

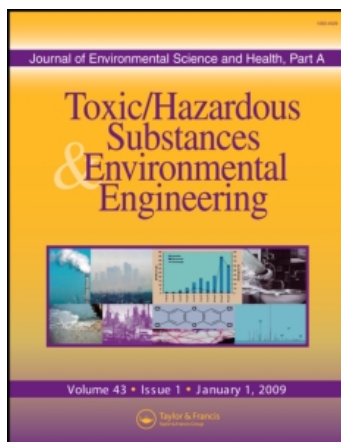
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Optimization of Microwave-Assisted Extraction of Polycyclic Aromatic Hydrocarbons from Sediments

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The extraction methods for the determination of polycyclic aromatic hydrocarbons (PAHs) from spiked sediment containing benzo[*k*]fluoranthene, chrysene, acenaphthene, anthracene, carbazole and indeno[1,2,3-*c,d*]pyrene, using pressurised microwave-assisted extraction (PMAE) and sonication extraction were optimised. Each PAH in the spiked sediment was quantified by using the spectrofluorometric method. The optimised methods were compared on the extraction efficiency of 14 PAHs in a certified sediment reference material, LGC6188. Recoveries of 14 PAHs were performed by HPLC-DAD at 254 nm using the standard addition method. The results showed that the most extraction efficient method was pressurised microwave-assisted extraction with cyclohexane:acetone (3:2) for 15 min at 140% boiling point of acetone. Under this condition for the extracted certified sediment reference material recovery was 96.55%. It can be concluded from the experimental results that pressurised microwave-assisted extraction in a closed system provides a good alternative to sonication extraction for the extraction of PAHs from sediments. Furthermore, the PMAE was also applied to determine PAHs from sediments taken from the main entrance of Kasetsart University on Paholyotin road in Bangkok.

Key Words: Pressurised microwave-assisted extraction; Sonication extraction; Polycyclic aromatic hydrocarbons.

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INTRODUCTION

Polycyclic aromatic hydrocarbons (PAHs) are widespread environmental contaminants possessing high mutagenicity and carcinogenicity.^[1,2] The most widely used liquid-solid Soxhlet extraction technique requires 6–24 hours, consumes a large volume of organic solvents and is laborious.^[3–5] Other extraction methods such as sonication,^[6] rotary tumbling^[7] and cyclic stream extraction^[8] were also used for the extraction of PAHs from samples. Disadvantage of these extraction methods were time consuming and using large volumes of extraction solvent. Of the new techniques that have appeared in the recent years, microwave-assisted extraction may provide a good alternative. This emerging technique has been used to reduce the volume of solvent required, improve the precision of analyte recoveries, reduce extraction time, minimize the consumption of energy and decrease costs.^[9] Microwave energy may be applied to the sample in two ways: by pressurized microwave-assisted extraction (PMAE) in a closed-system under high pressure and by focused microwave-assisted extraction (FMAE) in an open-system under atmospheric pressure.^[10] The closed nature of PMAE may prevent the loss of some PAHs during extraction.^[11,12] Furthermore, PMAE permits rapid extractions with reduced amounts of common laboratory solvents at elevated temperatures (under higher pressure the solvent mixture can be heated above its boiling point).

The aim of the present study was to compare the optimum extraction efficiencies of PAHs by the PMAE with the efficiencies obtained by conventional sonication extraction. This is to use the optimized method for the extraction of PAHs from sediment samples.

This paper discusses the use of PMAE to recover PAHs from sediments. Type of extraction solvents, time of irradiation and temperature of irradiation were varied systematically to determine the optimum conditions for the extraction of PAHs from spiked sediment samples. The optimized condition of PMAE was compared with the sonication method and was used to extract PAHs from sediment collected from the Paholyotin road, which is one of the most heavy traffic areas in Bangkok.

MATERIALS AND METHODS

Chemicals

All reagents used were of analytical reagent grade. Carbazole was purchased from Chem Service (West Chester, PA, USA). Benzo[*k*]fluoranthene, chrysene and anthracene were purchased from Fluka (Steinheim, Switzerland). Acenaphthene and indeno[1,2,3-*c,d*]pyrene were purchased from Merck (Darmstadt, Germany). Methanol, dichloromethane and cyclohexane were

purchased from Lab-scan (Bangkok, Thailand). Acetone was purchased from J. T. Baker (Phillipsburg, NJ, USA). Acetonitrile was purchased from BDH (Poole, England). n-Hexane was purchased from Fisher Chemicals (Fair Lawn, New Jersey, USA). Solvents for HPLC were of that grade. All other solvents were GPG.

Standard Compounds

A mixture of 16 components PAHs, each 10 ng μL^{-1} in acetonitrile: Acenaphthene (Act), acenaphthylene (Acy), anthracene (Ant), benzo[a]anthracene (B[a]A), benzo[a]pyrene (B[a]P), benzo[b]fluoranthene (B[b]F), benzo[g,h,i]perylene (B[g,h,i]P), benzo[k]fluoranthene (B[k]F), chrysene (Chr), dibenzo[a,h]anthracene (DB[a,h]A), fluoranthene (Flt), fluorene (Flu), indeno[1,2,3-c,d]pyrene (I[c,d]P), naphthalene (Nap), phenanthrene (Phe) and pyrene (Pyr) was purchased from Sigma-Aldrich (St. Louis, MO, USA). A certified sediment reference material, LGC6188 was purchased from Laboratory of the Government Chemist (Teddington, Middlesex, UK). Stock solutions of the standard mixture of PAHs were made up in acetonitrile from the received commercial standard mixture.

Apparatus

The MARS-X (CEM Corporation, Mathews, NC, USA), pressurised microwave-assisted extractor. The Cary Eclipse spectrofluorometer (Varian, Palo Alto, CA, USA) was used to analyze each PAH in the synthetic sample. The high performance liquid chromatographic system (HPLC) was composed of an Agilent 1100 series binary pump system, a 1100 series auto sampler, a 1100 photodiode array detector (Agilent, Palo Alto, CA, USA) and a ChromSpher PAH: 250 \times 4.6 mm I.D. (Varian, Palo Alto, CA, USA) column. A R-205 (BUCHI, Postfach, Switzerland) rotary evaporator was used to remove solvent. A CREST (Crest Ultrasonic Corporation, Pico Rivera, CA, USA) ultrasonic bath was used to dissolve and extract PAHs. The Centurion 2000 series (Centurion Scientific, West Sussex, UK) centrifuge was used at 5500 rpm in the cleanup step.

Spiked Sediment Samples

Sediment from the Mekong River, Thailand, was used for the preparation of spiked sediment samples. A 100 mL of solution consisting 5,000 mg L^{-1} of each PAH namely, benzo[k]fluoranthene, chrysene, acenaphthene, anthracene, carbazole and indeno[1,2,3-c,d]pyrene was added into 100 g of dried sediment in an Erlenmeyer flask. The mixture was shaken at 25°C for 48 hr. The treated sediment was washed with double-distilled water and filtered through a filter paper (Whatman, GF/C) and dried at room temperature for 48 h.

PAH Determination

The 6 PAHs in the spiked sediment sample were quantified by spectrofluorometry. Benzo[*k*]fluoranthene, chrysene, acenaphthene and carbazole were determined from their calibration graphs at the excitation and emission wavelength of each PAH in the presence of ethanol as solvent. The linear dynamic range was established from a 5-point calibration curve (i.e., 0.005, 0.010, 0.020, 0.030 and 0.040 mg kg⁻¹ for each PAH). Anthracene and indeno[1,2,3-*c,d*]pyrene were determined using the standard addition method to reduce the matrix effect. The excitation and emission wavelength were 267 nm and 380 nm for chrysene, 233 nm and 356 nm for carbazole, 227 nm and 321 nm for acenaphthene, 250 nm and 510 nm for indeno[1,2,3-*c,d*]pyrene, 307 nm and 407 nm for benzo[*k*]fluoranthene, 252 nm and 372 nm for anthracene. The 14 PAHs in the certified sediment reference material, LGC6188 were analyzed by HPLC using the EPA 8310 method. A 30 μ L of extract from each extraction method was injected and separated on the ChromSpher PAH column under the following conditions: gradient elution (A = water, B = acetonitrile: 0–25 min, 50–100% B), column temperature 25°C. Each PAH was identified by its retention time using the photodiode array detector at 254 nm.

EXTRACTION PROCEDURES

Microwave-Assisted Extraction

All extractions were carried out in triplicate. A 0.25 sample of the spiked sediment was placed in the lined Teflon vessel, covered by 20 mL of solvent and placed in the MARS-X microwave extraction system. The type of solvent, time of irradiation and temperature of irradiation were varied systematically. The extraction solvents used were methanol, acetonitrile, acetone, dichloromethane, hexane:acetone (3:2) and cyclohexane:acetone (3:2). The time of irradiation was varied from 3 to 20 min and the temperature of irradiation was varied from 60% to 120% of the boiling point of each extraction solvent. After extraction, the vessels were allowed to cool to room temperature before opening. The extract was carefully centrifuged and pipetted into a round-bottomed flask. The solvent was evaporated to dryness in a rotary evaporator at 20–30°C and the residue was transferred quantitatively into a 5 mL volumetric flask using ethanol for the synthetic sample and acetonitrile for the LGC6188. Blank experiments were also performed. The extraction efficiencies of solvents were determined in triplicate, PMAE being carried out for 10 min at a temperature of 80% of the boiling point of the solvent (80% of the boiling point of acetone when mixtures of solvents with acetone were used).

Sonication Extraction

The condition for the sonication extraction was optimised as previously report.^[13] Sonication extraction using hexane:acetone (3:2) mixture (20 mL) was performed for the LGC6188, using the same amounts of samples as for the PMAE technique. The durations of extraction were fixed at 40 min. The extract was transferred into a round-bottomed flask and evaporated to dryness by a rotary vacuum evaporator at 30°C and quantitatively transferred into a 5 mL volumetric flask by acetonitrile. All extractions were carried out in triplicate.

Recovery

The certified reference material, LGC6188 was used for the recovery study. Then, 0.25 g of LGC6188 was weighed accurately for determining the recovery of each method of extraction. Analyses of 14 PAHs from the extracts were performed by HPLC using the standard addition method. Each extraction was carried out triplicate.

Extraction of PAHs in Natural Sediment Sample

The sediment sample was taken from the main entrance of Kasetsart University on Paholyotin Road, which is one of the most heavily trafficked in Bangkok was extracted and analysed for PAHs. First, 1 g of the sediment was mixed with 1 g of LGC6188. LGC6188 was used as the internal standard. The mixed sediment samples and the unmixed sediment were extracted by the optimised condition for PMAE using cyclohexane:acetone (3:2) mixture as extracting solvent. The extracted were then analyzed for PAHs in the sediment from Paholyotin Road using HPLC-DAD as previously mentioned.

RESULTS AND DISCUSSION

Optimization of Microwave Conditions

The spiked sediment sample was selected to test the influence of different parameters (type of solvent, time of irradiation and temperature of irradiation) on the total amount of PAHs (Σ PAHs) extracted and also the amount of individual PAHs expressed in mg kg^{-1} of sediment.

Influence of Extraction Solvent

From the principles of microwave heating, it is understood that the choice of the microwave extraction solvent depends upon its ability to absorb microwaves as defined by its dielectric constant. Non-polar solvents cannot absorb microwave energy and despite the fact that they are known

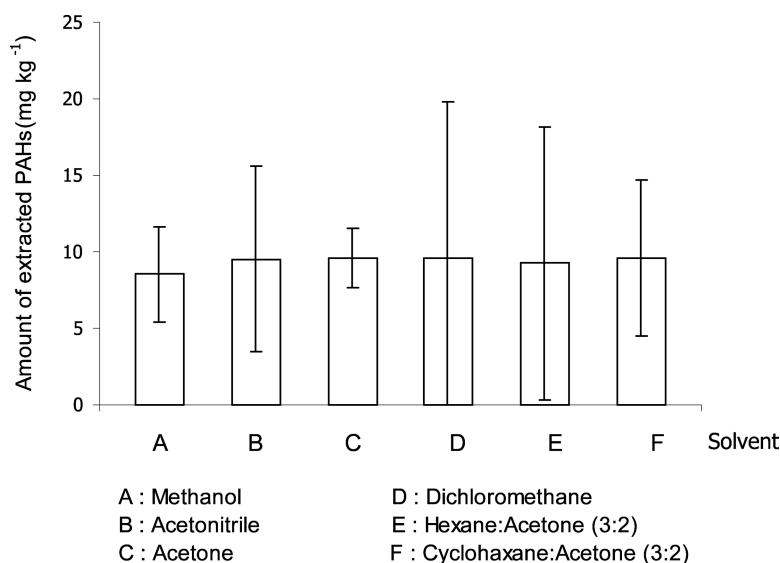


Figure 1: Total amounts of PAHs (Σ PAHs) obtained by PMAE with various solvents.

to be good extraction solvents for aromatic compounds they cannot be used alone. On the other hand, such polar solvents as dichloromethane, methanol, acetone and acetonitrile are also good extraction solvents for aromatic compounds, and therefore the extraction efficiencies of these solvents were studied.

Figure 1 suggests both dichloromethane and the cyclohexane:acetone (3:2) mixture to be suitable microwave extraction solvents. Acetone also gave a good total extraction yield but, because of its polarity, may not be suitable for the extraction of all PAHs in the environmental sample especially those that are highly non-polar. Figure 2 indicates the mixed cyclohexane:acetone and dichloromethane solvents gave good extraction yields of all the individual PAHs.

Influence of Extraction Time

Figure 3 shows the influence of extraction time on PMAE efficiency using dichloromethane and cyclohexane:acetone (3:2) at a temperature fixed at 80% boiling point of absorbing solvent (32°C and 45°C for dichloromethane and cyclohexane:acetone (3:2), respectively).

When dichloromethane was used as the solvent, the total amount of PAHs, Σ PAHs, showed no increase when the duration of extraction was increased from 5 min to 20 min. Subsequently, an extraction time of 5 min was selected when dichloromethane was used as the extraction solvent, since this minimized both energy and time.

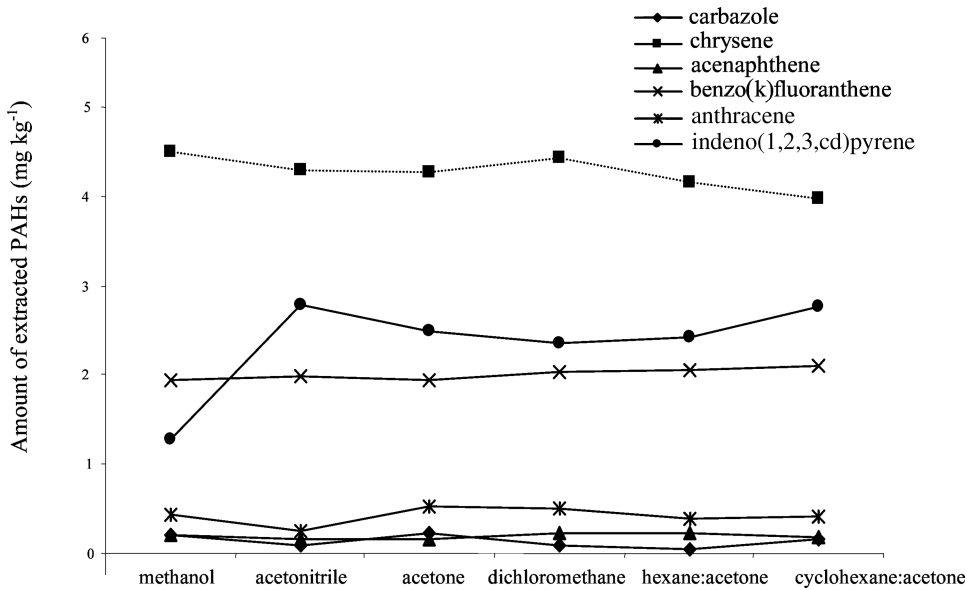


Figure 2: Amounts of individual PAH obtained by PMAE with various solvents.

When cyclohexane:acetone (3:2) was used the total yield of all PAHs, Σ PAHs, was constant only 15 min. It was also found that none of the individual PAHs have been extracted completely in less than 15 min. Subsequently, 15 min was selected as the extraction time when cyclohexane:acetone (3:2) was used for MW extraction.

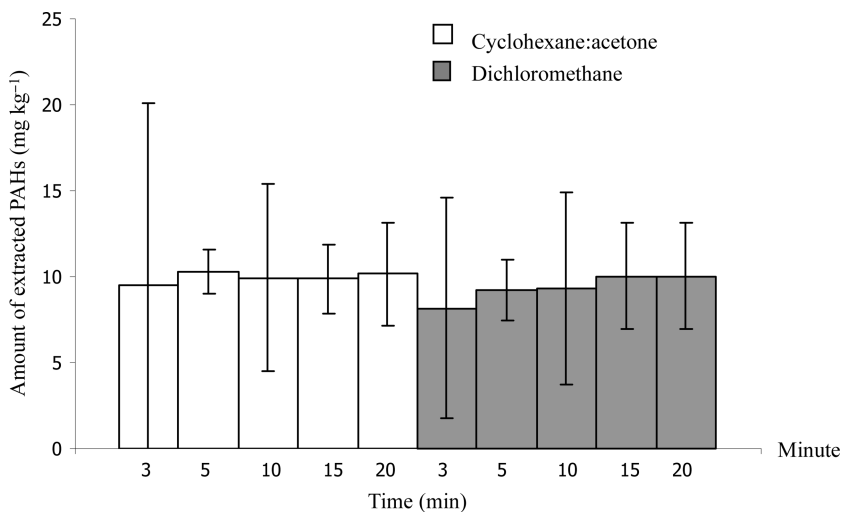


Figure 3: Total amounts of PAHs (Σ PAHs) obtained by PMAE as a function of extraction time using dichloromethane and cyclohexane:acetone (3:2).

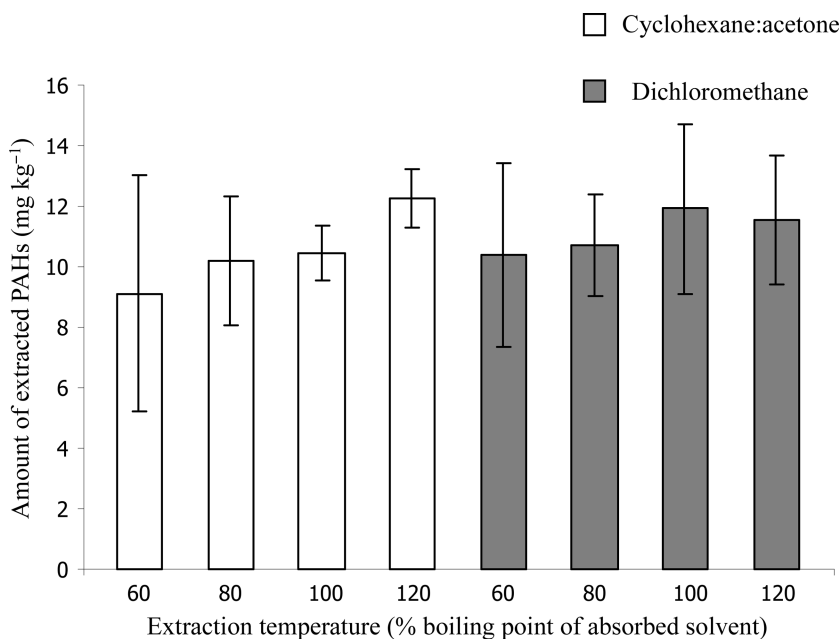


Figure 4: Total amounts of PAHs (Σ PAHs) obtained by PMAE at various temperatures of irradiation using dichloromethane and cyclohexane:acetone (3:2).

Influence of Extraction Temperature

Figure 4 shows the effects of varying the temperature of extraction when dichloromethane and cyclohexane:acetone (3:2) were used with extraction durations of 5 and 15 min, respectively. By using dichloromethane, the total amount of Σ PAHs extracted was largest at the boiling point of the solvent. It was found that lower temperatures fail to extract all of the individual PAHs. The suitable extraction temperatures for PMAE by cyclohexane: acetone (3:2) to be 140% and 160% of boiling point of acetone. Subsequently, the temperature of extraction was chosen as 140% of the acetone boiling point to minimize energy consumption.

Recovery

According to the chromatogram of PAHs in certified reference material (LGC6188), the retention time for acenaphthene, fluorene, phenanthrene, anthracene, fluoranthene, pyrene, benzo[*a*]anthracene, chrysene, benzo[*b*]fluoranthene, benzo[*k*]fluoranthene, benzo[*a*]pyrene, dibenzo[*a,h*]anthracene, benzo[*g,h,i*]perylene and indeno[1,2,3,*cd*] pyrene are 12.88, 15.22, 16.67, 18.01, 19.58, 20.83, 24.07, 24.87, 27.73, 28.91, 30.41, 32.16, 34.90 and 35.82 min, respectively.

Table 1: The % recovery of PAHs from LGC6188 by microwave-assisted and sonication extraction: based on certified value.

| % Recovery: based on certified value of LGC6188 (average \pm % RSD) | | | |
|---|----------------------------|-------------------|-----------------------|
| PAHs | PMAE | | Sonication extraction |
| | Cyclohexane: acetone (3:2) | Dichloromethane | Hexane: acetone (3:2) |
| Acenaphthene | 75.59 \pm 0.71 | 48.43 \pm 15.15 | 17.92 \pm 10.13 |
| Fluorene | 107.52 \pm 11.01 | 107.81 \pm 3.22 | 38.77 \pm 3.86 |
| Phenanthrene | 94.91 \pm 4.99 | 106.46 \pm 2.20 | 69.21 \pm 14.94 |
| Anthracene | 112.89 \pm 2.50 | 93.43 \pm 0.62 | 78.08 \pm 5.34 |
| Fluoranthene | 75.62 \pm 1.30 | 61.07 \pm 5.69 | 58.46 \pm 2.26 |
| Pyrene | 94.35 \pm 0.67 | 69.70 \pm 1.83 | 54.49 \pm 0.81 |
| Benzo(a)anthracene | 89.25 \pm 1.17 | 83.55 \pm 2.43 | 33.27 \pm 5.87 |
| Chrysene | 102.95 \pm 2.01 | 80.32 \pm 2.89 | 89.42 \pm 3.63 |
| Benzo(b)fluoranthene | 124.11 \pm 5.91 | 112.72 \pm 1.02 | 112.48 \pm 0.30 |
| Benzo(k)fluoranthene | 176.71 \pm 1.55 | 125.61 \pm 0.88 | 163.99 \pm 0.98 |
| Benzo(a)pyrene | 91.59 \pm 0.21 | 70.99 \pm 2.08 | 44.79 \pm 8.36 |
| Dibenzo(a,h)anthracene | 78.22 \pm 2.69 | 66.12 \pm 2.13 | 53.02 \pm 1.02 |
| Benzo(g,h,i)perylene | 109.92 \pm 3.39 | 91.76 \pm 1.09 | 82.27 \pm 3.10 |
| Indeno(1,2,3,cd)pyrene | 87.81 \pm 1.50 | 64.49 \pm 0.83 | 61.42 \pm 3.57 |

Table 1 and Figure 5 show PMAE with cyclohexane:acetone (3:2) to give a better % recovery than dichloromethane particularly for PAHs consisting of 4–6 rings. Similarly, sonication extraction (conventional method) with hexane:acetone (3:2) for 40 minutes gave a lower % recovery than either of the PMAEs, particularly for the extraction of PAHs with three or four aromatic rings.

The results shown in Figure 6 confirm PMAE when using cyclohexane:acetone (3:2) under optimum conditions to be the most suitable process for the extraction of PAHs in sediments, due to the high recovery, 96.55%, and the acceptable relative standard deviations.

Application

For the previous discussion, PMAE proved to be a more effective extraction method for PAHs over sonication. The optimised extraction method using PMAE was used to determine the PAHs in sediments from the main entrance of Kasetsart University on Paholyotin Road. To prove that the extraction using the optimized PMAE could give the effectively true concentration of PAHs in sample, the extracted values of each individual PAH from the sediment sample were compared with the values that were expected from the extraction of the mixed sediment sample with LGC6188 (50:50). Table 2 shows the amount of extracted PAHs, calculated from the mixed sediment sample with LGC6188 and unmixed sediment sample. It

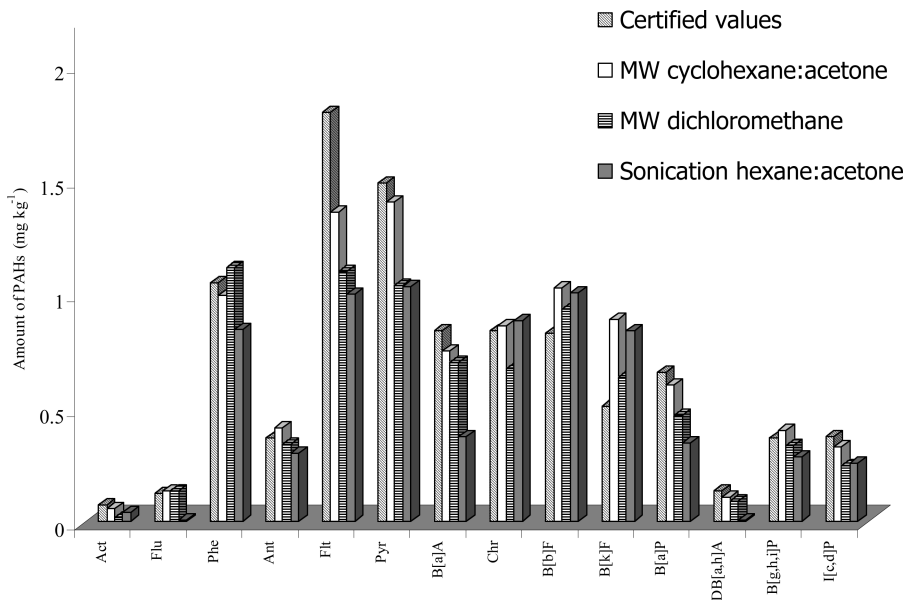


Figure 5: Amounts of individual PAH (mg kg^{-1}) in LGC6188 obtained, (i) from certified values; (ii) by PMAE with cyclohexane:acetone (3:2); (iii) by PMAE with dichloromethane; (iv) by sonication extraction for 40 min with hexane:acetone (3:2).

was found that the amount of almost individual of extracted PAHs in both samples had no significant difference. This proved that the optimised PMAE method is suitable for use as an extraction method for the PAHs in natural samples.

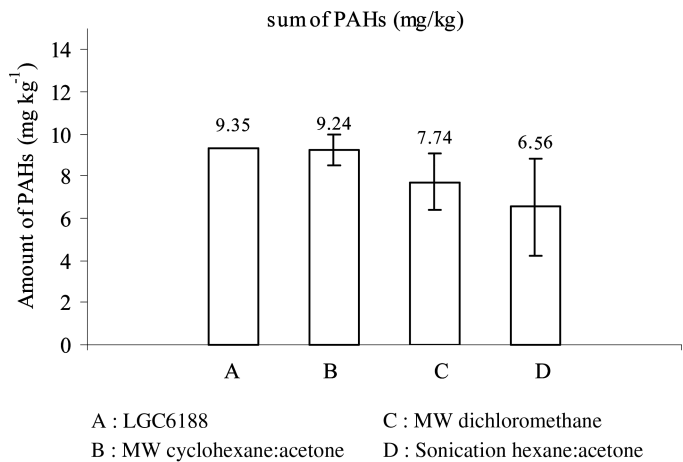


Figure 6: Total amounts of PAHs (Σ PAHs) in LGC6188 obtained, (i) from certified values; (ii) by PMAE with cyclohexane:acetone (3:2); (iii) by PMAE with dichloromethane; (iv) by sonication extraction for 40 min with hexane:acetone (3:2) mixture.

Table 2: Amounts of individual PAH (mg kg⁻¹) in samples obtained from mixed and unmixed sample by PMAE with cyclohexane:acetone (3:2).

| Amounts of individual PAH (mg kg ⁻¹) in samples (average ± % RSD) | | | |
|---|--------------|------------------|--------------------------|
| PAHs | Mixed sample | Unmixed sample | % different ^b |
| Acenaphthene | 0.102 ± 1.49 | 0.100 ± 1.55 | 1.15 |
| Fluorene | 0.030 ± 1.40 | 0.030 ± 1.63 | 0.029 |
| Phenanthrene | 0.081 ± 1.26 | 0.084 ± 3.38 | 4.059 |
| Anthracene | 0.070 ± 2.21 | 0.076 ± 2.08 | 8.748 |
| Fluoranthene | 0.230 ± 1.37 | 0.236 ± 1.27 | 2.771 |
| Pyrene | 0.056 ± 1.01 | 0.054 ± 0.99 | 2.567 |
| Benzo(a)anthracene | 0.001 ± 2.80 | BDL ^a | — |
| Chrysene | 0.004 ± 0.77 | 0.004 ± 0.77 | 4.944 |
| Benzo(b)fluoranthene | 0.016 ± 0.30 | 0.016 ± 0.20 | 1.47 |
| Benzo(k)fluoranthene | 0.004 ± 0.05 | 0.004 ± 0.98 | 2.268 |
| Benzo(a)pyrene | 0.027 ± 2.63 | 0.026 ± 1.89 | 6.114 |
| Dibenzo(a,h)anthracene | 0.003 ± 2.23 | BDL | — |
| Benzo(g,h,i)perylene | 0.007 ± 3.82 | BDL | — |
| Indeno(1,2,3,cd)pyrene | 0.007 ± 5.55 | BDL | — |

^aBDL = below detection limit.

$$^b\% \text{different} = \frac{(\text{mixed} - \text{unmixed})}{\left(\frac{\text{mixed} + \text{unmixed}}{2}\right)} \times 100.$$

CONCLUSION

The conditions for extracting PAHs from sediments by PMAE have been studied and optimized. Optimization of all parameters (type of extraction solvents, times of irradiation and temperatures of irradiation) was important for efficient extraction. The most efficient PMAE was obtained with 20 mL of cyclohexane:acetone (3:2) for 15 min at temperature equal to 140% boiling point of acetone. Under this condition for the extracted certified reference material recovery was 96.55%. The reproducibility was also satisfactory (relative standard deviation less than 15%). The PMAE was successfully applied for the extraction of PAHs in natural sediment sample. It can be concluded from the experimental results that PMAE provides a good alternative to sonication extraction for the removal of PAHs from sediments. Its main advantages in comparison to the sonication extraction are the reduction of the volume of volatile organic solvent, the reduction in extraction time, and the lower consumption of energy.

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REFERENCES

1. Perez, S.; Ferry, I.; Hennion, M.; Barcelo, D. Isolation of polycyclic aromatic hydrocarbons from natural sediments and sludge reference material by an anti-fluorene immunosorbent followed by liquid chromatography and diode array detection. *Anal. Chem.* **1998**, *70*, 4996–5001.
2. Pino, V.; Ayala, J.H.; Afonso, A.M.; Gonzalez, V. Ultrasonic micelle extraction of polycyclic aromatic hydrocarbons from marine sediments. *Talanta* **2001**, *54*, 15–23.
3. Guerin, T.F. The extraction of aged polycyclic aromatic hydrocarbons (PAHs) residues from a clay soil using sonication and Soxhlet procedure: a comparative study. *J. Environ. Monit.* **1999**, *1*, 63–67.
4. Shu, Y.Y.; Lao, R.C.; Chiu, C.H.; Turle, R. Analysis of polycyclic aromatic hydrocarbons in sediment reference materials by microwave-assisted extraction. *Chemosphere* **2000**, *41*, 1709–1716.
5. Song, Y.F.; Jing, X.; Fleischmann, S.; Wilke, B.M. Comparative study of extraction methods for the determination of PAHs from contaminated soils and sediments. *Chemosphere* **2002**, *48*, 993–1001.
6. Lopez-Avila, V.; Young, R.; Beckert, W.F. Microwave-assisted extraction of organic compounds from standard reference soils and sediments. *Anal. Chem.* **1994**, *66* (7), 1097–1106.
7. Noordkamp, E.R.; Grotenhuis, J.T.C.; Rulkens, W.H. Selection of an efficient extraction method for the determination of polycyclic aromatic hydrocarbons in contaminated soil and sediment. *Chemosphere* **1997**, *8* (5), 1907–1917.
8. Kawata, K.; Akiko, T.; Hiroaki, Y.; Tsuyoshi, I.; Akio, Y.; Takayuki, S. Determination of semivolatile organic compounds in environmental samples by gas chromatography/mass spectrometry after extraction by cyclic stream distillation. *J. AOAC Int.* **2003**, *86* (2), 246–256.
9. Tomaniova, M.; Hajslova, J.; Pavelka Jr, J.; Kocourek, V.; Holadova, K.; Klimova, I. Microwave assisted solvent extraction—a new method for isolation of polynuclear aromatic hydrocarbons from plants. *J. Chromatogr. A.* **1998**, *827*, 21–29.
10. Shu, Y.Y.; Lai, T.L. Effect of moisture on the extraction efficiency of polycyclic aromatic hydrocarbons from soils under atmospheric pressure by focused microwave-assisted extraction. *J. Chromatogr. A.* **2001**, *927*, 131–141.
11. Pastor, A.; Vazquez, E.; Ciscar, R.; Guardia, M. Efficiency of the microwave-assisted extraction of hydrocarbons and pesticides from sediments. *Anal. Chem.* **1997**, *344*, 241–249.
12. Numata, M.; Yarita, T.; Aoyagi, Y.; Takatsu, A. Microwave-assisted system distillation for simple determination of polycyclic aromatic hydrocarbons and organochlorine pesticides in sediments. *Anal. Chem.* **2003**, *75*, 1450–1457.
13. Bangkedphol, S. *Effective extraction method for the determination of polycyclic aromatic hydrocarbons (PAHs) in environmental samples*; M.Sc. Thesis. Kasetsart University: 2004; 88–91.