

# Application of microwave-assisted extraction to the analysis of PCBs and CBzs in fly ash from municipal solid waste incinerators

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## Abstract

Polychlorinated biphenyls (PCBs) and chlorobenzenes (CBzs) are two classes of dioxin precursors formed in municipal solid waste incinerators (MSWIs) producing negative health effects similar to those of dioxins. Reducing the analytical time required for determining the concentrations of these compounds in MSWIs is important for quickly evaluating their importance and associated health risks. In the present study, microwave-assisted extraction (MAE) is compared with traditional Soxhlet extraction (SE) to determine the extraction efficiencies attained for PCB and CBz analysis. The efficiencies of MAE are compared with those of SE under various experimental conditions, using fly ash spiked with standards. Water is used as a safe and environmentally friendly solvent in MAE for PCB and CBz analyses and MAE has high extraction efficiency for spiked fly ash compared with that of SE. Furthermore, the extraction time and organic solvent consumption are reduced with MAE compared with SE. The optimum conditions for MAE established in this study are using a 30-ml volume of toluene/acetone (1/1) or a 15-ml volume of toluene, samples with less than 60% water content (WC), and an irradiation time of 15 min.  
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## 1. Introduction

Mass production and increased standards of living has resulted in rising amount of waste. In many developed countries, the incineration of municipal solid waste (MSW) is considered a key component in an integrated waste management strategy. Incineration destroys pathogens, toxics and organic matter at large, and typically results in a 90% reduction in volume and a more than 60% reduction in weight of the waste being combusted [1]. In addition, the recovery of energy under the form of heat is another positive feature of the combustion process. Today, incineration is the predominant waste treatment method in Japan. The amount of non-industrial waste generated since 1990 has amounted to 51 million tonnes annually. It is assumed that approximately 41 million tonnes, or 78.1%, of this waste is incinerated each year [2].

Municipal solid waste incinerators (MSWIs) produce gaseous by-products including carbon dioxide, hydrogen chloride, sulfur dioxide, and nitrogen oxides. The emission of dioxins, first discovered by Olie et al. [3], were approximately 690–756 g TEQ per annum in Japan in 2003. Approximately 50%, or 277 g TEQ of dioxin emissions has been attributed to MSWI output [4]. The concentrations of polychlorinated biphenyls (PCBs) and chlorobenzenes (CBzs), two classes of dioxin precursors [5,6], as well as those of co-planar PCBs, a type of dioxin, have been shown to be strongly correlated with dioxin levels [7].

The extraction step in general has been the least developed aspect of most analytical procedures, including procedures for the analysis of PCBs and CBzs. Although the Soxhlet extraction (SE) method for solid samples, developed in 1879 [8], is still employed in many laboratories, several novel extraction techniques, such as supercritical fluid extraction (SFE) [9–11], microwave-assisted extraction (MAE) [12–14], and accelerated-solvent extraction (ASE) [15], have been used during the past decade to extract PCBs and other persistent organic pollutants (POPs) or PAHs from various matrixes. These newer meth-

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ods are less time-consuming and use much smaller amounts of organic solvents. The efficiency of MAE has been reported to be higher than that of SFE [14]. Furthermore, other drawbacks to SFE, as well as to ASE, have been described, such as the different extraction efficiencies by SFE of PAHs from spiked and natural samples [16] and the high cost of both SFE and ASE [17]. In the present study, MAE was used to analyze PCBs and CBzs present in MSWI fly ash.

The principle of heating with microwave energy is based on the direct effect of microwaves on molecules by ionic conduction and dipole rotation. Polar molecules and ionic solutions strongly absorb microwave energy because they have a permanent dipole moment that is affected by microwaves. Microwave energy absorption in a solution can occur by any of three mechanisms. Firstly, the sample can be immersed in a single solvent or a mixture of solvents that strongly absorbs microwave energy. Secondly, the sample can be extracted in a combined solvent containing solvents with both high and low dielectric losses mixed in various proportions. Thirdly, a sample with a high dielectric loss can be extracted with a microwave transparent solvent [18]. Extraction and partitioning of solutes can occur by any combination of these three MAE modes. Therefore, in order to enhance the absorption of microwave energy, solvents with a high dielectric constant, such as water, methanol, and acetone, are preferred.

In this study, the optimum solvent mixture, solvent volume, and irradiation time were established under different experimental conditions. Furthermore, the relative standard deviation (R.S.D.) values were used to evaluate repeatability. Finally, MAE efficiencies were compared with those of SE. The other objective of the study was to use water as a polar solvent in MAE. Because solvents with a high dielectric constant, such as water, are required to absorb the microwave energy and heat the solvent in MAE, the residual water after acid pre-treatment could be used in MAE. Therefore, the air-drying time, normally 3 days could be shortened with MAE following the acid pre-treatment required for digesting the matrix.

## 2. Experimental

### 2.1. Standards and internal standards

Seventeen isomers of PCB and nine isomers of CBz were used as native standards.  $^{13}\text{C}$ -PCBs (PCB-28, PCB-52, PCB-101, PCB-118, and PCB-138) and  $^{13}\text{C}$ -CBzs (1,4-DiCBz, 1,2,3-TriCBz, 1,2,3,4-TetraCBz, PentaCBz, and HexaCBz) were used as internal standards (ISs). The information of all of the standards and ISs were shown in Appendix A.

### 2.2. Samples

#### 2.2.1. Spiked fly ash

Fly ash-E was collected from an electrostatic precipitator (ESP) in a continuous stoker-type MSWI-E in Japan. The total organic carbon (TOC) of fly ash was measured by TOC analyzers of TOC-VCSH (Shimadzu, Japan) and a Solid Sample Combustion Unit SSM-5000A (Shimadzu, Japan). The TOC of

the fly ash-E was a relatively high 4.61%. The elemental distribution in the fly ash-E was measured by XRF-1700 (Shimadzu, Japan). The amounts of the maximum elements O, C, and Ca were about 35.7, 17.1, and 10.0%, respectively. The amounts of other elements were shown in Appendix B.

To eliminate metals and inorganic compounds from the fly ash-E, it was pre-treated with 2 M hydrochloric acid (HCl) for 2 h. After air-drying for 3 days, SE was conducted three times to eliminate POPs in the fly ash-E. The PCB concentrations in the fly ash-E were reduced to 0 ng/g after the second SE. The CBz concentrations in the cleaned fly ash-E following the third SE were reduced to 0.74% of the original concentrations. The residual CBz were about 77% of DiCBzs and 17% TriCBzs. The standards and ISs listed in Section 2.1 were then spiked into the cleaned fly ash-E.

#### 2.2.2. Actual fly ash

Fly ash-N was supplied by MSWI-N, which has two continuous stoker-type incinerators with a baghouse filter and a web scrubber. The fly ash-N had a TOC of 0.27%, which shows an excellent burnout of this fly ash. The distributions of the elements O, C, and Cl in the fly ash-N were about 33.7, 5.1, and 20.0%, respectively. The amounts of other elements were shown in Appendix B.

### 2.3. Analytical procedures

SE or MAE was conducted after spiking the standards and ISs into the cleaned fly ash. All the solvents used for SE and MAE were supplied by Kanto Kagaku, Inc. (Japan). For actual fly ash analyses, SE was conducted after air-drying for 3 days, and MAE was conducted after air-drying for various times that depended on the water content (WC). The extracts were then concentrated to a volume of 4 ml using a rotary evaporator at 40 °C. Following clean-up on a multi-layer silica gel column, the solvent containing the target compounds was concentrated to a volume of 2 ml using a rotary evaporator at 30 °C. The volume of the solvent was further reduced to 100  $\mu\text{l}$  by evaporation with compressed nitrogen gas. Finally, PCBs and CBzs were analyzed by GC-MS. The procedures for SE, MAE, clean-up, and GC-MS analyses are described below. All experiments were repeated three times.

#### 2.3.1. Microwave-assisted extraction

MAE was carried out using an MDS-2000 (CEM, Mathews, NC). This system allowed up to 12 extraction vessels to be irradiated simultaneously, in 1% increments, up to 2450 MHz of microwave energy at 100% power. In this study, we conducted three samples once. The pressure was measured using a water manometer that allowed readings of up to  $1.38 \times 10^6$  Pa. The temperature probe, a fiber optic with a phosphor sensor, allowed extraction temperatures between 20 and 200 °C to be selected in increments of 1 °C [17]. According to the results of our present study, the optimum temperature of 120 °C was conducted in this study [14]. The samples were placed in double-walled, lined digestion vessels with a volume of 110 ml. Inside each vessel, the samples came into contact with inner lin-

Table 1  
GC–MS conditions of PCBs analyzing

GC: HP6890 (Hewlett Packard, Palo Alto, CA, USA)

MS: HP5973 (Hewlett Packard, Palo Alto, CA, USA)

Column	HP-5MS	
Name	HP-5MS	
Film thickness	0.25 $\mu\text{m}$	
Length $\times$ inside diameter	60 m $\times$ 0.250 mm	
Inlet temperature	250 $^{\circ}\text{C}$	200 $^{\circ}\text{C}$
Injection volume	1.0 $\mu\text{l}$	1.0 $\mu\text{l}$
Interface temperature	295 $^{\circ}\text{C}$	280 $^{\circ}\text{C}$
GC oven program	150 $^{\circ}\text{C}$ $\rightarrow$ 185 $^{\circ}\text{C}$ (20.0 $^{\circ}\text{C}/\text{min}$ )	80 $^{\circ}\text{C}$ $\rightarrow$ 116 $^{\circ}\text{C}$ (2.0 $^{\circ}\text{C}/\text{min}$ )
	185 $^{\circ}\text{C}$ $\rightarrow$ 245 $^{\circ}\text{C}$ (2.0 $^{\circ}\text{C}/\text{min}$ )	116 $^{\circ}\text{C}$ $\rightarrow$ 122 $^{\circ}\text{C}$ (1.0 $^{\circ}\text{C}/\text{min}$ )
	245 $^{\circ}\text{C}$ $\rightarrow$ 290 $^{\circ}\text{C}$ (6.0 $^{\circ}\text{C}/\text{min}$ )	122 $^{\circ}\text{C}$ $\rightarrow$ 250 $^{\circ}\text{C}$ (10.0 $^{\circ}\text{C}/\text{min}$ )
Purge time	2.0 min	0.5 min
Analytical time	47.0 min	38.0 min
Carrier gas	Helium	Helium
Flow rate of carrier gas	1.0 ml/min	1.0 ml/min

ers and covers, made of Teflon perfluoroalkoxy (PFA). Each extraction vessel contained a rupture membrane designed to fail at elevated pressure ( $1.38 \times 10^6$  Pa). Each extraction vessel was located in a carousel, which resided within the Teflon-lined microwave cavity and rotated  $180^{\circ}$  during the microwave operation.

In the present study, 3.0 g fly ash was put into PFA vessels. For quantification, a standard solution (100  $\mu\text{l}$ ) and an IS solution (10  $\mu\text{l}$ ) of PCBs and CBzs were added into cleaned fly ash-E, and 10  $\mu\text{l}$  of an IS solution of PCBs and CBzs were added into actual fly ash-N. The extraction was performed at 120  $^{\circ}\text{C}$  [14] with a heating time of 15 or 25 min. After extraction, the vessels were cooled to room temperature before being opened. The MAE solvent mixtures applied for spiked fly ash-E were hexane/acetone (1:1), toluene/acetone (1:1), toluene/ $\text{H}_2\text{O}$ , methanol, or dichloromethane. The solvent mixtures for actual fly ash-N were toluene/acetone (1:1), toluene/acetone (5:3), and toluene/ $\text{H}_2\text{O}$ . The dielectric constants of water, acetone, methanol, dichloromethane, toluene, and hexane are 80.1, 20.0, 33.0, 8.9, 2.4, and 1.9 at 20–25  $^{\circ}\text{C}$ , which were concerned with polarity of solvents. On the other hand, the larger the dielectric constant, the more thermal energy is released and the more rapid is the heating for a given frequency.

When water was used as a solvent, the results are discussed as a function of the WC of fly ash [19]. The WC was calculated by fly ash's weights before and after acid pre-treatment or air-drying. And before concentration, the water was separated from toluene, and the residual water was eliminated by dried sodium sulfate.

### 2.3.2. Soxhlet extraction

The heating temperature was about 80  $^{\circ}\text{C}$ . Toluene (120 ml) was used as the extraction solvent, and 3.0 g fly ash was put into a cylindrical filter. For quantification, a standard solution (100  $\mu\text{l}$ ) and an IS solution (10  $\mu\text{l}$ ) of PCBs and CBzs were added to cleaned fly ash-E, and 10  $\mu\text{l}$  of an IS solution of PCBs and CBzs were added to actual fly ash-N. The extraction lasted

for 18–24 h. Following extraction, the apparatus was air cooled for 30 min.

### 2.3.3. Clean-up

All silica gels were purchased from Wako Pure Chemical Industries (Japan). The column for clean-up was filled with layers of KOH silica gel, 44%  $\text{H}_2\text{SO}_4$  silica gel, 22%  $\text{H}_2\text{SO}_4$  silica gel,  $\text{AgNO}_3$  silica gel, and dried sodium sulfate. Hexane (50 ml) was used to pre-wash the multi-layered column [20], and 4 ml of extraction solvent that had been concentrated by rotary evaporation flowed through the column. The target compounds were eluted from the column with 120 ml hexane at 2.5 ml/min.

### 2.3.4. Gas chromatography–mass spectrometry (GC–MS) analysis

The analysis was performed on an HP6890 series gas chromatograph (Hewlett Packard, Palo Alto, CA) equipped with a splitless injector (purge delay 1 min, purge flow 60 ml/min). The carrier gas was helium at a constant flow rate of 1 ml/min. The capillary column used was an HP-5MS (Hewlett Packard, Palo Alto, CA) with a length and inside diameter of 60 m and 0.250 mm, respectively, and a film thickness of 0.25  $\mu\text{m}$ . The gas chromatograph was connected to an HP 5973 mass-selective detector (electron impact, 70 eV), operated under the selected ion-monitoring (SIM) mode using the molecular ion of each compound at 1.5–2 scan/s. The interface temperature was maintained at 295  $^{\circ}\text{C}$  for PCBs and 280  $^{\circ}\text{C}$  for CBzs. The program condition for PCBs and CBzs of GC–MS were shown in Table 1, respectively.

## 3. Results and discussion

### 3.1. MAE efficiencies with different organic solvent mixtures

Organic solvent mixtures of hexane/acetone, methanol, dichloromethane, and toluene/acetone were used for the MAE

Table 2  
Recoveries of PCBs and CBzs in spiked fly ash using different organic solvent mixtures ( $n = 3$ )

	Solvent mixture	Volume (ml)	MAE recovery (%)±RSD							
			Di-	Tri-	Tetra-	Penta-	Hexa-	Hepta-	Octa-	
MAE	CBzs	Hexane/Acetone (1/1)	30	89.4±4.2	81.4±6.7	82.7±6.0	102.4±4.8	95.6±10.6		
		Methanol	15	41.5±19.2	113.4±25.5	112.5±4.4	117.0±4.8	99.4±6.8		
		Dichloromathane	15	-	-	-	-	-		
		Toluene/Acetone (1/1)	30	103.3±1.3	120.1±3.6	105.2±12.1	115.7±5.2	110.8±3.8		
		Toluene/Acetone (1/1)	20	104.8±5.1	116.9±11.0	101.6±14.6	121.0±12.2	114.4±8.1		
	PCBs	Hexane/Acetone (1/1)	30	91.5±7.5	109.3±9.5	82.5±9.3	89.9±5.2	59.5±5.0	84.8±3.4	73.5±2.4
		Methanol	15	29.8±0.5	81.5±3.4	112.9±9.3	146.4±8.1	77.0±4.6	104.2±9.0	101.9±3.8
		Dichloromathane	15	42.1±2.5	90.5±10.5	84.4±3.9	122.7±16.9	66.0±3.2	91.9±5.2	90.2±4.9
		Toluene/Acetone (1/1)	30	99.5±3.0	97.6±4.3	99.0±3.3	95.9±3.5	98.9±3.2	100.6±3.5	84.5±3.4
		Toluene/Acetone (1/1)	20	91.3±3.2	95.2±4.7	92.0±5.1	95.0±3.6	89.0±5.9	92.6±2.0	84.8±9.6
	Congener		PCB-105	PCB-156	PCB-157	PCB-169				
Co-PCBs	Hexane/Acetone (1/1)	30	54.3±5.3	39.9±4.9	63.7±9.8	31.3±4.3				
	Methanol	15	127.6±6.2	81.2±6.2	80.8±6.0	6.4±0.8				
	Dichloromathane	15	102.1±7.9	68.9±0.2	66.1±0.4	5.3±0.8				
	Toluene/Acetone (1/1)	30	106.5±3.9	107.6±3.7	104.2±3.9	90.2±2.1				
	Toluene/Acetone (1/1)	20	97.1±4.7	91.9±7.2	91.0±6.1	76.6±10.3				
SE	CBzs	Toluene	120	66.8±0.2	66.4±0.3	67.9±0.5	69.3±0.4	70.6±4.2		
	PCBs	Toluene	120	89.3±1.5	79.4±1.6	93.8±3.2	89.5±2.7	97.3±3.0	96.0±2.8	100.9±3.6
		Congener		PCB-105	PCB-156	PCB-157	PCB-169			
	Co-PCBs	Toluene	120	95.0±3.3	98.0±3.6	98.0±3.2	101.1±3.2			

Symbol ‘-’ denotes no data found. Extraction time: 15 min; water content: 20%.

of spiked fly ash. The recoveries of PCBs and CBzs are shown in Table 2. DiCBs extracted in a solvent of methanol or dichloromethane, and DiCBzs extracted with methanol had low recoveries, as shown in Table 2. Furthermore, Co-PCBs, especially PCB-169, were not extracted sufficiently in hexane/acetone, methanol, or dichloromethane as a solvent. When dichloromethane was used as a solvent, no CBzs were extracted from fly ash-E.

From Table 2, it showed higher extraction efficiencies in the non-polar solvent of toluene and hexane than the others. It could be supposed that solvent with low dielectric constant, which had weak polarity, was favor MAE extraction for PCBs and CBzs. As well, toluene performed better than hexane as a non-polar solvent, although their dielectric constants were not changed much. The principle “like dissolves like” could explain this result. Based on these results, toluene was used as the non-polar solvent in the following experiments.

### 3.2. Effect of irradiation time on MAE

As shown in Fig. 1, when irradiation time was decreased from 25 to 15 min, the extraction efficiencies increased slightly for PCBs in both spiked fly ash and actual fly ash. The lower recoveries at 25 min were originally attributed to analyte degradation as resulting from prolonged exposure to heat [18]. An irradiation time of 15 min was therefore used for the remainder this study.

### 3.3. Effect of water content on MAE

Water was used as a polar solvent in MAE instead of acetone. From our previous study, the extraction recoveries were deemed stable with a WC below 60% by using spiked fly ash [19].

With actual fly ash, the extraction efficiencies of PCBs and CBzs decreased about 80 and 21% with the reduction of WC from 20 to 90% [19].

When water was used as a polar solvent instead of acetone, the residual water after pre-treatment could be used as a polar solvent. Therefore, the air-drying time after acid pre-treatment could be shortened [19].

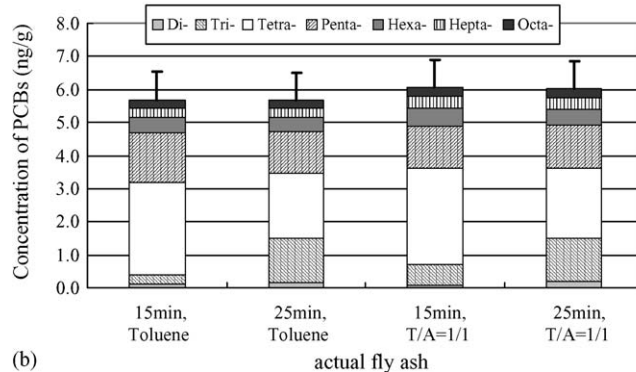
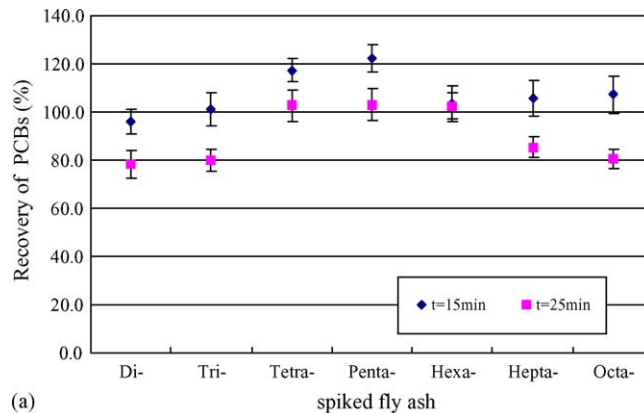


Fig. 1. Recoveries and concentrations of PCBs in fly ash at different irradiation time using MAE with a toluene/acetone solvent mixture ( $n = 3$ ): (a) spiked fly ash and (b) actual fly ash.

### 3.4. The effect of solvent mixture volume on MAE

#### 3.4.1. Spiked fly ash

When organic solvent mixture used for MAE, toluene/acetone solvent mixtures of 20 and 30 ml produced similar recoveries of PCBs, Co-PCBs, and CBzs, as shown in Table 1. This indicates that 20 ml of toluene/acetone was sufficient for the extraction of PCB and CBz from fly ash-E. The R.S.D. values of TetraCBzs and PentaCBz using 20 ml of solvent were about 25%, however, indicating that the experimental repeatability under these conditions was not very good.

In the other cases, the R.S.D. values for PCBs were less than 5% and those for CBzs, except those repeated above, were less than 10%.

On the other hand, water also applied as a polar solvent to extract PCBs and CBzs in fly ash by MAE in our present study [19]. When WC was 60%, the recoveries of PCBs, Co-PCBs, and CBzs homologue except PCB-169 increased as the volume of toluene increased. The recovery reduction of PCB-169 could have been due to the facts that non-polar substances are difficult to heat with microwave energy and that water acts as a barrier in MAE. As a result, it was difficult to transfer PCB-169 from fly ash with a high WC to the solvent using MAE [19].

#### 3.4.2. Actual fly ash

**3.4.2.1. The combined effects of different volume and ratio on MAE.** The concentrations of PCBs and CBzs extracted by MAE with different solvent mixtures, e.g., 30 ml (1/1), 50 ml (1/1), 30 ml (5/3), and 40 ml (5/3) of toluene/acetone, are shown in Fig. 2. Under the same ratio of the solvent mixture, a low solvent volume resulted in high MAE efficiencies. This could have been due to inadequate stirring of the solvent by the microwaves [13,21]. On the other hand, with a volume of 30 ml, the toluene/acetone ratio of 5/3 produced higher efficiencies than did a ratio of 1/1.

### 3.5. Comparison of MAE efficiencies and SE

#### 3.5.1. Spiked fly ash

The recoveries of PCBs by SE were about 80–100%, as shown in Table 2 and our previous study [19]; by MAE were about 90–110%. The recovery of PCB-169 from fly ash-E containing 60% water, however, was about 70%. The recoveries of CBzs by SE were approximately 60–70%, which are lower than the recoveries by MAE (about 90–120%). The homologues of CBzs are easily volatilized during the SE process because CBzs have low boiling points of about 180–320 °C. On the other hand, CBzs are difficult to volatilize using the closed vessels required for MAE. Thus, MAE had higher efficiencies than did SE for CBz analysis.

The R.S.D. values of PentaCBz and HexaCBz in fly ash with WC of more than 60% were high using MAE. Under all the experimental conditions, including those of SE, the R.S.D. values of all PCBs and CBzs, except PentaCBz and HexaCBz, were less than 15%. The repeatability of the extraction of higher

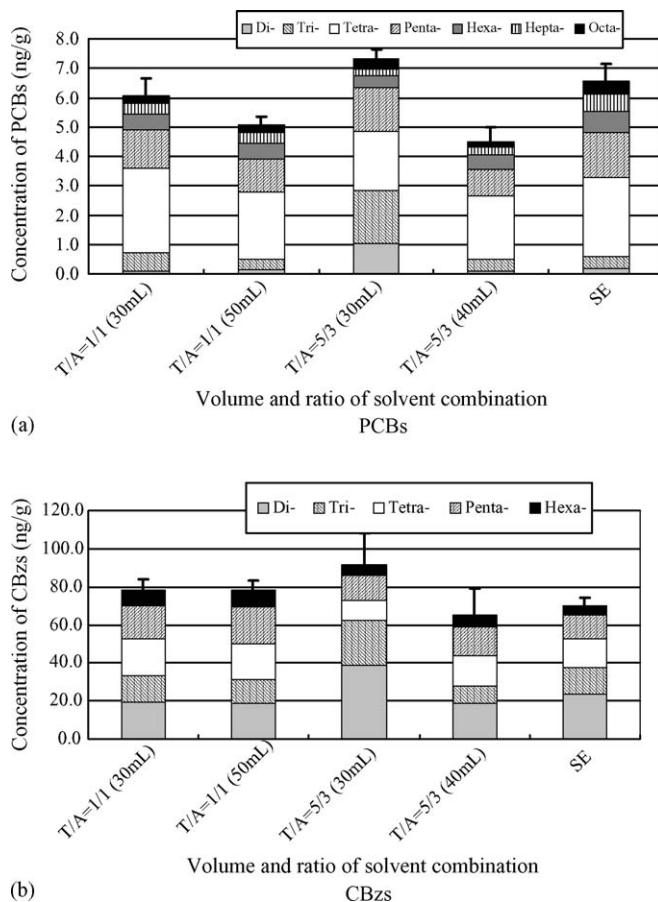


Fig. 2. Concentrations of (a) PCBs and (b) CBzs in actual fly ash with different volumes and ratios of solvent mixtures ( $n = 3$ ).

CBz homologues from fly ash containing water was therefore poor.

With water as the polar solvent, however, the recoveries of PCBs and CBzs varied little. Therefore, with the exception of the non-polar substance PCB-169, MAE produced almost the same efficiencies with a solvent mixture of either toluene/acetone or toluene/H<sub>2</sub>O. Moreover, MAE had higher efficiencies than did SE in spiked sample analyses.

#### 3.5.2. Actual fly ash

Both PCBs and CBzs, especially the lower homologues, were extracted better by MAE than by SE, as shown in Fig. 2.

The large amount of solvent needed for SE (120 ml of toluene was used in this study) meant that more time was required for the rotary evaporation process. Less than 30 ml of solvent was used in MAE, so the concentration process was faster approximately 20 h of extraction time.

## 4. Conclusions

In the present study, the optimum conditions for MAE included either a 30-ml volume of toluene/acetone (1/1) or a 15-ml volume of toluene, samples with less than 60% WC, and an irradiation time of 15 min. Under optimum conditions, MAE

offered a greater extraction efficiency than did SE for both PCBs and CBzs. In addition, MAE decreased the extraction time and reduced the solvent consumption: whereas SE can take 18–24 h and consume 120 ml of solvent, MAE took only 15 min and used 30 ml organic solvent. The technique is easy to manage, and the system is not costly compared with other modern techniques, such as SFE and ASE.

The water content of the sample was shown to be an important factor in MAE efficiency especially for PCBs. When the WC was under 60%, MAE showed a higher efficiency than SE and water can be used as a polar solvent in MAE.

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### Appendix A. Concentrations of PCBs and CBzs solution (solvent: *n*-hexane)

	IUPAC no.	Congener	Concentration (μg/ml)	Manufacture	
PCBs	PCB-14	3,5-Dichlorobiphenyl	5	Accustandard, USA	
	PCB-18	2,2',5-Trichlorobiphenyl	2.5	Accustandard, USA	
	PCB-28	2,4,4'-Trichlorobiphenyl	1	Accustandard, USA	
	PCB-52	2,2',5,5'-Tetrachlorobiphenyl	1	Accustandard, USA	
	PCB-66	2,3',4,4'-Tetrachlorobiphenyl	2	Accustandard, USA	
	PCB-95	2,2',3,5',6-Pentachlorobiphenyl	0.5	Accustandard, USA	
	PCB-105	2,3,3',4,4'-Pentachlorobiphenyl	0.5	Accustandard, USA	
	PCB-103	2,2',4,5',6-Pentachlorobiphenyl	1	Accustandard, USA	
	PCB-129	2,2',3,3',4,5-Hexachlorobiphenyl	0.5	Accustandard, USA	
	PCB-149	2,2',3,4',5',6-Hexachlorobiphenyl	0.5	Accustandard, USA	
	PCB-169	3,3',4,4',5,5'-Hexachlorobiphenyl	0.5	Accustandard, USA	
	PCB-156	2,3,3',4,4',5-Hexachlorobiphenyl	0.5	Accustandard, USA	
	PCB-157	2,3,3',4,4',5'-Hexachlorobiphenyl	0.5	Accustandard, USA	
	PCB-180	2,2',3,4,4',5,5'-Heptachlorobiphenyl	0.5	Accustandard, USA	
	PCB-187	2,2',3,4',5,5',6-Heptachlorobiphenyl	0.5	Accustandard, USA	
	PCB-194	2,2',3,3',4,4',5,5'-Octachlorobiphenyl	0.5	Kanto Kagaku, Japan	
	PCB-199	2,2',3,3',4,5,6,6'-Octachlorobiphenyl	0.5	Kanto Kagaku, Japan	
	<sup>13</sup> C-PCBs	PCB-28	2,4,4'-Trichlorobiphenyl	3	Wellington Lab., Canada
		PCB-52	2,2',5,5'-Tetrachlorobiphenyl	3	Wellington Lab., Canada
PCB-101		2,2',4,5,5'-Pentachlorobiphenyl	3	Wellington Lab., Canada	
PCB-118		2,3',4,4',5-Pentachlorobiphenyl	3	Wellington Lab., Canada	
PCB-138		2,2',3,4,4',5'-Hexachlorobiphenyl	3	Wellington Lab., Canada	
CBzs	1,3-DiCBz	1,3-Dichlorobenzene	10	Nacalai, Japan	
	1,3,5-TriCBz	1,3,5-Trichlorobenzene	10	Tokyo Kasei, Japan	
	1,2,4-TriCBz	1,2,4-Trichlorobenzene	10	Nacalai, Japan	
	1,2,3-TriCBz	1,2,3-Trichlorobenzene	10	Nacalai, Japan	
	1,2,4,5-TetraCBz	1,2,4,5-Tetrachlorobenzene	10	Nacalai, Japan	
	1,2,3,5-TetraCBz	1,2,3,5-Tetrachlorobenzene	10	Nacalai, Japan	
	1,2,3,4-TetraCBz	1,2,3,4-Tetrachlorobenzene	10	Tokyo Kasei, Japan	
	PentaCBz	Pentachlorobenzene	10	Tokyo Kasei, Japan	
	HexaCBz	Hexachlorobenzene	10	GL Sciences, Japan	
<sup>13</sup> C-CBzs	1,4-DiCBz	1,4-Dichlorobenzene	10	Wellington Lab., Canada	
	1,2,3-TriCBz	1,2,3-Trichlorobenzene	10	Wellington Lab., Canada	
	1,2,3,4-TetraCBz	1,2,3,4-Tetrachlorobenzene	10	Wellington Lab., Canada	
	PentaCBz	Pentachlorobenzene	10	Wellington Lab., Canada	
	HexaCBz	Hexachlorobenzene	10	Wellington Lab., Canada	

### Appendix B. Element amount by XRF analysis

Element	Element amount (%)	
	Fly ash-E	Fly ash-N
O	35.6	33.7
C	17.1	5.1
Ca	10.0	18.7
Si	9.0	3.5
Cl	6.7	20.0
K	3.8	4.4
Al	3.6	1.4
Zn	2.6	3.3
Na	2.4	5.1
Fe	2.1	0.5
Mg	1.7	0.8
S	1.3	1.5
Ti	1.1	0.6
Pb	1.1	0.6
Others	1.7	0.8

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