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Determination of Methyl Tert-Butyl Ether in Fish Tissue by Gas Chromatography after Using Ultrasonic Extraction and Headspace Liquid Phase Micro Extraction

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Abstract
In the present study a combined analytical method involving ultrasonic extraction (USE) and headspace liquid phase micro extraction (HS- LPME) was used to extract methyl tert-butyl ether (MTBE) in fish tissue for analysis by gas chromatography (GC). Extraction of MTBE from 2 g of fresh fish tissue was performed by ultrasonication for 10 min (in 25 ºC after 3.5 h maceration) in the glass barrel that was filled with water and sealed. Headspace solvent microextraction was done for the extraction of MTBE after centrifuge of sonicated sample. The optimized method (under the optimal experimental conditions: ultrasonic extraction time: 10 min, ultrasonic extraction temperature: 25 °C, fish particle size: a particle of 2 grams, media: distilled water, maceration time: 3.5 h and centrifuge of sonicated sample: 2500 rpm in 15 ˚C for 10 min) had good linearity (R^2 >0.99) over the range 0.5- 50 µg g^{-1}, and showed satisfactory level of precision, with RSD values in the range of 1.1 to 9.7 %. The LOD about 0.2 µg g^{-1} was obtained.

Keywords
MTBE, Fish Tissue, Utrasonic Extraction, HS-LPME Extraction

1.INTRODUCTION

Methyl tert- butyl ether (MTBE) is the most commonly used fuel oxygenate, and is added to gasoline to enhance the octane number of gasoline. Because a large amount (8.8×10^9 Kg in 1997) of MTBE has been produced in the USA, and because a number of investigations have shown that MTBE may pollute the aquatic and atmospheric environment, its environmental fate has led to cancer. MTBE is present at a relatively high concentration in some urban air due to its use as a gasoline additive. The high solubility of MTBE in water, combined with its high concentration in some types of gasoline, can result in high concentrations of MTBE in surface water, ground water and storm water [1, 2]. In fact, MTBE was reported as the second most frequently detected chemical (after chloroform) in shallow ground water [3, 4]. The US environmental protection agency (EPA) has set the advised level for taste and odor for MTBE in water at 2.0 and 2.5 µg L^{-1} respectively [5].

Analytical methods were used to determine MTBE including: purge and trap, head space (HS) or direct aqueous injection (DAI) on to gas chromatography (GC) [6-12]. A few reports have been published that describe the use of solid phase micro extraction (SPME)-gas chromatography to determine MTBE concentrations in water [13- 18]. Also, there are a few reports that describe the use of headspace- liquid phase micro extraction to determine MTBE concentrations in water [19-21]. In another report, the situ solvent formation microextraction technique was used for pre-concentration and separation of MTBE from water samples in order to trace analysis by GC-FID [22]. In headspace- liquid phase micro extraction (HS-LPME) system (Fig. 1), extraction of analytes occurs via suspending a micro-liter drop of a proper non-aqueous solvent from the tip of a micro-syringe that is located in the headspace of a stirred aqueous sample solution thermostated at a given temperature for a pre-set extraction time. The drop remained at the tip of the micro-syringe throughout the extraction period and then was retracted back into the needle and injected into a GC for the identification and quantification of the extracted analytes [23].

In the present study, a procedure for extraction and determination of MTBE in fish tissue has been developed. Fish tissue is an important medium for study purposes due to its importance as a food source and as an indicator of the quality of the environment [24]. In this procedure was used from HS-LPME to determine MTBE in sonicated aqueous sample.

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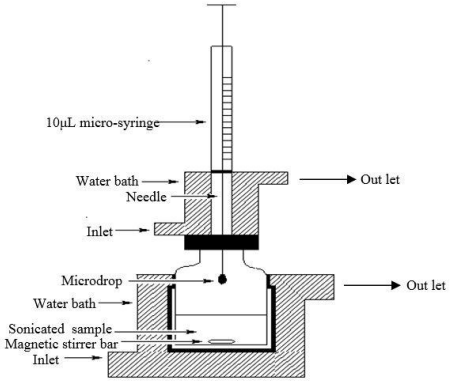

Fig 1. Diagram showing HS- LPME method

A combined analytical method involving ultrasonic extraction and headspace solid- phase microextraction was used for the determination of chlorinated pesticides (CPs) and polychlorinated biphenyls in bird livers [25].

2.EXPERIMENTAL

2.1.Instrumentation

All of the separation stage was carried out on a GC Buck Scientific model 910 gas chromatograph with a flame ionization detector (FID). The separation stage performed on a 30 m × 0.25 mm i. d. capillary column with 0.25 μm AT- WAX coating (Altech). Helium was used as carrier gas with column head pressure 14 Psi. The injection port was held at 250 ˚C and used in the splitless mode. Oven temperature programming was used to facilitate separation with an initial oven temperature of 43 ˚C (held for 5 min) ramping at a rate of 20 ˚C/ min to a temperature of 200 ˚C (held for 10 min). Detector oven was held at 250 ˚C. A 10μl micro-syringe with an angled- cut needle tip (S. G. E., Australia) was used for headspace solvent micro extraction and injection into the GC system. HS-LPME was performed in a 10 mL vial with a silicon rubber septum the temperatures of the droplet and vial were controlled thermostically using two water jackets. Two circulating water baths (HAAK DC 30) were used for adjusting the temperatures of syringe needle and sample solutions with an accuracy of ±0.1 ˚C and a magnetic stirrer was used (MR 3001 K, heidolph, Germany) Fig. 1 shows the apparatus used for the HS-LPME.

An ultrasonic water bath (ultra wave limited Cardiff CF2 1 YY UK) and a 10 mL vial with a silicon rubber septum filled with water were used in the ultrasonic extraction procedure. The generator of this apparatus has a frequency of 30 KHz. Also was used from a centrifuge set (MSE MISTRAL 2000R) for centrifuge of ultrasonic aqueous sample.

2.2.Reagents

Reagent grade benzyl alcohol (Fluka, Buchs, Switzer land), ethanol, MTBE, NaCl, HNO_3 (Merck, Darmstadt, Germany) were used as received. Ethanolic stock standard solutions of MTBE (10 mg mL^{-1}) were stored at 4 ˚C. Working standard solutions were prepared daily by diluting stock standard solutions with ethanol to the required concentrations. The extractant was an organic solvent containing a fixed concentration of impurity. Doubly distilled water was prepared daily.

2.3. Procedure

2.3.1. Ultrasonic extraction of MTBE from fish tissue into water

Before HS-LPME, MTBE was extracted from the fish tissue to water by ultrasonic extraction. It was achieved by ultrasonic extraction in the glass barrel that was filled with water and sealed. The sonicated aqueous sample (in the sealed and filled glass barrel) was then centrifuged and analyzed by HS-LPME- GC- FID.

About 2 grams of fresh fish tissue were weighed and for 1 hour was macerated into 50 mL water (for exit probable MTBE from the fish tissue). Then, 10 μL from 10 mg mL^{-1} MTBE in ethanol was injected in the fish tissue with micro-syringe. Fish samples were placed into the 10 mL vial that were filled with water and sealed with a silicon rubber septum. The samples were macerated for a given time. Then the samples were sonicated for a given time and in a given temperature. Sealed vials containing sonicated samples were centrifuged, later the syringe was then lowered and the needle passed through the vial septum (containing centrifuged samples) and 6 mL of aqueous centrifuged samples were drown up into the syringe and removed to another vial (content of NaCl and magnet stirrer) for HS-LPME. This vial was sealed with silicon and rubber septum.

2.3.2. Headspace solvent micro extraction of MTBE from sonicated aqueous sample into a benzyl alcohol single drop

The 10μL micro-syringe containing the appropriate solvent was clamped above the vial containing sonicated aqueous sample solution. The micro-syringe was then lowered and its needle passed through the vial septum until the tip of the needle was 5- 15 mm above the surface of the stirred sample solution (depending on the sample volume). Then, the syringe plunger was pressed so that the solvent drop was exposed to the headspace of the stirring sample solution for pre-set extraction time. After headspace extraction, the plunger was withdrawn and the micro drop was

retracted into the micro-syringe. The syringe was then removed from the top of the sonicated sample vial and was injected into the gas chromatograph for analysis. Parameters such as extraction time (10 min), nature of extraction solvent (benzyl alcohol), size of microdrop (2μ L), sample volume (6 mL), stirring rate (1000 rpm), ionic strength (4 mol L^{-1} NaCl), sonicated sample solution temperature (35 ˚C), micro-syringe needle temperature (-4 ˚C) were had been previously studied and optimized [21]. Dynamic linear range (calibration curve) for MTBE in HS-LPME step was calculated using 10 spiking levels of MTBE in double distilled water in the concentration range of 40-100 $\times 10^3$ ng mL^{-1}.

3.RESULTS AND DISCUSSIONS

3.1.Ultrasonic extraction development

The main parameters affecting the ultrasonic extraction process, such as sonication time, sonication temperature, particle size, concentration of HNO3 in medium of ultrasonic extraction, time length of maceration before sonication and centrifuge of sonicated sample were investigated and optimized. In the final stage the linear range and the precision of the method were studied.

Ultrasonic waves are generated by vibrations that act through expansion and compression cycles on bubbles preexisting in aqueous solutions. These cycles can lead to an implosive collapse of such cavities resulting in an increase in the temperature and pressure at microsites. Occasionally, solvent or solute molecular fragmentation into reactive free radicals (such as hydrogen atoms and $OH^{\cdot}$ radicals from water vapour) may occur during sonication. These effects, however, do not significantly alter the chemical properties of sample or native organic matter. Therefore, sonication disrupts aggregates both by formation of collapsing bubbles and friction between and within aggregates.

Optimum performance of the method can be achieved by appropriate optimization of these parameters. All quantifications made in this study were based on the percentage of extraction of MTBE from fish tissue.

The percentage of extraction depends on factors, such as analyte- to- matrix interaction and the capability of the ultrasonic processor used to dissipate ultrasound. Thus, quantitative extraction was reported for biological matrices but it was more difficult for typical inorganic materials. Likewise, the use of high intensity ultrasonic processors should be attempted in order to achieve good performance for extraction. To optimize each parameter, the results from at least three replicate analyses were averaged.

3.1.1. Ultrasonic extraction time and temperature

Temperature of the extraction media increases with increasing of sonication time. Usually, as the temperature increases up to 50 ˚C, the extraction efficiency will be increased as a result of the large number of cavitation nucleus formed. As the temperature approaches the boiling point of the liquid, infective sonication occurs as a result of a decrease in surface tension and an increase in vapor pressure within the micro bubble, which in turn cause the damping of the shock wave. In ultrasonic extraction, MTBE with a composition of fish tissue are extracted simultaneously that presence of fish tissue composition prevent from extraction of MTBE in HS-LPME step.

The extraction efficiency of a 0.05 mg g^{-1} MTBE in fish tissue as a function of ultrasonic extraction time and temperature are illustrated in Fig. 2. According to Fig. 2, further extractions were performed for 10 min in 25 ˚C.

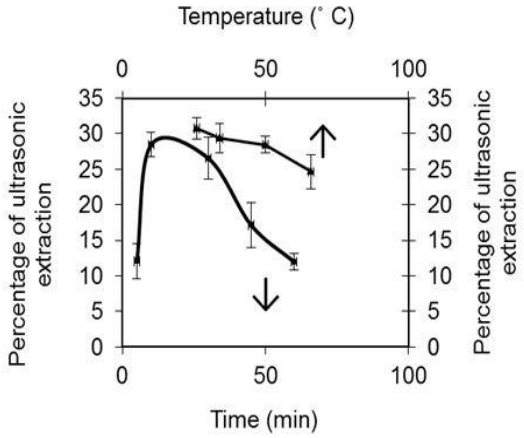

Fig 2. effect of ultrasonic extraction time (■) and temperature (▲) on extraction efficiency. Conditions for ultrasonic extraction time: amount of MTBE, 0.05 mg g^{-1}; number of particles of fish tissue, a 2 gram particle; ultrasonic extraction medium, distillate water; without maceration time and centrifuge. Conditions for temperature experiments as before, except 10 min for ultrasonic extraction time and varying temperature.

3.1.2. Number of particles of fish tissue

The effect of the number of particles of fish tissue on the extraction of the MTBE by ultrasonic extraction was investigated as follows. A set of experiments was performed with injection of 10 μL from 10 mg mL^{-1} MTBE in Ethanol in a particle of fish tissue (2 g) and in two particles of fish tissue (1 g) and in four particles of fish tissue (0.5 g). The results are shown in Fig. 3. The presence of a composition of fish tissue was increased sharply with an increase in the number of particles of fish tissue. Therefore, the extraction of MTBE in HS-LPME for analysis was decreased.

3.1.3. Ultrasonic extraction media: Effect of acid concentration

The use of an acidic diluent combined with the ultrasonic action makes the solid particles more flocculent, hence helping the extraction process. Among the acids employed as diluent, HNO_3 shows an enhanced performance due to its oxidant

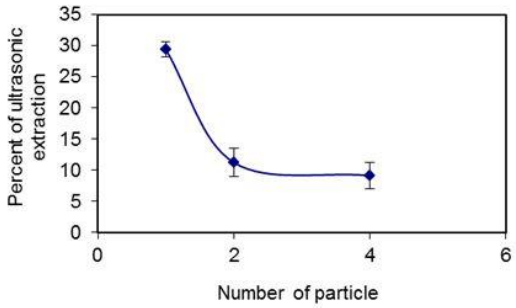

Fig 3. Effect of number of particles of fish tissue on ultrasonic extraction efficiency. Conditions for ultrasonic extraction: time, 10 min; temperature, 25 ˚C; amount of MTBE 0.05 mg g^{-1}; ultrasonic extraction medium, double distillated water; with out maceration time and centrifuge.

properties. In this study, the HNO_3 concentration varied from 0 to 1 M. From Fig. 4, it can be concluded that with increase in acid concentration, the presence of a composition of fish tissue will increase. Also, in the presence of the acid, extracted MTBE was hydrolyzed to alcohols (methanol, tert- butyl alcohol). Therefore, the extraction of MTBE in HS-LPME for analysis was decreased. Thus, doable distillated water was chosen as the extraction medium for the further studies.

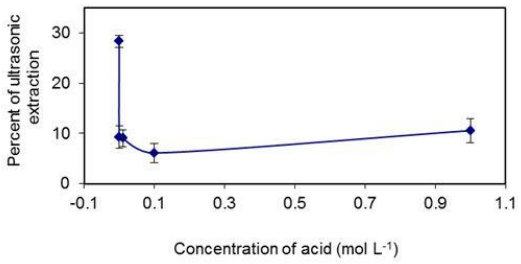

Fig. 4. Effect of acid concentration on ultrasonic extraction efficiency. Conditions for ultrasonic extraction: time, 10 min; temperature, 25 ˚C; amount of MTBE, 0.05 mg g^{-1}; number of particles of fish tissue, a 2 gram particle; with out maceration time and centrifuge.

3.1.4. Effect of time length of maceration before sonication

The time length of maceration in water (filled with water and sealed with rubber and silicon septum vial) affected ultrasonic extraction efficiency and the precision of extraction (Fig. 5). Fish tissue samples were macerated at room temperature for 0 to 24 h. With an increase in the of maceration time, the diffusion of water in fish tissue increased. Therefore, the extraction of MTBE was increased. But in longer times, the interference of the presence of a composition of fish tissue as was observed. Maximum extraction occurred in 3.5h, thus further extractions were performed with 3.5 h maceration time.

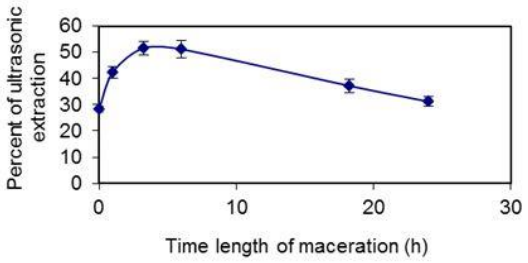

Fig 5. Effect of time length of maceration before sonication on ultrasonic extraction efficiency. Conditions for ultrasonic extraction: time, 10; temperature,25 ˚C; amount of MTBE 0.05 mg g^{-1}; number of particles of fish tissue, a 2 gram particle; with out centrifuge.

3.1.5. Effect of centrifuge after sonication

The interference of presence of fish tissue composition that is insoluble in water was eliminated by using centrifuge. The effect of the centrifuge of sonicated sample on the extraction efficiency of the MTBE was investigated as follows. A set of experiments was performed 1) ultrasonic extraction after 3.5h maceration time without centrifuge 2) ultrasonic extraction after 3.5h maceration time then sonicated sample without opening of seal was centrifuged with 2500 rpm in 15 ˚C for 10 min 3) ultrasonic extraction after 24h maceration time without centrifuge 4) ultrasonic extraction after 24h maceration time then sonicated sample with out opening of seal was centrifuged with 2500 rpm in 15 ˚C for 10 min. The results were shown in Fig. 6. Maximum efficiency occurred with 3.5h maceration time before sonication and centrifuge after sonication. Thus, in the present work these conditions were adopted for further studies.

3.1.6. Evaluation of the method performance

Dynamic linear range (calibration curve) was calculated using 5 spiking levels of MTBE in the range of 0.5- 50 μg g^{-1}. For each spiking level three replicate analyses were performed. The calibration curve is given in Fig. 7. The regression equation and correlation coefficient (in parenthesis) were A_r= 0.0064 C + 0.1125 (R^2 =0.9992) where A_r is relative peak area and C is the amount of MTBE (μg g^{-1}). This method revealed good reproducibility with R.S.D. values in the range of

1.1 to 9.7 %.

The limit of detection (LOD) of the proposed method for the determination of MTBE was studied under the optimal experimental conditions. LOD obtained from $C_{LOD}= KS_b/m$ where K=3, S_b is the standard deviation of six replicate blank measurements and m is the slope of the calibration curve. The LOD obtained was 0.2 μg g^{-1}.

In order to test the applicability of the proposed method to real samples, a fish tissue sample was obtained directly from fish pisciculture pool and tested by the recommended procedure (Table 1).

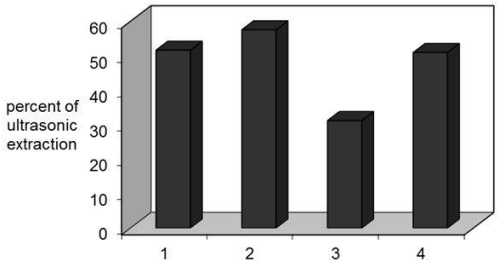

Fig 6. Effect of centrifuge after sonication. Conditions for ultrasonic extraction: time, 10 min; temperature , 25 ˚C; amount of MTBE , 0.05 mg g^{-1};; number of particles of fish tissue, a 2 gram particle.

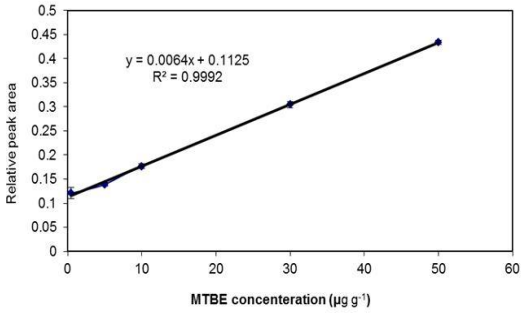

Fig. 7. Calibration curve of MTBE in optimal conditions (optimal conditions of ultrasonic extraction: ultrasonic extraction time: 10 min, ultrasonic extraction temperature: 25 °C, fish particle size: a particle of 2 grams, media: distilled water, maceration time: 3.5 h and centrifuge of sonicated sample: 2500 rpm in 15 ˚C for 10 min. optimal conditions of headspace liquid phase micro extraction: extraction time (10 min), nature of extraction solvent (benzyl alcohol), size of microdrop (2μL), sample volume (6 mL), stirring rate (1000 rpm), ionic strength (4 mol L^{-1} NaCl), sonicated sample solution temperature (35 ˚C), micro-syringe needle temperature (-4 ˚C)).

Table 1. Determination of MTBE in fish tissue sample

Sample	Concentration (μg g^{-1}) added	found	Recovery (%)	RSD (%) N=3
1[a]	-	-	-	-
2[b]	-	-	-	-
3[b]	25.0	25.6	102.4	3.1
4[b]	15.0	14.1	94.0	7.0
5[b]	35.0	34.0	97.1	5.3

a. Without maceration. b. Aftter one hour maceration into 50 mL water.

4. CONCLUSIONS

In this study a combined analytical method involving ultrasonic extraction (USE) and headspace liquid phase micro extraction (HS-LPME) was used to extract MTBE from fish tissue for analysis by gas chromatography- flame ionization detector (GC-FID). This method, using very low organic solvent (1 -2 μL) and sample solution volumes (1- 10 mL) was described. There is no need for delicate and expensive apparatuses for the proposed method. The LOD about 0.2 μg g^{-1} was obtained. Although the LOD is not ideal, but can be improved with mass spectrometry detector.

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تعیین مقدار متیل ترشیو بوتیل اتر در بافت ماهی با کروماتوگرافی گازی پس از استخراج به روش التراسونیک و میکرواستخراج با فاز مایع از فضای فوقانی

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چکیده

در این کار تحقیقاتی یک روش تجزیه‌ای ترکیبی شامل استخراج با التراسونیک (USE) و میکرواستخراج با فاز مایع از فضای فوقانی (HS-LPME) برای استخراج متیل ترشیو بوتیل اتر (MTBE) موجود در بافت ماهی به منظور آنالیز با دستگاه کروماتوگرافی گازی (GC) استفاده شد. استخراج MTBE از ۲ گرم بافت تازه ماهی با التراسونیک کردن به مدت ۱۰ دقیقه (در دمای ۲۵ سانتی‌گراد بعد از ۳/۵ ساعت خیساندن) در داخل یک ظرف شیشه‌ای پر و مهر و موم شده، انجام شد. میکرواستخراج حلالی از فضای فوقانی به منظور استخراج MTBE از نمونه بدست آمده پس از سانتریفوژ، انجام شد. روش بهینه شده (شرایط بهینه آزمایشات عبارتند از: مدت زمان استخراج به روش التراسونیک: ۱۰ دقیقه، دمای استخراج در مرحله التراسونیک: ۲۵ درجه سانتی‌گراد، اندازه قطعه ماهی: قطعات ۲ گرمی، محیط مرحله استخراج به روش التراسونیک: آب مقطر، زمان خیساندن: ۳/۵ ساعت و شرایط سانتریفوژ نمونه التراسونیک شده: در دمای ۱۵ سانتی‌گراد به مدت ۱۰ دقیقه با سرعت ۲۵۰۰ دور در دقیقه) همبستگی خطی خوبی (Rc۰/۹۹) در ناحیه ۰/۵ تا ۵۰ $\mu g\ g^{-1}$ دارد و سطح رضایت بخشی از دقت با مقادیر RSD ۱/۱ تا ۹/۷ درصد را نشان می‌دهد. مقدار حد تشخیص (LOD) ۰/۲ $\mu g\ g^{-1}$ بدست آمد.

واژه‌های کلیدی

متیل ترشیو بوتیل اتر; بافت ماهی ؛ استخراج اولتراسونیک؛ میکرواستخراج با فاز مایع از فضای فوقانی