Determination of 26 polychlorinated biphenyls congeners in soil samples using microwave-assisted extraction with open vessel and gas chromatography

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ABSTRACT
A procedure focused on microwave-assisted extraction in open vessel (MAE-OV) and gas chromatography with electron capture detection (GC-ECD) was used for the determination of 26 congeners of polychlorinated biphenyls (PCBs) in soil samples. The limit of detection (LOD) and limit of quantification (LOQ) were evaluated for commercial PCBs mixture Aroclor1260. LOD and LOQ were calculated for each PCB congener, in the ranges (0.03–0.27 ng g⁻¹) and (0.11–0.70 ng g⁻¹), respectively. After optimization, 26 PCBs congeners were successfully extracted from soil samples with recovery amounts ranging between 84.7% and 117.3% for all PCBs congeners. The evaluated method of MAE-OV showed good separation and extraction of all PCBs congeners from soil samples. Extraction parameters such as solvent choice, power and extraction time were investigated. This study indicated that MAE-OV could be an interesting alternative method to extract PCBs from soils, since it is economical, easy, fast and requires low amounts of solvents.

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1. Introduction
Polychlorinated biphenyls (PCBs) are one group of persistent organic pollutants that are the synthetic aromatic compounds (209 congeners) with the general structure of C_{12}H_{10−x}Cl_{x} (1). PCBs have been produced commercially since 1930, and they were mostly used as coolants, lubricants and fluids in electrical transformers and capacitors (2, 3). However, PCBs congeners are identified as carcinogenic, immunotoxic and affect behavioral stability (4). Due to their chemical nature, some PCBs congeners are highly persistent in the environment mainly in soil and sediment and can be accumulated in the food chain (5). Many studies assessed the risk associated with high levels of PCBs in contaminated soil (6, 7). It is therefore important in green chemistry to develop the extraction step in the analytical procedure for an efficient determination of PCBs traces in soil samples. Furthermore, many studies investigated in comparison a range of methods for the extraction of PCBs and other organic traces from the soil, and some of them referred...
to the conventional Soxhlet extraction method (8, 9). An advanced microwave-assisted extraction method was widely carried out for the extract of trace organic pollutants from environmental samples (10). The microwave-assisted extraction in open vessel (MAE-OV) is a method that used the microwave power for heating solvents which exchange with a sample for extract analytes from the sample matrix into an organic solvent (11, 12).

The application of microwave extraction in closed or open vessel under controlled parameters such as choice of extracting solvent, volume of solvent, power and processing extraction time can be conducted to obtain higher extraction recoveries and good separation of persistent organic pollutants from the environment matrices such as soil and sediment (13). The choice of the nature and the volume of the extracting solvent have an important effect on the extraction efficiency. Furthermore, the polarity of the solvent is another important factor in the MAE-OV extraction process. The aim of the present study is to develop a simple and efficient MAE-OV method with an open-vessel extraction apparatus for the determination and analysis of 26 PCBs congeners of spiked soil samples. To evaluate this technique for efficiency recoveries, extraction parameters have been optimized such as extraction time and power and extracting solvents with different polarities. The results obtained by MAE-OV were compared to Soxhlet extraction (SE), as a conventional and referential method of extraction of trace organic from environmental samples.

2. Material and methods

2.1. Instrument of MAE-OV in open vessel

To perform the extraction of 26 PCBs from soil samples, the MAE-OV in an open vessel was performed with a modified kitchen microwave oven (2450 MHz, M1933N Samsung, Korea) (cf. Figure 1). The modified parameters accorded with those given by previous studies, essentially based on the works of Ratola et al. (14) and Herbert et al. (15). The system operated as an open-vessel multimode extraction system. With the multimode system, the dispersion of microwave radiation within microwave cavity, all contents (cavity and sample) were irradiated (16). The solvent is heated and refluxed through the sample.

2.2. Standards and chemicals

All used solvents such as acetone, dichloromethane (DCM) and hexane were of high-performance liquid chromatographic (HPLC) purity grade. Florisil was purchased from Sigma-Aldrich. Standard PCBs solutions Aroclors Ar1260 and Ar1248 were purchased from Supelco farm, USA.

A standard solution that contained internal standard (IS) and Ar1260 at a concentration of 100 µg mL\(^{-1}\) was prepared in hexane. Dichlobenil as IS at a concentration of 100 µg mL\(^{-1}\) was prepared in hexane. All standard solution series in the range 0.1–3 µg mL\(^{-1}\) were stored at 5°C, until used for calibration curve.

2.3. Sampling, storage and homogenization

For the optimization of the extraction process, unpolluted soil samples were collected from an agricultural site from eastern Algiers, where it was used in the preparation of spiked soil samples. According to Aydin et al. (17), collected soils were sieved to <2 mm and then stored in a freezer until use in the spiking procedure step. Total PCBs concentrations in spiked soil samples were 80 ng g\(^{-1}\). The spiking soil was homogenized by mixing and shaking for 30 min, using a horizontal shaker at 220 rpm. Then, the soil samples were stored in a freezer for 3 days prior to the extraction investigation step.

2.4. Microwave-assisted extraction in open vessel

The spiked sample (10 g) was added to the extraction corvettes, in which an amount of extraction solvent will be additional. In open-vessel extraction, the power is limited to the boiling point of the used solvent. In order to perform this opening extraction system, three powers (150, 300 and 600 W) of microwave heating have been
tested. The extracts were cleaned through mini-columns with 1.5 g of 6% (w/w) deactivated Florisil (18).

2.5. Conventional SE

The spiked soil (10.0 g) mixed with anhydrous sodium sulfate (Na₂SO₄) (2 g) was placed in a prewashed cellulose thimble, and then extracted with 250 mL of n-hexane/acetone for 8 h at a rate of 7–8 cycles/h, in a prewashed glass fiber thimble. Extracts were concentrated with Rota-vapor system (Heidolph, LABORATQ 4001, Germany), and then were cleaned through Florisil column.

2.6. GC-ECD chromatography analysis of PCBs in the soil samples

PCBs congeners (26) in extracts were determined using a gas chromatograph (GC) apparatus (500 Clasus, Perkin Elmer instruments) equipped with an electron capture detector (ECD). The GC-ECD was equipped with a DB5-MS capillary column (5% phenyl: 95% dimethylpolysiloxane, 60 m length × 0.25 mm i.d × 0.25 mm film thickness) (J&W Scientific, USA). Ten microliters of Dichlobenil at 100 µg mL⁻¹ were added to 980 µl of each liquid extract, and then 1 µl of the extract was injected in splitless mode at 280°C. Separation was carried out by using He as carrier gas, with the following oven temperature programs: 75°C for 2 min, then up to 150°C (15°C min⁻¹), then to 300°C (1.5°C min⁻¹) and finally maintained at 300°C for 2 min. The total analysis time was 90 min. For acquisition of GC data, TotalChrom software (Perkin Elmer Technologies) was used. Target compounds were identified according to the retention time of standards Aroclors (Ar1260 and Ar1254), and internal standards for theirs quantification. The PCBs calibration curve was prepared with six concentration levels of standard solution. All steps involved in the procedure of extraction and analysis of PCBs in soil samples are illustrated in Figure 2.

All data were reported as the mean ± standard deviation of three replicates (n = 3). Statistical analyses were performed using SPSS 18 software and Microsoft Excel.

2.7. Quality control and quality assurance

In the present work, the analytical procedure was monitored by strict quality assurance and quality control. All the glasswares used in this study were pre-cleaned with hexane and acetone and dried in the oven at 280°C overnight. The solvents used were of high purity (HPLC grade) and all other materials were rinsed with hexane and acetone before use.

![Figure 2. The flow chart representing experimental design of analysis procedure in this study.](image)

Table 1. Calibration parameters for the extraction and analysis of PCBs by gas chromatography GC-ECD.

| No | Compound | R²  | LOD (ng g⁻¹) | LOQ (ng g⁻¹) |
|----|----------|-----|--------------|--------------|
| 1  | PCB52    | 0.9976 | 0.03 | 0.11 |
| 2  | PCB 64   | 0.9923 | 0.15 | 0.51 |
| 3  | PCB 95   | 0.9962 | 0.09 | 0.29 |
| 4  | PCB101   | 0.9978 | 0.10 | 0.33 |
| 5  | PCB136   | 0.9952 | 0.17 | 0.62 |
| 6  | PCB 110  | 0.9968 | 0.20 | 0.69 |
| 7  | PCB 151  | 0.9991 | 0.13 | 0.42 |
| 8  | PCB 135/144 | 0.9964 | 0.08 | 0.26 |
| 9  | PCB149/118 | 0.9974 | 0.09 | 0.31 |
| 10 | PCB134   | 0.9977 | 0.12 | 0.39 |
| 11 | PCB 132  | 0.9972 | 0.21 | 0.69 |
| 12 | PCB153   | 0.9975 | 0.06 | 0.22 |
| 13 | PCB141   | 0.9973 | 0.12 | 0.47 |
| 14 | PCB179   | 0.9968 | 0.14 | 0.36 |
| 15 | PCB138   | 0.9945 | 0.11 | 0.43 |
| 16 | PCB187   | 0.9971 | 0.06 | 0.31 |
| 17 | PCB183   | 0.9967 | 0.10 | 0.33 |
| 18 | PCB174   | 0.9959 | 0.27 | 0.68 |
| 19 | PCB177   | 0.9963 | 0.09 | 0.30 |
| 20 | PCB171/156 | 0.9968 | 0.15 | 0.48 |
| 21 | PCB180   | 0.9984 | 0.22 | 0.72 |
| 22 | PCB170   | 0.9976 | 0.08 | 0.33 |
| 23 | PCB199   | 0.9979 | 0.13 | 0.49 |
| 24 | PCB203   | 0.9966 | 0.016 | 0.042 |
| 25 | PCB195   | 0.9965 | 0.011 | 0.033 |
| 26 | PCB194   | 0.9979 | 0.021 | 0.063 |
LOQs ranged from 0.11 to 0.70 ng g$^{-1}$. The linear dynamic range was established by six points of known concentrations of certified standard solution of Aroclor1260 (e.g. 0.1–3 µg L$^{-1}$). The calibration curves of all PCBs were performed with six-point levels of Aroclor1260 before each batch of samples was analyzed, and the correlation coefficients ($R^2$) obtained were >0.992 for 26 PCBs. For each PCB congener, LOQs, LODs and $R^2$ are listed in Table 1.

3. Results and discussion

3.1. Optimization of the MAE-OV

3.1.1. Influence of the extraction solvent

In order to evaluate the efficiency recoveries of all the 26 PCBs congeners from spiked soil samples, three organic solvents were investigated: hexane/acetone, acetone/DCM and DCM (cf. Table 2). The testing of solvents showed a remarkable difference in total recoveries of PCBs from soils (cf. Figure 3). Highest recoveries were obtained by using hexane/acetone (v/v, 1:1), which is the most organic solvent chosen for extracting PCBs from environmental solid matrices (19–22). Dichloromethane has a higher dipole moment (index of polarity) than hexane/acetone (3.3 and 2.7, respectively), but gives a lower extraction recovery. This effect can be explained by taking into consideration the higher boiling temperature degree Celsius observed with hexane/acetone, than with DCM (52°C and 38.6°C, respectively) (cf. Table 2). Nevertheless, the mixture of acetone/DCM (v/v, 1:1) showed a much lower recovery. However, the mixture of hexane/acetone (v/v, 1:1) is a most efficient solvent for extracting all 26 PCBs congeners from spiked soil. This result is in concordance with other cited works for the extraction of organic chloride compounds and petroleum hydrocarbons in environmental samples (20, 23).

3.1.2. Influence of extracting power

Three levels (150, 300 and 600 W) of extracting power were tested on the extraction efficiency of PCBs. For all testing steps, the samples were preheated at 150°C for 2 min, in order to avoid vigorous reactions. Furthermore, the extraction time of 30 min for each power level was defined.

The results of the influence of the microwave power on the recovery of PCBs in MAE-OV extraction method in open-vessel mode indicated that a high extraction recovery was obtained by the application power of 600 W (cf. Figure 4). Generally, in open-vessel microwave extraction which operates in multimode type, the temperature distribution is usually asymmetrical and can therefore influence recovery (15, 24). An advantage of

![Figure 3](image-url) Individual recuperation of 26 PCBs and total PCBs recoveries in soil using three extracting solvents (hexane/acetone, acetone/DCM and DCM).
open-vessel extraction mode is the increased safety due to lower pressure (25, 26).

3.1.3. Influence of extracting time

An important parameter in the extraction process is the time of extraction, which can influence directly the recoveries of analytes. In order to determine the optimal extraction time for good recoveries of PCBs from soil using microwave-assisted extraction in open vessels, soil samples (10 g) were extracted by solvent (three testing solvents) at time periods of 20, 30, 40, 45 and 60 min. Figure 5 represents recoveries obtained for total of a total of 26 PCBs congeners with highest recoveries (92–113%) observed at 30 min, and then this time can be considered as an optimal time to apply in the extraction process with all testing solvents. The

![Figure 4. Total of PCB recoveries in soil under different powers of microwave.](image)

![Figure 5. Recoveries of 26 PCBs congeners obtained from soil samples using acetone/DCM, hexane/acetone and DCM as extractions solvents at 600 W during several extraction times.](image)
The extraction period of 5–30 min showed much lower recoveries for PCBs. The extraction time longer than 30 min showed different recoveries and could be due to the degradation transformation of PCBs under longer exposition to pressure (27).

### 3.2. MAE-OV versus closed vessel

MAE-OV with an open-vessel system operated for the extract of PCBs at the atmospheric pressure then might indicate a more safety condition than the closed system. Also, the open system can give a possibility to add solvent to the system during the extraction process. Also, no further clean-up steps were required for the system before analysis, and we can also use vessels made of various materials. Therefore, MAE-OV with open system required low cost and easy automation of the equipment (lower investment for the apparatus).

Despite the fact there are some advantages of microwave-assisted extraction with closed vessel system (MAE-CV), such as possibility of operating the system of MAE-CV with higher temperatures than with open systems, and also no risk of airborne contamination and no loss of volatile compounds, this closed system needs an expensive equipment, and also it must be sealed well in the microwave and pressure-compatible vessel before the extraction takes place. Also, when the extraction is finished, the vessels must be cooled to room temperature before they can be opened. These steps can increase the overall sample treatment time, subsequently increasing the cost and energy involved (28, 29).

### 3.3. MAE-OV versus referential Soxhlet extraction

The results of extractions of PCBs from soil samples by both methods, MAE-OV and Soxhlet, are presented in Figure 6 and show good recoveries for most of the 26 PCBs congeners by MAE-OV versus theirs recoveries with conventional Soxhlet extraction, excepting for some lower weight PCBs including PCB 52, PCB64, PCB136 and PCB101, and for co-eluted PCBs such as PCB135/144 and PCB 149/118, which have little superior recoveries with Soxhlet extraction. The results (Figure 6) indicate better recoveries obtained by MAE-OV for Hexachlorobiphenyls (Hexa-CBs), Heptachlorobiphenyls (Hepat-CBs) and Octachlorobiphenyls (Octa-CBs) than those obtained with the Soxhlet method. Also, no difference in recoveries of Petachlorobiphenyl (Penta-CBs) was obtained with both methods. Therefore, a better recovery of tetrachlorobiphenyl (Tetra-CBs) with the Soxhlet method than those obtained with the MAE-OV method was reported (41.86 versus 27.27 ng g⁻¹, respectively). Moreover, MAE-OV has more advantages versus conventional Soxhlet extraction method in reducing the execution time, which is 30 min with MAE-OV versus 8 hours with Soxhlet, and reduction of amounts of solvents with 30 mL with MAE-OV versus 250 mL with referential Soxhlet extraction.

Generally, MAE-OV can be proposed as an alternative technique with many advantages over the Soxhlet method, which is considered the referential method for extraction of PCBs in solid environmental matrices. MAE-OV has shown a reduction in the extraction time and volume of solvents, with improving efficiency, selectivity and good recoveries for PCBs in contaminated soils, also with a decreased energy and process cost in this present work.

### 4. Conclusions

These results indicated that the microwave-assisted extraction in the open vessel method can be efficiently used to extract PCBs from soil samples. Recoveries of
26 PCBs from the spiked soil ranged from 84.7% to 117%, with the RSD less than 10%. Highest levels of recuperation PCBs were obtained with an extraction time of 30 min, hexane/acetone as solvent and under a power of 600 W (100%). Moreover, MAE-OV gave several advantages over the traditional Soxhlet extraction including use of less solvent, short time of extraction, low cost and easy to use.

**Disclosure statement**

No potential conflict of interest was reported by the authors.

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