

Application of Microwave-Assisted Extraction to the Analysis of Chlorinated Aromatic Compounds
in Solid Samples from Municipal Solid Waste Incinerators
ごみ焼却施設における固体サンプル中芳香族塩素化合物に対するマイクロウェーブ抽出法の適用

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Abstract: Two classes of dioxin precursors: polychlorinated biphenyls (PCBs) and chlorobenzenes (CBzs), which can produce dioxin-like health risk effects, are formed from Municipal Solid Waste Incinerators (MSWIs). Shortening the analytical time is important to determine the concentration of these compounds and to evaluate the risk effects quickly. In this study, Microwave-Assisted Extraction (MAE) is compared with the traditional extraction method, Soxhlet Extraction (SE) to determine the extraction efficiency for PCBs and CBzs analysis. Under different experimental conditions, MAE efficiency using the samples with spiked standards and actual samples were compared with those of SE, respectively. Water is a safe and environmentally friendly solvent, was used as an MAE solvent for PCBs and CBzs analysis. Evaluating the extraction efficiency of MAE and SE, MAE proved to have high extraction efficiency on spiked samples and fly ash containing water. Furthermore, MAE decreased the extraction time and reduced organic solvent consumption.

Keywords: dioxin, polychlorinated biphenyls (PCBs), chlorobenzenes (CBzs), Municipal Solid Waste Incinerators (MSWIs), soxhlet extraction (SE), microwave-assisted extraction (MAE), chlorinated aromatic compounds

論文要旨：ごみ焼却施設において、PCBs や CBzs がダイオキシン類の前駆物質として発生する。このような化合物の濃度やリスクの迅速な評価を行うために、分析時間の短縮が重要な課題となっている。本研究では、分析手順の中で最も時間を要する抽出操作に着目し、マイクロウェーブ抽出法を用いて固体試料中に含まれる PCBs と CBzs を分析し、従来法であるソックスレー抽出法による結果と比較した。クリーンアップ済の飛灰に既知量の PCBs と CBzs を添加した試料と、元の飛灰や汚泥を対象として、抽出時間や溶媒の種類と量を変化させて抽出効率を調べた結果、マイクロウェーブ抽出法では、短時間、低溶媒量で抽出が可能であることが分かった。さらに汚泥や酸処理後飛灰などの水分を含む試料にも適用できることが確認できた。また溶媒の種類として水を利用できることから、低環境負荷の方法としてさらなる応用の可能性が示唆された。

キーワード：ダイオキシン類、PCBs、CBzs、ごみ焼却施設 (MSWI)、ソックスレー抽出法 (SE)、マイクロウェーブ抽出法 (MAE)、芳香族塩素化合物

I. Introduction

Some kinds of chlorinated organic compounds, such as two classes of dioxins precursors: PCBs and CBzs are generated from MSWIs. The emitted concentration of them has a strong correlation with dioxins levels, which have high toxicity. Therefore, it is necessary to measure the concentrations of PCBs and CBzs in various medias of MSWIs. There are many procedures to analyze PCBs and CBzs. Among them, the procedure of extraction is the most important. Accompanied with the quantity of waste generation, it is necessary to seek a rapid extraction method to evaluate the amount of dioxins from MSWIs accurately.

Comparing with the traditional extraction method SE, MAE is becoming attractive these days. MAE is less time-consuming and uses much smaller amount of organic solvents. In this study, first, we try to determine the best experimental conditions of MAE to analyze PCBs and CBzs in fly ash and sludge from MSWIs. Secondly, water as a solvent was used as the polar solvent in MAE. And then, the extraction efficiency of MAE was compared with that of SE in spiked samples and actual samples containing water.

II. MAE efficiency on spiked samples

Extraction efficiency of MAE is influenced by many factors. The most common parameters are extraction time, solvent combination, solvent volume, extraction temperature and matrix characteristics including water content. The extraction time of 15min, the extraction temperature of 120°C, and 30mL organic solvent mixture of toluene/acetone (1/1), proved to be the best experimental conditions for samples without water. MAE had a high extraction efficiency when the water content was under 40%, using 15mL toluene, with an irradiation time of 15min and extraction temperature of 120 °C. Under the experimental conditions above, MAE had higher extraction efficiency than SE. And the relative standard deviation (RSD) values were stable.

Fig.1 and Fig.2 show the recoveries of PCBs and CBzs in MAE and SE. The figures showed that MAE had similar extraction efficiency in analyzing PCBs, while gave higher extraction efficiency than SE did for CBzs analysis.

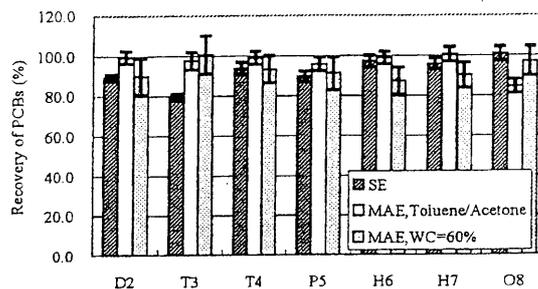


Fig.1 Recoveries of PCBs in fly ash-E by SE and MAE with different polar solvent

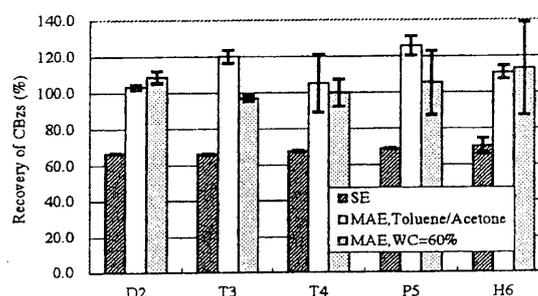


Fig.2 Recoveries of CBzs in fly ash-E by SE and MAE with different polar solvent

III. MAE efficiency on actual samples

In the case of using samples of fly ash, under the experimental conditions of an extraction temperature of 120°C, irradiation time of 15min, using 30mL solvent mixture of toluene/acetone (5/3), water content of 20%, MAE had higher extraction efficiency than that of SE. In the case of sludge, for it is difficult for sludge to be air-dried, high MAE efficiency was not obtainable. However, when the water content decreased, the MAE efficiency increased significantly. Therefore, it is necessary to try to find a good method to reduce the water content of sludge.

IV. Conclusion

PCBs and CBzs in spiked samples and in fly ash, MAE showed higher extraction efficiency than that of SE. The RSD values were stable for both of them. The RSD values of PCBs were lower than those of CBzs. Since the boiling points of CBzs are lower than PCBs, it was a possibility that CBzs were volatile during the concentration process.

V. Future Trends

In this study, when sludge samples were used SE had a higher extraction efficiency than MAE. It is hoped that MAE efficiency will increase by reducing the water content of the sludge in the future. As MAE is a method that can be utilized to analyze chlorinated aromatic compounds in solid samples, therefore other chlorinated organic compounds in solid samples with water, such as soil, sediment and sewage sludge could also be expected to be analyzed using MAE.