

## **RAPID DRYING OF SOIL AND SEDIMENT USING A HOME MICROWAVE OVEN FOR ANALYSIS OF PCDDs, PCDFs AND PCBs**

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### **Introduction**

For determination of polychlorinated dibenzo-p-dioxin (PCDD), polychlorinated dibenzofuran (PCDF), and polychlorinated biphenyl (PCB) concentrations in sediment and soil, complex analytical steps are required; typically, air-drying, Soxhlet extraction, purification using a multilayer column, and instrumental analysis. Moreover, soil samples including fly ash sometimes require treatment using diluted hydrochloric acid (HCl) prior to air drying. Since the analytical process takes a long time, much research has been conducted in an attempt to shorten it by substituting the conventional Soxhlet extraction with accelerated/pressurized extraction<sup>1,2</sup>, microwave-assisted extraction<sup>3,4</sup>, or super critical fluid extraction<sup>5</sup>. Although these methods may successively shorten the extraction time, the period required for the conventional Soxhlet extraction is over 16 h, which is much shorter than that necessary for air drying.

Drying sediment and soil is necessary to maintain the extraction efficiency of PCDDs, PCDFs, and PCBs at approximately 100%. Although the use of acetone as a constituent of the extraction solvent and addition of sodium sulfate anhydride to wet sediment and soil samples allows extraction without drying<sup>6</sup>, the possibility remains that water interferes with extraction itself and/or further analytical steps. As a rapid drying method, lyophilization has been used<sup>6</sup>; however, it takes one day or more for completion. In this study, a microwave oven (MO) was used as a simple and quick drying tool for sediment and soil samples for PCDD, PCDF, and PCB analyses.

### **Materials and Methods**

A home MO (EM-M201, Sanyo electric, Japan) was used as a cheap drying device, employing the defrosting (160 W) or cooking (500 W) mode. Andosol was used for preliminary experiments to determine which mode is appropriate for drying and to estimate drying time, whereas contaminated soil and sediment samples were then treated to confirm the feasibility of the MO in drying soil and sediment as a pretreatment process for PCDD, PCDF, and PCB analyses. The contaminated soil included stack dust from municipal waste incineration, while the sediment was contaminated with PCBs. Because of the existence of stack dust, the soil sample was treated with HCl prior to MO drying. As for the andosol, water was added to adjust its water content.

Since a radiation intensity of 500 W caused the andosol sample to increase in temperature to over 100°C, the defrosting mode (160 W) was used for drying (data not shown). The andosol samples were prepared by adding

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water to dried andosol to achieve a water content of 33, 50, and 66%; no water was added to the contaminated soil and sediment samples. A portion of the wet sample (1–10 g) was flatly placed on glass fiber (GB140, 9 cm $\phi$ , Advantec, Japan) laid on the bottom of a 9 cm $\phi$  Petri dish, which was then placed in the MO. During drying treatment, the temperature of the samples was monitored using infrared thermography (TVS-8000 MkII, Nippon Avionics, and Japan). Finally, the weight of the sample and its PCDD, PCDF, and PCB concentrations were measured. The PCDD and PCDF concentrations of the contaminated soil and sediment samples were measured according to the methods designed for the measurement of dioxins<sup>7</sup>. Since PCBs vaporize more easily than PCDDs and PCDFs, the sediment sample was also analyzed for PCBs. As a control, air-dried soil and sediment samples were also analyzed for these compounds.

### Results and Discussion

#### Temperature and water content of andosol as estimated by MO treatment

Figure 1 shows the amount of water with time in the andosol samples, where the time required for removal of water varies depending on the initial water content. The results of infrared thermography showed that the temperature of the andosol samples did not exceed 41°C (data not shown) during the period of drying treatment. Since the temperature level was comparable to atmospheric temperature, the results indicate the possible speed of drying samples for PCDD, PCDF, and PCB analyses using a MO.

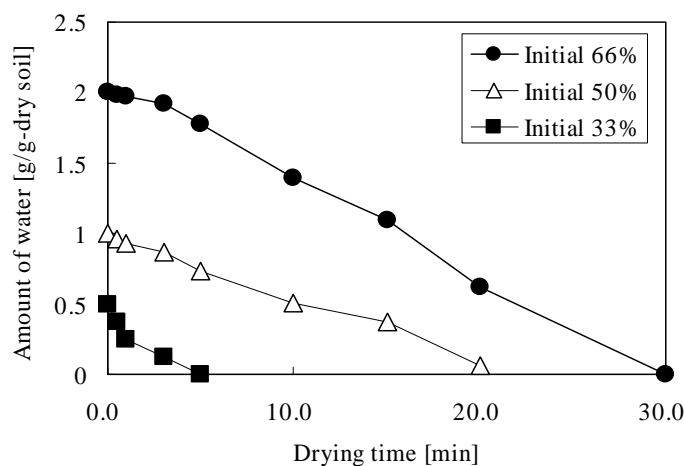


Figure 1 Change in the water content of the andosol samples.

#### Hydrochloric acid-treated soil

Since the HCl-treated soil sample was estimated to contain about 80% water, treatment time using the MO was set at 40 min on the basis of the results presented in Fig. 1. The temperature of the sample did not exceed 40°C throughout the treatment period. After MO drying, about 1 g of dried soil sample remained and was used for PCDD and PCDF analysis.

Table 1 shows the sum of the PCDD and PCDF concentrations and their TEQ concentrations in both the MO- and air-dried samples. The patterns of PCDD and PCDF homologues and isomers with chlorine atoms at

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2,3,7,8 positions are respectively illustrated in Figures 2a and 2b. Although it was suspected that MO drying might enhance volatilization of these compounds, the PCDD and PCDF concentrations of the MO-dried real soil sample were no lower than that of the air-dried sample. Furthermore, the homologues and isomers patterns of these samples were identical, thereby ruling out this possibility and confirming the feasibility of using a home MO to dry samples for PCDD and PCDF analyses.

Table 1 Comparison of the PCDD and PCDF concentrations of the air- and MO-dried soil samples.

	Air-dried	MO-dried
PCDDs	106,000	143,000*
PCDFs	29,000	34,000*
PCDDs/DFs	135,000	177,000*
TEQ	1,500	1,900*

\*Values are the average of n=2.

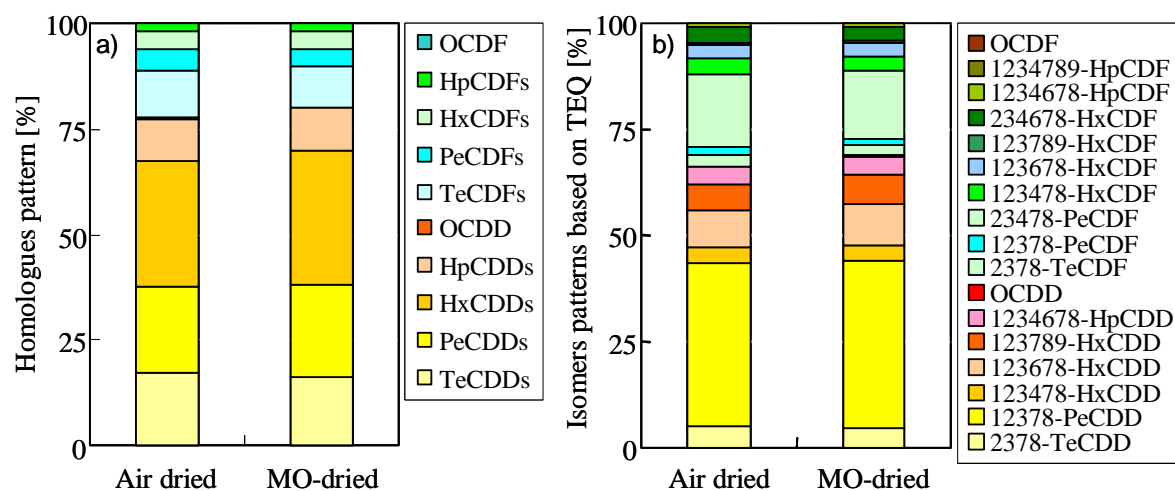


Figure 2 Comparison of the patterns of a) PCDD and PCDF homologues and b) isomers with chlorine atoms at 2,3,7,8 - positions in the air-dried and MO-dried real soil samples.

### Sediment

Although the sediment was mainly contaminated with PCBs, PCDDs and PCDFs were firstly measured to confirm the feasibility of the method. Because the PCDD and PCDF concentrations were expected to be low, samples of about 10 g (water content of 60%) were treated in the MO for 15 min. The concentrations of PCDDs and PCDFs in the sediment samples are shown in Table 2, where the PCDD and PCDF concentrations of the MO-dried sediment were no lower than those of the air-dried sample, as in Table 1. Furthermore, the PCBs demonstrated similar behaviors; the total PCB concentration in the MO-dried sediment was 1.2 times higher than that of the air-dried sample, and the homologues pattern also showed good agreement as shown in Figure 3.

In conclusion, the presented results confirm the feasibility of using a home MO to dry soil and sediment samples for PCDD, PCDF, and PCB analyses.

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Table 2 Comparison of the PCDD and PCDF concentrations of the air- and MO-dried sediment samples.

	Air-dried	MO-dried
PCDDs	4,400	4,500*
PCDFs	1700	3,000*
PCDDs/DFs	6,100	7,500*
TEQ	45	78*

\*Values are the average of n=2.

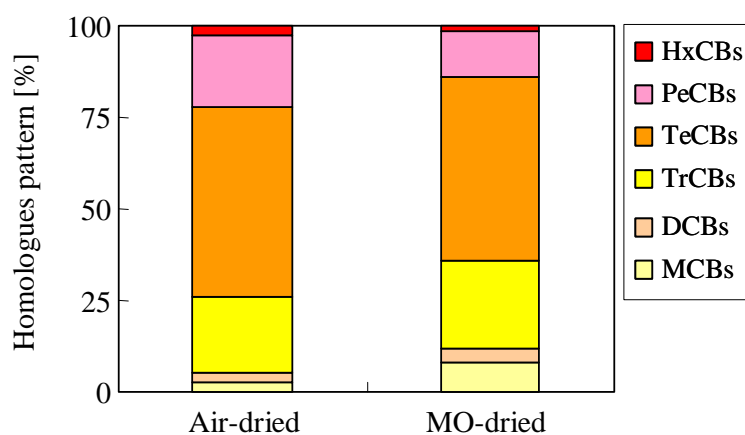


Figure 3 Comparison of the PCB homologues patterns of the air- and MO-dried real sediment samples.

### Acknowledgements

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