting in 500 GWh of energy.

In Switzerland more than 60% of the energy from wood is produced in small scale log wood boilers with untreated wood as a fuel. The overall combustion of wood generates approximately 24 000 t of ash annually (Table 3).

The investigation of three classes of wood (LRV, 1998) and two types of solid residues were the main subject of this work: *waste wood* (painted, glued and waste wood from construction and demolition of buildings and furniture, excluding PVC coated waste and impregnated wood; *residual wood* products (all types of wood products which have been processed such as chipboard and wood dust from machining); *native wood*

Table 1
Number and size of wood combustion systems used in Switzerland (1993) (Nussbaumer et al., 1998)

Combustion system	Heat output (kW)	Number of facilities in Switzerland
Log wood boiler	<30	490 000
	>30	115 000
Automatic wood chip furnace	<70	1400
	>70	2400
Automatic urban waste wood chip furnace	>350	15

Table 2
The estimated amount of wood used for energy production in Switzerland (1993; 10^6 m^3 of wood equals to 2500 GWh of energy) (Nussbaumer et al., 1995)

Combustion system	Native wood, residual wood (m^3)	Waste wood (m^3)	Total wood (m^3)
Log wood boilers	1 310 000	Combustion not allowed	1 310 000
Automatic wood chip furnaces	647 000	110 000	780 000
Total	1 980 000	110 000	2 090 000

Table 3
Wood ash generated annually in Switzerland (1993) (Noger et al., 1996)

Combustion system	Heat power output (kW)	Wood ash generated (t/a)
Log wood boiler	<30	6100
	>30	7200
Automatic wood chip furnace	<70	400
	>70	6500
Automatic urban waste wood chip furnace	>350	4000
Total	–	24 000

(natural wood without any treatment, including saw dust, shavings and bark).

The classification differs from other countries (e.g., Germany). The solid residues were classified as *fly ash* and *bottom ash.* Fly ash includes all solid material being separated from the flue gas stream by cyclones, electrostatic precipitators (ESP), fabric filters and ceramic filters. Bottom ash contains all solid residues (commonly known as wood ash) which are collected after passing through a moving grate or remaining in the furnace compartment depending on the construction of the incinerator. The bottom ash was sometimes partially transformed into slag. From some incinerators only a mixture of cyclone ash and ash from grate separation was obtainable for analysis. Such sample materials were classified as fly ash.

2. Experimental

Representative sampling was performed according to DIN and ISO standards (Sommer, 1979). In large facilities the sample object sometimes could not be accessed as a whole. In those cases subsamples were collected periodically, mixed and divided later. Finally, about 1 kg of sample was collected, ground in the laboratory and stored in the darkness under argon at −20°C.

The grinding equipment consisted of a rotation type mill. It had a swinging cylinder and a flat torus in a flat cylindrical box all part made of tungsten carbide closed with a rubber sealed tungsten carbide cover (Fritsch, Pulverisette, Germany). The mean diameter of the sample particles after grinding was 0.05–0.1 mm. Grinding quartz sand followed by water and acetone rinsing was used to clean the grinding equipment after each sample. Subsamples of 10–50 g, depending on the amount of PCDD/PCDF presumed to exist, were used for the analysis after a homogenising step by turning and shaking the glass bottles.

Soxhlet extraction, clean-up and detection were performed according to a method described in reference (Doležal et al., 1995). Soxhlet extraction instead of supercritical fluid extraction (SFE) using carbon dioxide/acetone was chosen because the thimble volume was too small for certain samples. Furthermore, no extraction efficiency study could be performed for all the different type of samples. A short description of the procedure is given:

The air-dried ash was treated with diluted hydrochloric or sulphuric acid, the residue was filtered, washed free of acid and dried again. The aqueous phase was extracted using first methylene chloride and then re-extracted with cyclohexane for the collection of the remaining methylene chloride. The air-dried solid phase was then transferred to a pre-extracted thimble, the diluted multi spike solution was distributed over the solid, covered by dry sodium sulphate and Soxhlet extracted for 24 h using toluene. The organic phases were combined and the volume reduced to about 1 ml by evaporation of the solvents at a reduced pressure. For the multistage clean-up, a semi-automatic low pressure chromatography system (Fluid management system, FMS, Watertown MA, USA) with a combined silica column (acid/base), alumina column (ICN supe B) and carbon celite column (PX 21) was used. For each sample the whole set of columns was exchanged completely in order to avoid any cross contamination. Toluene was used to backflush the PCDD/PCDF from the carbon column. After high level samples, the clean-up system was additionally rinsed using different solvents. No significant memory signals from the tubing could be detected. No sulphur removal step was necessary for any of the investigated sample extracts.

GC-separation was achieved with a J&W DB-Dioxin column (length 60 m, inner diameter 0.25 mm, film thickness 0.25 μm). Helium carrier gas with an inlet pressure of 6.9×10^4 Pa was used. Gas chromatographic separation was carried out on a Varian 3400 GC after automatic splitless injection (splitless period 40 s) of 2–3 μl of sample at 260°C injector temperature. The GC was programmed starting at 140°C with a hold-time of 1 min. The temperature was then increased at a rate of 11.43°C/min up to 220°C following by the second temperature rate of 2°C/min up to 260°C. The end temperature of 260°C was hold 260°C for 62 min. Each chromatographic run took 80 min. The Finnigan MAT 95 HRGC/HRMS mass spectrometric resolution was set to 10 000 (10% valley definition).

Electron impact ionisation EI^+ at 70 eV was used. The two most abundant ions of the molecular isotope cluster were monitored with multiple ion detection (MID) at 0.8 s cycle time. In the first three mass windows (18 masses per window monitored) the dwell time was 31 ms for each mass and 75 ms in the forth mass window (10 masses monitored).

All the solvents used were from a single batch (Merck quality: Supra Solv). They were used without further pre-treatment.

3. Results and discussion

The combined measurement uncertainty for individual congeners concentrations is estimated to be 20–30% based on samples used in a BCR certification exercise and a comparison of three selected individual samples. Comparison measurements of these three samples were performed in the University of Umeå, Sweden. The results (cf. Table 4) agreed reasonably.

The recovery values for all the individual 2,3,7,8-chlorine substituted PCDD/PCDF ranged from 60% to 100% with a median value of 90%. The whole method was tested in several certification exercises conducted by BCR for the analysis of PCDD/PCDF congeners in different candidate certified reference materials. The linearity of the mass spectrometric response was at least four orders of magnitude and ranged from 100 ng to 10 pg of 2,3,7,8-chlorine substituted congener per injection. The linearity of the mass spectrometric response was determined by plotting the relative response factors calculated from the sum of the peak areas of two mass traces from the native and the sum of two peak areas of mass traces from the labelled compounds versus the mass injected for each 2,3,7,8-chlorine substituted congener. A series of five different standards was used for this purpose. The native standards were supplied from BCR (Bureau Communautarie de Référence, Geel, Belgium) and the $^{13}C_{12}$-labelled internal isotope standards originated from CIL (Cambridge Isotope Laboratories, MA, USA). The reference material used to check the analytical method was CRM 429 (BCR fly ash extract). The concentration values for most of the congeners measured in the reference material CRM 429 agreed within the ranges of the certified values.

3.1. Classes of different wood ash samples

A total of 37 samples were quantified for PCDD/PCDF. Table 5 shows I-TEQ values measured.

The bar in Figs. 1–5 illustrate the concentration distributions and indicate the minimum, 10% percentile, median, 90% percentile and the maximum value for each variable (cf. Fig. 1). Mostly the highest contribution to the I-TEQ value derives from the 2,3,4,7,8-PeCDF. OCDD, OCDF and 1,2,3,4,6,7,8-HpCDD are dominating in the distribution of the 2,3,7,8-chlorine substituted congeners.

A median value of 2–3% is found for 2,3,7,8-chlorine substituted TCDD concentration compared to OCDD (cf. Fig. 2). Fig. 3 demonstrates that the highest contribution to the I-TEQ value stems from the 2,3,4,7,8-PeCDF. Highest concentrations are found for OCDD, 1,2,3,4,6,7,8-HpCDD and 1,2,3,7,8,9-HpCDF. Fig. 4 shows that again the highest contribution to the I-TEQ value originates most often from the 2,3,4,7,8-PeCDF.

Table 4
Measurement comparison from the two laboratories, EMPA and University of Umeå, Sweden

Residue type	I-TEQ values $\pm u_c$ (ng/kg)	
	EMPA	University of Umeå
Electrofilter ash from a modern MWI	1600 ± 400	810
Cyclone filter ash from residual wood	2000 ± 500	2500
Grate ash from deciduous trees	7.1 ± 1.8	6.3

Table 5
Levels of I-TEQ values (ng/kg) in fly and bottom ash from the different classes of wood; *n* is the number of samples analysed in that class; median values instead of mean values are given

Residue type	Waste wood[a]			Residual wood[b]			Native wood[c]		
	Min	Max	Median (*n*)	Min	Max	Median (*n*)	Min	Max	Median (*n*)
Fly ash	730	21 000	2800 (10)	18	6300	2000 (3)	1.5	4.0	2.6 (6)
Bottom	4.2	11	6.1 (4)	–	–	– (0)	0.60	8.6	4.9 (9)

[a] For the distributions (cf. Figs. 1–5), the three residual waste wood fly ash samples of this table were included in the waste wood fly ash class as no distinct difference in I-TEQ values between ashes from waste wood and residual wood could be seen.

[b] No Bottom ash samples were available.

[c] The native wood bottom ash sample class was extended by 5 samples (cf. Fig. 2). One ash from a forest fire (I-TEQ = 9.9 ng/kg), two charcoal samples from charcoal works (I-TEQ = 4.9 and 8.9 ng/kg) and two samples from an open forest fire with low and high carbon burnout (I-TEQ = 31 and 32 ng/kg).

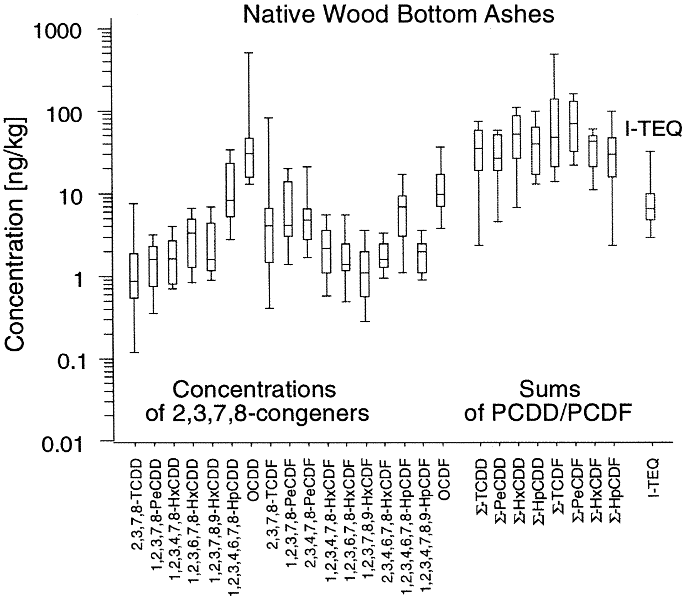

Fig. 1. Native wood bottom ash samples. Distributions (strip box plots) for all the 2,3,7,8-chlorine substituted congeners concentrations, the sums for the each congener group and for the I-TEQ values.

The most prominent individual congeners are OCDD, 1,2,3,4,6,7,8-HpCDD, 1,2,3,7,8-PeCDF, 2,3,4,7,8-PeCDF and 1,2,3,4,6,7,8-HpCDF.

The distribution of I-TEQ-levels of bottom ash from natural wood is comparable to that of the fly ash from natural wood (Fig. 5), whereas the congener concentration distribution patterns of the two classes, are not similar. In Fig. 5 residual wood ash samples are included in the waste wood fly ash class. Ash from waste wood samples is heavily loaded compared to other types of wood ash samples. For the 2,3,7,8-chlorine substituted congener distributions see Figs. 1–4. Interestingly Fig. 5 shows that native wood fly ash samples have slightly reduced compared to the corresponding bottom ashes. Generally the concentrations increase with the degree of chlorination for the dibenzo-*p*-dioxins, whereas the concentrations of the 2,3,7,8-PCDFs do not show this increase but have elevated levels for the lower and higher chlorinated congeners. It is evident that the I-TEQ values from fly ash and bottom ash originating from native wood are considerably less different than the values for waste wood where they differ by a factor of more than one thousand. Native wood fly ash comprises either mixed grate and cyclone ash or mixed furnace and cyclone ash or pure cyclone ash. Waste wood fly ash comprises either ashes from fabric filters, electrostatic precipitators, or cyclone separators.

Some typical patterns of the 2,3,7,8-chlorine substituted PCDD/PCDF in fly ash samples from waste wood and bottom ash, as well as from the burning of natural wood have been presented earlier by our group

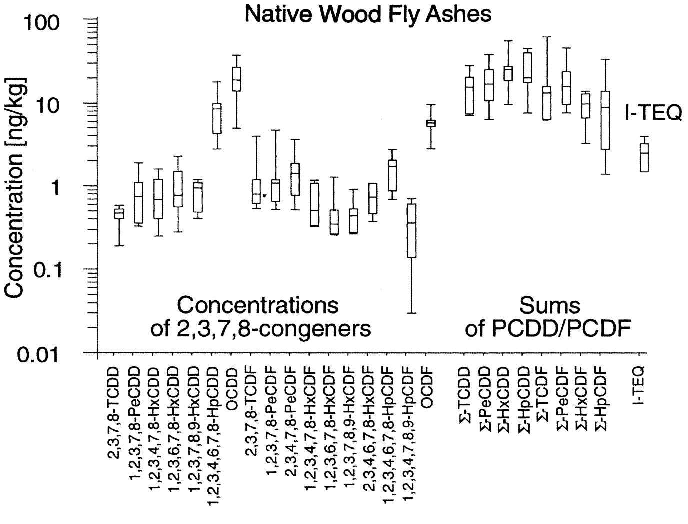

Fig. 2. Native wood fly ash samples.

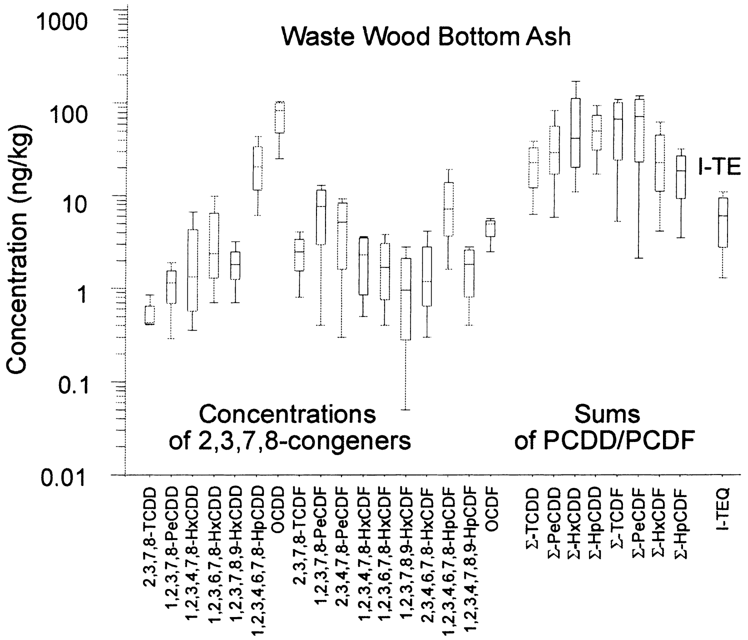

Fig. 3. Waste wood bottom ash samples.

(Wunderli et al., 1996). The distribution of I-TEQ-levels of bottom ash from natural wood is comparable to that of the fly ash.

A single grate ash sample (cf. Fig. 6(a)) from waste wood with low carbon burn-out showed much higher PCDD/PCDF levels. This grate ash contained a high content of unburned carbonised pieces of wood that means a low carbon burnout. The hot ash fell through a grate into a water-containing tank from where the ash cooled down instantly. The ash sediment, the so-called wet ash from this tank was removed continuously by a conveyor belt. An I-TEQ value of 17 000 ng/kg was determined in the fly ash (cf. Fig. 6(b)) obtained from the fabric filter of the same burner. Both patterns of the 2,3,7,8-chlorine substituted congeners from the fly and grate ash look similar, but the PCDD/PCDF content of the fly ash is approximately four to five times higher. In this specific case the PCDD/PCDF content of the grate ash from waste wood does not differ from fly ash samples originating

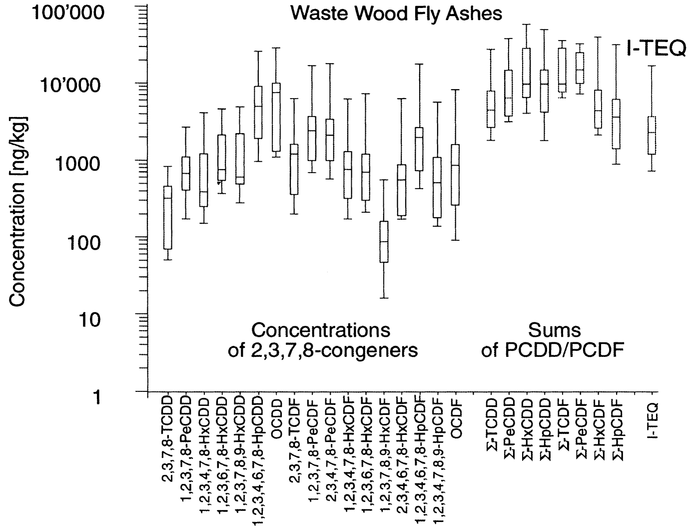

Fig. 4. Waste wood fly ash samples.

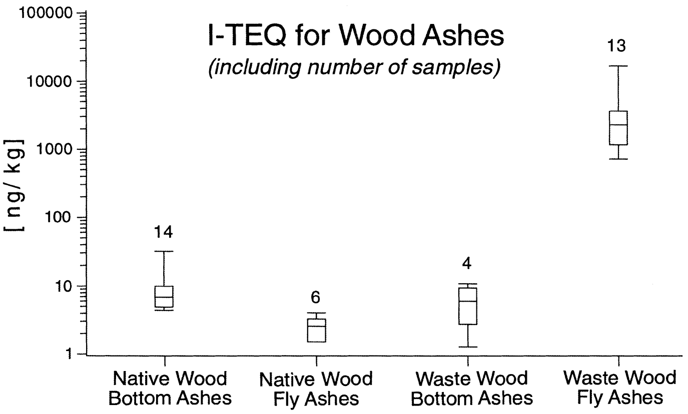

Fig. 5. Distribution of the I-TEQ values in the ashes from the four classes of wood combustion.

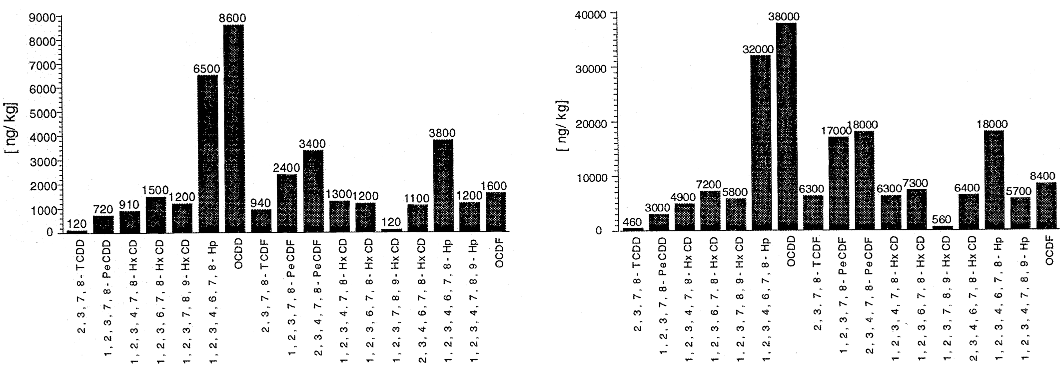

Fig. 6. (a) Waste wood grate ash (I-TEQ = 3300 ng/kg) from a 0.9 MW grate burner (same burner as in (b)) low carbon burn-out. (b) Waste wood fly ash (I-TEQ = 17 000 ng/kg) from a 0.9 MW grate burner equipped with fabric filter.

from other waste wood incineration facilities. The wet ash contained unburned carbon piece. Activated carbon or household cokes are very efficient adsorption materials for PCDD/PCDF (Dubinin, 1965, Hasler and Nussbaumer, 1995). The smallest carbon particles originating from the solid carbonised waste wood containing the PCDD/PCDF are transported by upstreaming hot air and become part of the fly ash.

The degree of carbon burnout of the waste influences the PCDD/PCDF concentration in the ashes. When waste wood is combusted a high carbon burnout yields ashes with low PCDD/PCDF contents, whereas a low carbon burnout produces ashes with high PCDD/PCDF contents. An additional reason for increased levels of PCDD/PCDF is the possible content of pentachlorophenol (PCP) in the waste wood. PCP is a well-known precursor of PCDD/PCDF or which contains substantial traces of PCDD/PCDF from the manufacturing process. PCDD/PCDF will be formed when PCP is subjected to a high temperature treatment. These factors may have caused the unusual high I-TEQ values in the grate ash.

4. Conclusions

The I-TEQ-values of all the analysed solid residues from the incineration of native wood are below 10 ng/kg. For such ashes disposal problems are minor. The I-TEQ-values determined in the ashes from combustion of residual wood are not different from those of waste wood. The production of PCDD/PCDF from solid waste wood material may be very significant when combustion is incomplete. In this case, the grate ash can be heavily loaded with PCDD/PCDF. Generally, considerably lower values, usually 4–10 ng/kg, are found in urban waste wood bottom ashes. A high carbon burnout is highly recommendable in order to lower the PCDD/PCDF load in the grate ash and to minimise the amount of ash. The produced PCDD/PCDF adhere to the activity carbon matrix which is formed in situ. When such carbon particles are left unburned, increased levels of PCDD/PCDF are found in the residue.

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