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APPLICATION OF THE SOLID PHASE MICROEXTRACTION (SPME) AND GAS CHROMATOGRAPHY (GC, GC/MS) IN FOOD ANALYSIS

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Key words: volatile organic compounds, food analysis, sample preparation, solid phase microextraction (SPME), GC, GC/MS

In the current contribution, the application of the Solid Phase Microextraction (SPME) as a modern sample preparation method was discussed. Based on theoretical and practical aspects of the SPME method, a comparison of extraction efficiency of aromatic hydrocarbons (benzene, toluene, ethylbenzene, m,p-xylene (BTEX)) as testing substances was performed. The various SPME coated fibers: commercial (polydimethylsiloxane – PDMS, polyacrylate – PA) and home-made (etoxy-polydimethylsiloxane – PDES, polyurethaneacrylate, fused silica, and fused silica after etching of hydrogen fluoride acid) were compared in these experiments. The extraction efficiency was displayed as the extracted mass for BTEX after extracting of standard solution with various SPME fibers. The most efficient extraction (adsorption on the fiber surface) is for the etoxy-polydimethylsiloxane-coated fibers after drying at 200°C than for other tested fibers. In addition, the possibility of SPME use (with the new PDES fibers) in the food and natural products analysis (wine, candies, herbs) was discussed. Many advantages, as well as disadvantages of the SPME method are described in details.

INTRODUCTION

In the current paper, the theoretical and practical aspects including advantages as well as drawbacks of the solid phase microextraction (SPME) use as a sample preparation method will be discussed in details. In addition, the application of SPME in the food and natural products analysis (wine, candies, herbs) will be presented.

Sample preparation methods include physical, physicochemical and chemical methods of the compounds isolation from a sample. In this area, the leading ones are physicochemical methods that base on the phase boundary effects: liquid-liquid, liquid-solid, gas-liquid, gas-solid phase. The first classical technique of extraction was liquid-liquid extraction (LLE), followed by solid phase extraction (SPE) and microwave extraction (ME), and finally supercritical fluid extraction (SFE) [Poole *et al.*, 1990; Buszewska *et al.*, 1998]. One of the most frequently applied methods based on the phase boundary processes is static and dynamic headspace (HS) and purge and trap technique (PT) [Poole *et al.*, 1990]. Both of these techniques are applied to isolate and determine volatile organic contaminations in liquid and solid food matrices. An alternative to HS and SPE is modern, solid phase microextraction (SPME). It has been widely propagated by Pawliszyn and other authors until 1989 [Arthur & Pawliszyn, 1990; Zhang & Pawliszyn, 1993].

During the SPME process, an equilibrium between the coated fiber and sample matrix is observed. At equilibrium, the distribution of the organic compound between the solid phase and the aqueous phase has been shown by equation (1) according to the extraction theory [Arthur & Pawliszyn, 1990; Zhang & Pawliszyn, 1993; Pawliszyn, 1997; Ligor & Buszewski, 1997]:

$$k = C_s/C_a \quad (1)$$

where C_s is the concentration of the organic compound in the solid phase, C_a is the concentration of organic compound in aqueous phase, and k is the partition coefficient for solid phase/aqueous phase system.

The k values have been calculated for many environmental compounds and defined efficiency of pre-concentration of organic compounds from water sample.

During the SPME extraction process, especially for reproducible results, some variables must be controlled. These include sample volume, sample agitation, the sampling method (headspace or immersion), sample pH, ionic strength, extraction time, and temperature.

In SPME, a fused silica fiber is coated with a sorbent (which is mounted on a modified chromatographic syringe) to extract sample components and pass analytes directly into gas chromatography (GC) or high performance liquid chromatography (HPLC) [Arthur & Pawliszyn, 1990; Zhang & Pawliszyn, 1993; Pawliszyn, 1997]. The typical SPME manual holder is presented in Figure 1. A scanning electron micrograph picture of polymer-coated film

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of SPME fiber [Lin *et al.*, 1997] confirmed the uniformity of fiber coating.

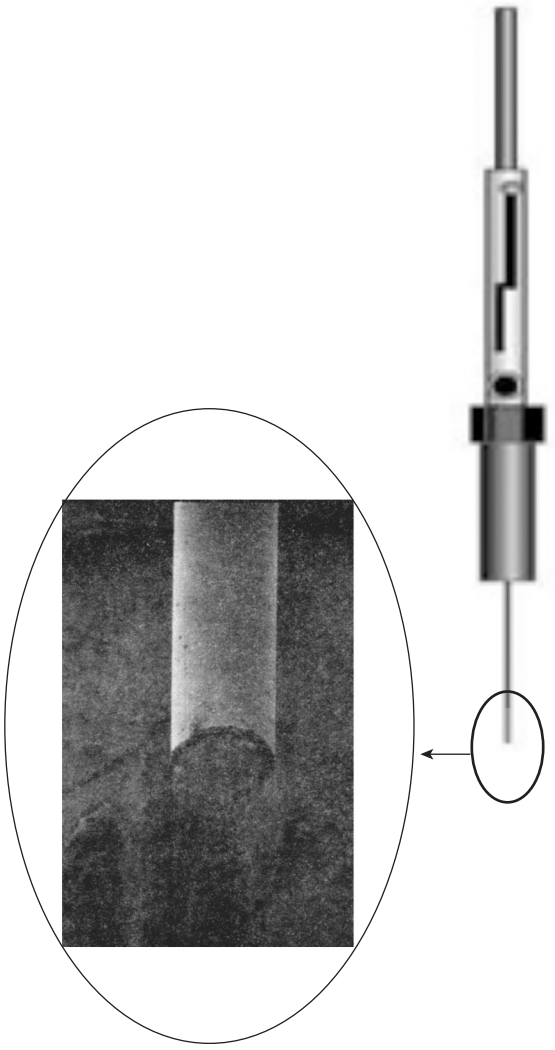


FIGURE 1. SPME apparatus offered by Supelco Inc. with the picture of a scanning electron micrograph of polymer-coated film of SPME fiber.

During the SPME extraction a short length of fused silica fibers coated with adsorbents or absorbets are used. At present some commercial SPME fiber coatings are available, *e.g.* polydimethylsiloxane (PDMS – 100 μm, 30 μm, 7 μm), polydimethylsiloxane/divinylbenzene (PDMS/DVB – 65 μm, 60 μm), polydimethylsiloxane/Carboxen (PDMS/Carboxen – 75 μm), Carbowax/divinylbenzene (CW/DVB – 65 μm), Carbowax/templated resin (CW/TPR – 50 μm), polyacrylate (PA – 85 μm).

There are nonpolar, bipolar and polar fiber coatings. For selective extraction new stationary phases are however needed. For this reason, analytical studies of new, selective SPME coatings on contaminants in various matrices (environmental samples, food, juice, beverages, *etc.*) are currently underway [Ligor *et al.*, 1999].

Very satisfactory results of isolation of natural and harmful compounds from alcoholic beverages, herbs and spices have been obtained by the SFE technique [Vejrosta *et al.*, 1998]. However, solid-phase extraction with some modification, *i.e.* Empore™ or Speedisk™ and solid-phase microextraction (SPME), are generally preferred for alcoholic beverages analysis especially for organic compounds responsible for colours and tastes of wines [Vejrosta *et al.*, 1998; Jeleń *et al.*, 1998; Gibson, 1997; De la Calle Garcia *et al.*, 1997; Ligor & Buszewski, 2001; Vas, 1996].

In this article, the applications of polymer phases: commercial (polydimethylsiloxane – PDMS, polyacrylate – PA) and home-made (etoxy-polydimethylsiloxane – PDES, polyurethaneacrylate, fused silica, and fused silica after etching of hydrogen fluoride acid), which were used as fibers coatings for SPME, are discussed in details. The extraction efficiency for BTEX after extracting a standard solution with various SPME fibers was displayed. The commercial (PDMS, PA) as well as home-made (PDES) coated fibers could be successfully used in the food and natural products analysis (wine, candies, herbs). These applications of SPME will be presented as advantageous compared to other extraction techniques. This study confirms that SPME is a very useful method for qualitative and quantitative analyses of volatile organic compounds in various food and beverages samples.

MATERIAL AND METHODS

Apparatus. The Hamilton syringe 7000 series with Chaney adaptor (Hamilton Co., Reno, NV, USA) after special modification was used for testing new types of coated fibers. Home-made modification of syringe consisted in cutting a piston short and after that, a 12 cm fiber of fused silica coated with 1 cm of stationary phase was connected to the fused silica tubing by polyimide resin. This fused silica tubing was also connected with the piston by polyimide resin. The adsorption was carried out in vials by dipping in water containing BTEX. The sample was completely stirred by a magnetic stirrer in the adsorption time.

The desorption and chromatographic analysis was performed by GC (Fisons 8160, Fisons Instruments, Milano, Italy). Carrier gas: helium (99.999 %) – pressure on the column head 20 kPa. The temperature of injector split-splitless was 200°C. The FID detector temperature of 250°C was used. The RTX 200 (Restek, Bellefonte, PA, USA) column (30 m × 0.53 mm × 0.25 μm) was used. Oven temperature programme was as follows: 40°C (2 min) to 60°C (0 min) at 5°C/min, then to 150°C (4 min) at 20°C/min. The acquisition was performed by ChromCard computer software (Fisons Instruments, Milano, Italy).

Reagents. 2 mL of BTEX standard solution (Promochem, Warsaw, Poland) in redistilled water (Milli-Q system, El Paso, TX, USA) was used. The concentration of benzene was 4.74 μg/mL, toluene – 4.33 μg/mL, ethylbenzene – 4.54 μg/mL, and m,p-xylene – 4.33 μg/mL.

Preparation of fiber surfaces. Native quartz fibers from Optical Quartz Fibers Laboratory (UMCS, Lublin, Poland) o.d. 120 μm and polyurethaneacrylate-coated fibers from the same laboratory were applied. The different plasma conditions for fiber coatings preparation were used. The structures of fused silica fiber, an ethoxy-polydimethylsiloxane-coated fiber (PDES), and polydimethyl-siloxane (PDMS – Supelco Inc., Bellefonte, PA, USA), which were used for experiments, are shown in Table 1. An ethoxy-polydimethylsiloxane polymer coating (PDES) was prepared

during the telomerization process of cyclic siloxanes where the tetraetoxysilane was added to the reaction. The trifluoromethanesulfonic acid (CF_3SO_3H) was necessary as a catalyst of the reaction process. Approximately 10 μm-thick PDES-coated fibers were prepared for all experiments. For experimental preparation, the PDES-coated fibers were dried at a temperature of 200°C and 450°C for 30 min, under air conditions. This operation was performed for the curing and for the network of the polymer surface. The measurements of the heat distortion temperature for PDES and for polyurethaneacrylate coatings were performed before all experiments. A critical heat distortion temperature of 450°C for PDES was evaluated. At this temperature, it loses molecular stability and at temperatures higher than 450°C, the chemical decomposition of coating takes place. The high temperature influences molecular degradation and slows the decomposition of polymer. For this reason the final results of adsorption profiles, which are characterised by the higher extracted mass of determined compounds, are better for PDES dried at 200°C than for those dried at 450°C, because the latter's poor PDES polymer coating has a worse affinity for analytes.

Preparation of standards. The extraction of BTEX as testing substances from water solution (2 mL) was carried out at room temperature (20±0.5°C). The sample was stirred during the isolation. The isolation process was performed for 1, 2, 5, 7, 10, 15, 20 and 30 min. After isolation the syringe was transported to the GC injector where the analytes were desorbed. Finally, chromatographic analysis was performed.

Conditions for food analysis

Wine sample

sample	15 mL, 5.25 g NaCl added
SPME fiber	PA (85 μm film), PDES (*ca.* 10 μm film)
extraction	headspace, 20 min at 40°C
desorption	1 min, 220°C
column	Stabilwax, 30 m × 0.25 mm × 0.25 μm
oven	30°C (6 min) to 150°C at 5°C/min (hold for 0 min) then to 190°C at 20°C/min (hold for 4 min)
carrier	helium, 100 hPa
injector	splitless (closed 1 min), 200°C
detector	FID, 200°C

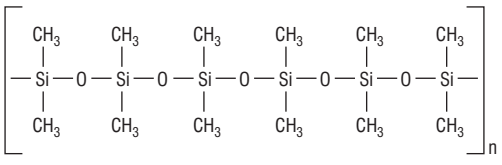
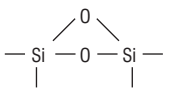
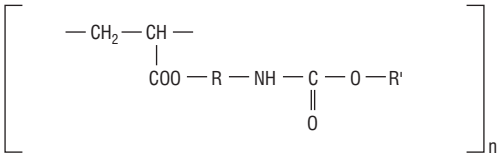
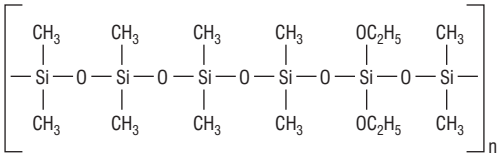
Peppermint candies

sample	46.6 mg, 10 μL methanol and 2 mL water added
SPME fiber	PDES *ca.* 10 μm film
extraction	headspace, 15 min at 30°C
desorption	2 min, 200°C
column	Stabilwax, 30 m × 0.25 mm × 0.25 μm
oven	40°C (2 min) to 110°C at 10°C/min (hold for 4 min) then to 160°C at 10°C/min (hold for 5 min)
carrier	helium, 100 hPa
injector	splitless (closed 2 min), 200°C
detector	FID, 250°C

Mentha piperita folium

sample	50 mg, 2 mL water added
SPME fiber	PDMS, 100 μm film
extraction	headspace, 15 min at 30°C
desorption	2 min, 250°C
column	SB-11, 30 m × 0.25 mm × 0.20 μm
oven	40°C (2 min) to 110°C at 10°C/min (hold for 4 min) then to 160°C at 10°C/min (hold for 5 min)
carrier	helium, 20 cm/sec, splitless/split (closed 2 min)
detector	quadrupole MS, m/z = 35÷500 at 1 scan/sec

TABLE 1. Characterization of the SPME fiber coatings used in the presented experiments.

Description	Chemical character	Fiber size	Structure	K value for toluene (K_{ow}=490)
Polydimethylsiloxane (PDMS)	Nonpolar	7 μm (bonded)		189.0
Fused silica	Polar	120 μm (non-coated)		–
Polyuethaneacrylate	Polar	45 μm (non-bonded)		2.2
Ethoxy-polydimethylsiloxane (PDES)	Nonpolar	*ca.* 10 μm (non-bonded)		292.1

RESULTS AND DISCUSSION

The characterization of prepared and commercial fibers is presented in Table 1.

The chemical properties of coatings, fibres size, structures and K values were taken into consideration. The following fibers were tested: ethoxy-polydimethylsiloxane-coated fibers (PDES), polyurethaneacrylate-coated fiber, fused silica fiber, and fused silica fiber after etching of hydrogen fluoride acid. All fiber sorption properties were examined using BTEX standards in methanol as testing substances. The obtained data were compared with those for commercial SPME polydimethylsiloxane fiber (PDMS – 7 μm thickness) from Supelco Inc. Various extraction times of BTEX isolation were taken into consideration, but 20 min was a sufficient time for reproducible results obtained for the extracted mass of BTEX. The comparison of extraction recoveries determined by extracted mass for the tested fibers and for PDMS is presented in Figure 2.

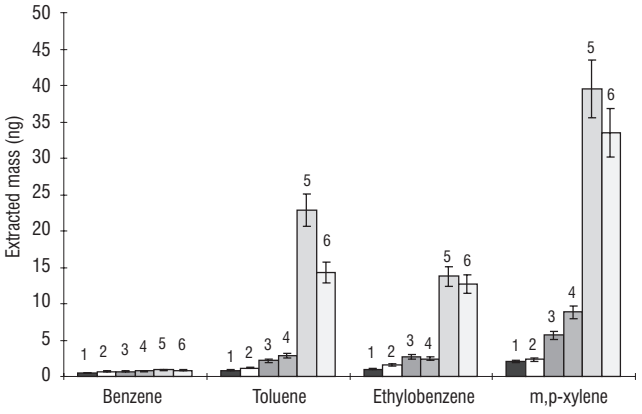


FIGURE 2. Comparison of extraction efficiency for BTEX for five different coated fibers (1) fused silica, (2) fused silica after etching by HF, (3) polyuethaneacrylate, (4) ethoxy-polydimethylsiloxane (PDES) after drying at 450°C, (5) ethoxy-polydimethylsiloxane (PDES) after drying at 200°C, (6) polydimethylsiloxane (PDMS).

It shows the efficiency of the extraction displayed as the extracted mass for five compounds after extractions of standard solutions with various SPME fibers. The most efficient extraction (adsorption on the fiber surface) is for the etoxy-polydimethylsiloxane-coated fibers (PDES) after drying at 200°C. On the other hand, the adsorption of BTEX on fused silica fiber etched with hydrofluoric acid HF (0.5%) is much better compared to untreated fused silica fiber. An interesting problem is that for the studied volatile organic compounds with larger molecular weight (more CH_3 – groups in the molecule) a higher sorption level was observed. A comparison of new modified fibers with commercial fiber PDMS (7 μm coating thickness) shows a higher recovery of extraction (*ca.* 20%) for PDES (*ca.* 10 μm coating thickness). This data also confirms that the thickness of the fiber coating and chemical properties of coatings exert an influence on extraction recoveries. In addition, the relative standard deviation values (RSD) determined in this study are below 10% for all tested substances. Similar experiments were performed for terpenes (menthol and menthon) and monoterpenes (linalool, α-terpineol, citronellol, nerol and geraniol) compounds [Ligor & Buszewski, 1999, 2001]. The correlation between the monoterpenes concentrations in food samples after the use of commercial PDMS and home-made PDES as SPME fiber coatings is presented in Figure 3. The obtained results are characterized by the equation of a straight line $y=1.1238x+0.0019$ and R^2 value 0.9964. These highly correlated results confirm that home-made SPME fiber coatings (PDES) could be widely used in qualitative and quantitative analysis of food samples.

The commercial and home-made coated fibers were used for the qualitative and the quantitative analysis of volatile organic compounds in wine samples. The concentrations of extracted components from wine samples are presented in Table 2.

TABLE 2. The results of quantitative analysis of wine samples (n=5).

No.	Determined compound	Concentration of determined compounds in red wine sample No. IF (mg/L)	Concentration of determined compounds in rose wine sample No. IIA (mg/L)	Concentration of determined compounds in white wine sample No. IIIF (mg/L)
1	Acetic aldehyde	ND	6.72 ± 0.21	51.62 ± 0.75
2	Ethyl acetate/methanol	104.46 ± 1.86	30.63 ± 1.40	67.70 ± 0.88
3	n-propyl alcohol	15.51 ± 0.38	13.27 ± 0.25	26.19 ± 0.20
4	iso-butyl alcohol	63.24 ± 1.41	55.13 ± 1.32	93.73 ± 1.15
5	n-butyl alcohol	1.29 ± 0.07	0.41 ± 0.04	7.36 ± 0.40
6	iso-amyl alcohol	208.80 ± 4.32	207.77 ± 5.19	354.96 ± 10.93
7	n-amyl alcohol	2.21 ± 0.08	9.08 ± 0.28	5.86 ± 0.06
8	Ethyl hexanoate	0.130 ± 0.005	0.09 ± 0.01	ND
9	Linalool	0.025 ± 0.003	0.022 ± 0.003	0.023 ± 0.002
10	Ethyl octanoate	1.51 ± 0.02	1.51 ± 0.05	1.50 ± 0.43
11	α-Terpineol	1.21 ± 0.06	0.89 ± 0.19	1.04 ± 0.05
12	α-Citronellol	0.88 ± 0.08	0.024 ± 0.005	ND
13	Nerol	0.017 ± 0.005	0.05 ± 0.01	0.05 ± 0.01
14	Geraniol	0.56 ± 0.13	0.54 ± 0.05	0.27 ± 0.05
15	Phenethyl alcohol	38.74 ± 0.55	25.75 ± 0.85	37.01 ± 057

ND – not detected

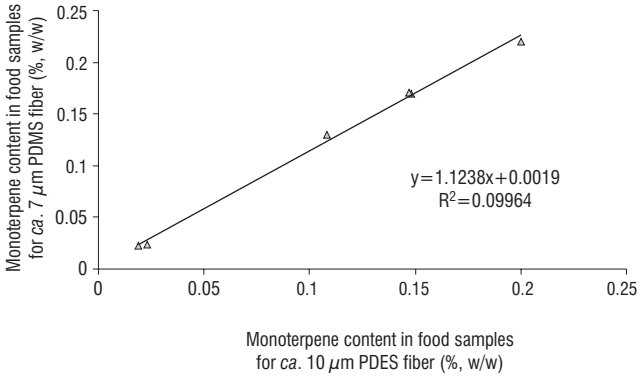


FIGURE 3. The correlation of monoterpenes concentrations in food samples isolated using various SPME fibers.

The highest concentration of alcohols was found in all wines. If the concentration of ethyl alcohol is omitted, the highest concentration of iso-amyl alcohol is detected in all samples. On the contrary the lowest concentration of ester groups was determined. In general, a higher concentration of volatile organic compounds in red wine than in white wine was observed. In particular, the concentration of sum of ethyl acetate and methanol (104.46±1.86 mg/L), linalool (0.025±0.003 mg/L), citronellol (0.88±0.08 mg/L) and nerol (0.017±0.005 mg/L) is higher in red wine. The typical chromatogram of red wine samples (No. I, semi-dry, French, vintage 1997) is shown in Figure 4. It was produced after fermentation of two species of grapes *Sauvignon* and *Syrah*. From an analytical point of view, these investigations could be helpful with regard to the possibility of further application of SPME method for the investigation of wine quality control during the production process.

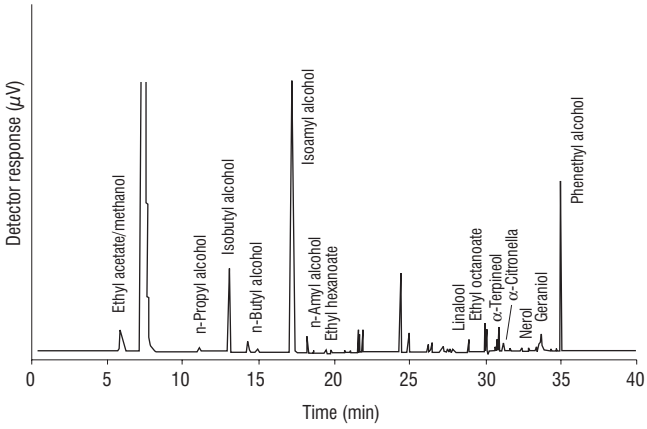


FIGURE 4. SPME-GC chromatogram of a red wine sample.

From an analytical point of view, the SPME method for the isolation and pre-concentration of terpenes from natural products is very interesting. As an example, the external and the internal standard methods were used for quantitative analyses of menthol and menthone, respectively. The detection limits for determined menthol and menthone at the ppb level were observed. These values ranged from 0.05 to 0.40 μg/mL for menthol and from 0.06 to 0.45 μg/mL for menthone. The detection limits values were different for various coated fibers used. Typical chromatograms obtained from peppermint candies, where *ca.* 10 μm PDES-coated fiber was used, are presented in Figure 5.

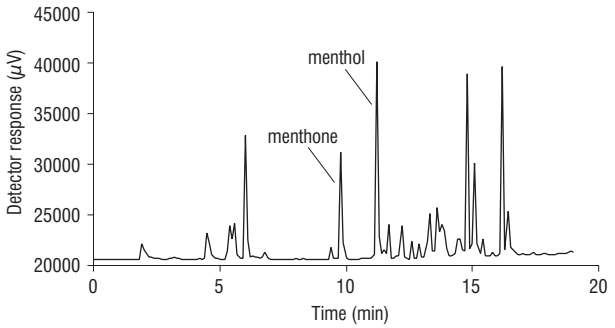


FIGURE 5. Typical chromatogram obtained from peppermint candy sample after the use of *ca*. 10 μm PDES-coated fiber.

This PDES-coated fiber is selective not only for menthol and menthone, but also for other volatile organic compounds in this sample. The results of quantitative analysis of menthol and menthone from peppermint candies confirmed the concentration of menthol 0.023% w/w and the concentration of menthone 0.019% w/w. For peppermint candy preparation, peppermint oil (customary obtained by steam distillation), is widely used. The presence of menthol, menthone and other terpenes in peppermint leaves is confirmed by SPME-GC/MS analysis. The GC/MS chromatogram obtained from *Mentha Piperita* extract is presented in Figure 6.

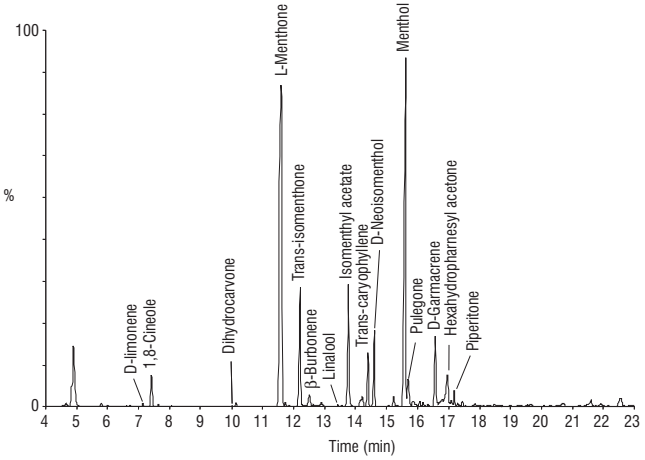


FIGURE 6. GC/MS chromatogram of volatile organic compounds obtained from *Mentha piperita* extract.

The retention data and m/z values of compounds extracted from Mentha Piperita leaves are presented in Table 3.

The identification of determined compounds were performed by the comparison of mass spectra data from libraries and by spectrum of standards.

CONCLUSIONS

The SPME method offers many advantages over other sample preparation techniques: it does not require organic solvent; it is relatively cheap; it is highly sensitive (analytes down to the ppm, ppb, and sometimes ppt levels can be detected); it uses short extraction time (in the order of minutes); it often does not require any other sample preparation step; it can easily be automated; it is easy to use. However, there are two main disadvantages to this technique: it is limited to aqueous samples; it cannot be used for highly concentrated analytes.

TABLE 3. Retention data of *Mentha piperita* extract composition by GC/MS.

Compound	t_R (min)	Molecular ion m/z
D-Limonene	7.140	136
1,8-cineole	7.410	154
Dihydrocarvone	10.010	152
L-Menthone	11.610	154
Trans-menthone	12.210	154
β-Burbonene	12.510	204
Linalool	13.427	154
Isomenthyl acetate	13.769	198
Trans-caryophyllene	14.394	204
D-neoisomenthol	14.602	156
Menthol	15.619	156
Pulegone	15.703	152
D-Garmacrene	16.570	204
Hexahydropharnesyl acetone	16.945	268
Piperitone	17.186	152

SPME was originally used for trace analysis of impurities in water. It has also been used in pharmaceutical, environmental, foods and flavours, forensic, and toxicology application. From an analytical point of view, it could be recommended for routine food and pharmaceutical analyses.

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ZASTOSOWANIE MIKROEKSTRAKCJI DO FAZY STACJONARNEJ (SPME) I CHROMATOGRAFII GAZOWEJ (GC, GC/MS) W ANALIZIE ŻYWNOŚCI

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W niniejszej pracy przedstawiono możliwości zastosowania mikroekstrakcji do fazy stacjonarnej (SPME) jako nowej metody przygotowania próbek do analizy. Bazując na teoretycznych i praktycznych aspektach wykorzystania SPME, dokonano porównania wydajności ekstrakcji węglowodorów aromatycznych (benzen, toluen, etylobenzen i m,p-ksylen – BTEX) jako substancji testowych. W przeprowadzonych eksperymentach posłużono się różnymi rodzajami włókien: komercyjnie dostępnymi (polidimetylosiloksan – PDMS, poliakrylan – PA) i nowo otrzymanymi (etoksy-polidimetylosiloksan – PDES, poliureatnoakrylan, szkło kwarcowe i szkło kwarcowe po trawieniu kwasem fluorowodorowym) – tab. 1. Wydajność ekstrakcji wyznaczono porównując masę związków wyodrębnionych za pomocą SPME. Najwyższą wydajność ekstrakcji BTEX zaobserwowano dla włókna PDES (utwardzanego w temperaturze 200°C) – rys. 2. Z wykorzystaniem nowych włókien do SPME, głównie PDES, dokonano analizy żywności i produktów pochodzenia naturalnego (wina, cukierki, zioła). Szczegółowo opisano zarówno zalety jak i wady wykorzystania SPME w analityce.