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Improving the detection limits of near infrared spectroscopy in the determination of aromatic hydrocarbons in water employing a silicone sensing phase

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Abstract

This work describes the use of a silicone sensing phase for the detection of aromatic hydrocarbons in water employing near infrared spectroscopy and the exploitation of the salting-out effect as a means of improving the limits of detection of the method. Sensing phases of polydimethylsiloxane (PDMS) were prepared from Silastic T2 (Dow Corning). Determinations were carried out by immersing a disk in a flask containing an aqueous solution of aromatic hydrocarbon (benzene, toluene, ethylbenzene and the xylenes, commonly called BTEX) and left under constant stirring. Afterwards, the disk was removed from the contaminated water, dried and placed in a home-made holder for absorbance measurements from 800 to 2500 nm, employing an MB 160 Bomem spectrophotometer. By employing a 2.0-mm height disk, measurements performed in water provided detection limits ($3s_{\text{blank}}$/sensitivity) of 1.1, 1.8, 1.1 and 1.1 mg L^{-1} for benzene, toluene, ethylbenzene and xylenes, respectively, while LOD values of 0.6, 0.7, 0.6 and 0.7 mg L^{-1} were obtained when sample solutions contained 2.0 mol L^{-1} sodium chloride. Detection limits of 0.080, 0.12, 0.14 and 0.27 mg L^{-1} were obtained with the use of a 5.0-mm height disk and 2.0 mol L^{-1} sodium chloride. Sodium chloride improves the limits of detection due to its salting out effect; however, headspace in the flask must be avoided during measurements, as the hydrocarbons are preferentially lost to the air. The precision of the proposed method was evaluated by constructing three analytical curves for toluene, providing average slopes and linear coefficients with relative standard deviations of 5.8 and 3.8%, respectively.

Keywords: BTEX; NIR spectroscopy; Optical sensor; NIR optode

1. Introduction

Near infrared (NIR) spectroscopy has proven to be a powerful analytical tool that has been applied in many fields, such as in the agriculture [1,2], food [3], pharmaceutical [4] and petroleum [5] industries and for environmental analyses [6]. The high acceptance of NIR spectroscopy and its widespread use relies on the fact that it can provide fast analytical methods, is non-destructive, does not need extensive sample pre-treatment and does not generate toxic residues, besides allowing simultaneous determinations with the aid of multivariate calibration chemometric techniques.

Most applications of NIR spectroscopy involve the determinations of species in organic matrices or non-aqueous solutions. In fact, water presents combination and overtone absorption bands due to the O–H bonds, which are very intense and broad, covering almost all the NIR region. These bands are stronger than the C–H absorption bands of the organic compounds, impairing direct determination of these species in water. Furthermore, non-polar organic compounds, such as benzene, toluene, ethylbenzene and the xylenes (BTEX), present low solubility in water, being found under the usual limits of detection of NIR spectroscopy, which is usually about 0.1%.

BTEX compounds are frequently determined in water by gas chromatography (GC), coupled to some pre-concentration technique, such as purge and trap [7,8] or solid phase micro-extraction (SPME) [9–12]. SPME is the most practical alternative to extract and pre-concentrate these aromatic compounds, performing a chromatographic analysis in two main

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steps. In the first, a silica fiber covered with a thin film of a suitable polymeric material (polydimethylsiloxane, polyacrylate, polystyrene–divinylbenzene) is immersed in the contaminated water or preferentially kept in its headspace for a period of time in order to reach equilibrium. This procedure allows extracting and pre-concentrating the hydrocarbons in the polymeric cladding of the fiber. Afterwards, the fiber is placed in the injection device of the chromatograph; the analytes are then thermally desorbed and carried towards the column and detection system. Although the SPME-chromatographic methods present high sensitivity, they are costly and bulky, offering difficulties for field monitoring.

Spectroscopic techniques have been proposed for determination of aromatic hydrocarbons in water samples. Although usually less sensitive, they provide simpler methods than the chromatographic ones. The direct determination of BTEX in water by UV spectroscopy has been proposed, presenting as its main disadvantage the interference due to the turbidity of the water samples [13]. This problem can be circumvented with the use of a liquid core waveguide based on Teflon AF, which is permeable to non-polar substances, and permits the construction of long optical path cell, improving the detectivity of the method [14].

SPME has been also applied in conjunction with spectroscopic methods of analysis for the determination of aromatic hydrocarbons in water. As in the chromatographic methods, an appropriate polymer (often polydimethylsiloxane, PDMS) is used to extract and pre-concentrate the organic compounds before measuring the attenuation of radiation in the ultraviolet [15–17], infrared [18–20] or near infrared [21–25] regions of the electromagnetic spectrum. In fact, these contributions described in the literature can be related to research on optical sensors, as a sensing phase is used to interact with the analyte prior to measurement.

Tilotta and co-workers [15] proposed the use of a chip made of PDMS (OV-1 stationary phase), with dimensions of 10 mm × 2 mm × 4 mm (volume of 80 μL), for UV determination of BTEX in water. After extraction, a 10-mm optical path chip provided detection limits of 97, 10, 12 and 5.5 $\mu g\,kg^{-1}$ for benzene, toluene, ethylbenzene and *p*-xylene, respectively. The sensor was subsequently applied to the determination of fuel aromatics in water [16]. Merschman and Tilotta [17] used a PDMS-clad silica optical fiber cable for determination of BTEX by employing evanescent wave detection. Detection limits 18.2, 6.2, 3.6 and 3.0 $mg\,L^{-1}$ for benzene, toluene, ethylbenzene and *p*-xylene, respectively, were obtained, with the use of a 10 cm long optical fiber and measurements at 254 nm.

Attenuated total reflectance spectroscopy with detection in the infrared region has been employed by Mizaikoff and co-workers [18] for determination of these aromatic hydrocarbons in water. A trapezoidal ZnSe ATR element was coated with ethylene/propylene copolymer film of 4.2 μm, providing detection limits of 45, 80 and 20 $\mu g\,L^{-1}$ (v/v) for benzene, toluene and *p*-xylene, which can be simultaneously determined by carrying out measurements in the fingerprint region (800–650 cm^{-1}). Parafilm M [19] and PDMS [20] films have been used by Tilotta and co-workers as sensing phases for extraction and pre-concentration of aromatic hydrocarbons, followed by detection in the infrared region. Detection limits of 182 and 66 $\mu g\,L^{-1}$ for benzene and *p*-xylene were obtained with the use of Parafilm M, while a value of 4400 $\mu g\,L^{-1}$ was achieved for *p*-xylene when PDMS was employed.

Evanescent wave optical sensors with near infrared detection have frequently been used for determination of hydrocarbons in water. Burck and co-workers extensively studied this approach [21–24], achieving limit of detections of 0.9 and 0.4 $mg\,L^{-1}$ for toluene and *p*-xylene, respectively, with the use of an 11-m long PDMS-clad optical fiber [24].

Recently, Albuquerque et al. [25] proposed the use of a silicone sensing phase for detection of BTEX in water. A transflectance probe with an optical path of 10 mm was employed, in which a silicone rod was adapted, and measurements made from 850 to 1800 nm resulted in detection limits of 8.0, 7.0, 2.6 and 3.0 $mg\,L^{-1}$ for benzene, toluene, ethylbenzene and *m*-xylene, respectively.

Although the contributions listed above are based on the concepts of solid phase micro-extraction, none of them employed the salting out effect in order to improve the limit of detection of the method, a procedure often employed in the headspace SPME procedure. This work describes the use of a PDMS phase for determination of BTEX in water based on transmittance measurements in the 780 to 2500 nm region and studies the effect of the salinity of the solution as a means of improving the detectivity of the proposed method.

2. Experimental

2.1. Reagents and solutions

Benzene, toluene, ethylbenzene and xylene were purchased from Merck and used without any purification. Methanol (spectroscopic grade, Merck) was used to prepare stock solutions of the BTEX compounds. Sodium chloride (Vetec) was used to adjust the salinity of the water samples. Silastic T2 and its curing agent were supplied by Dow Corning. Distilled-deionized water was used to prepare the contaminated water samples.

2.2. Sensing phase preparation

The PDMS polymer was prepared as recommended by the manufacturer and according to the procedure previously described [25]. The polymer was cast in PTFE templates and cut into small disks of 3.2 mm diameter with the aid of a cork borer. The heights of the disks were optimized for maximum sensitivity.

2.3. Reference solutions

Stock solutions of each BTEX compound were prepared in methanol (50,000 $mg\,L^{-1}$ for benzene and toluene; 10,000 $mg\,L^{-1}$ for ethylbenzene and xylene), which were then properly diluted to obtain aqueous reference solutions in the range from 0 to 80 $mg\,L^{-1}$. Aqueous toluene solutions from 0 to 400 $mg\,L^{-1}$ were also prepared to perform the initial experiments. The

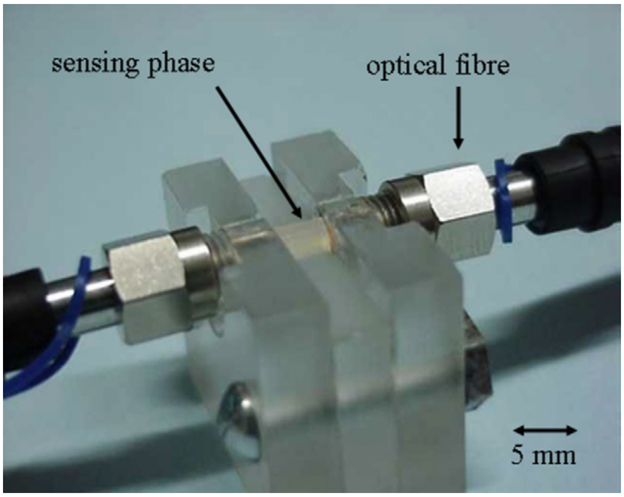

Fig. 1. Home-made acrylic cell used for transmittance measurements. The central part is an interchangeable spacer (2.0, 5.0 and 10 mm were employed).

methanol concentration in all solutions, including the blank, was maintained at 1.0%.

2.4. Apparatus

An MB 160 Bomem spectrophotometer was used for measurements in the 800–2500 nm range. Fig. 1 shows the home-made cell employed for transmittance measurements, which is furnished with acrylic spacers of different lengths (1.0, 2.0, 5.0 and 10 mm) in order to accommodate sensing phases of different optical paths. The radiation was conducted from the source towards the InGaAs detector, passing through the transmittance cell, by means of optical fiber cables (600 μm, 2 m, Hellma 041.202-NIR). A similar cell was also constructed that would be placed in the optical path of the spectrophotometer, allowing to carry out measurements without the aid of fiber optics.

2.5. Procedure

Determinations were carried out by immersing a PDMS disk in an 85-mL flask containing an aqueous solution of aromatic hydrocarbon (benzene, toluene, ethylbenzene or xylenes) and left for one hour under constant stirring (2700 rpm) at 22 °C for extraction of the hydrocarbon. Afterwards, the disk was removed from the sample, rapidly dried with a tissue and placed in the home-made cell for absorbance measurements. Each spectrum was acquired as an average of 100 scans, with a resolution of 8 cm^{-1}. For sample salinity studies, an adequate amount of NaCl was transferred to the flask, which was then filled with the water sample. All the extractions were carried out in a sealed flask, without headspace. The spectrum of a PDMS disk previously immersed in a 1.0% aqueous methanol solution for 1 h was employed as reference for absorbance calculations.

3. Results and discussion

The extraction of aromatic hydrocarbons by the PDMS sensing phase depends on the distribution constant of the analyte between the organic solid phase and the aqueous phase [19]. Therefore, the composition of the aqueous solutions directly affects this proportionality constant, as would be expected for a reference solution containing 1.0% methanol. The strategy of using methanolic stock solutions was adopted for convenience, as it helps to solubilize the aromatic hydrocarbons in water, making the preparation of the reference solutions easier. Preliminary studies with toluene solutions showed that 1.0% methanol does not affect the analytical signals when compared with those obtained in pure water, a result that is in agreement with the literature on SPME [26].

It has been shown that the diameter of the PDMS disk directly affects the time necessary to reach the equilibrium state as well the intensity of the analytical signal, as it defines the volume of the solid phase for a given height [25]. In the present work, a sensing phase of 3.2 mm diameter was always employed as this value was the lowest allowed by the diameter of the optical fiber cables employed to guide the radiation.

The position of the sensing phase in the transmission cell affects the baseline of the spectrum, as can be seen in Fig. 2, which shows the absorbance spectra of benzene, toluene, ethylbenzene and xylene. It can also be noted in this figure that these aromatic hydrocarbons do not present any absorption around 1300 nm. Therefore, the value of absorbance at 1298 was taken as reference for baseline correction and all the absorbance values presented in the present work are referred to the difference between the absorbance intensities at the wavelength of interest and those obtained at 1298 nm.

PDMS disks with heights of 2.0, 5.0 and 10 mm (that define the optical path) were evaluated regarding the signal intensities and absorbance values. Table 1 shows the sensitivities, expressed as the slopes at different wavelengths of the analytical curves constructed with 0–400 mg L^{-1} aqueous toluene solutions. When the measurements are carried out with the cell that employs optical fibers, the highest sensitivity is obtained with the 5.0 mm optical path. The 10 mm optical path does not present the best performance because the low intensity of radiation that reaches the detector due to the absorption of light by the silicone disk and, principally, due to the light attenuation by the fiber optic cables. This assumption is confirmed by the highest sensitivity provided by the 10-mm disk when the analyt-

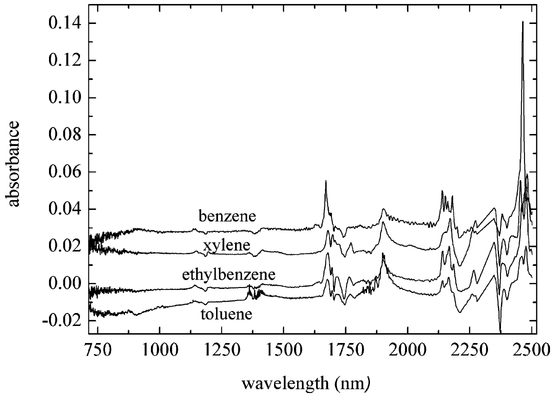

Fig. 2. Spectra of benzene, toluene, ethylbenzene and the xylenes obtained after their extraction from 100 mg L^{-1} aqueous solutions (3.2 mm diameter and 2.0 mm high PDMS disk, extraction for 60 min, cell with optical fiber).

Table 1
Sensitivities (slopes) of the analytical curves for toluene as a function of the optical path (extraction time of 60 min)

Optical path (mm)	Wavelength (nm)			
	1675	2140	2165	2183
2.0[a]	1.6×10^{-4}	1.4×10^{-4}	1.9×10^{-4}	1.0×10^{-4}
5.0[a]	5.4×10^{-4}	4.9×10^{-4}	6.7×10^{-4}	3.4×10^{-4}
10.0[a]	5.0×10^{-5}	2.4×10^{-5}	4.1×10^{-5}	7.9×10^{-5}
10.0[b]	4.4×10^{-4}	3.4×10^{-4}	4.6×10^{-4}	1.7×10^{-4}

[a] Cell with optical fiber.
[b] Cell without optical fiber.

ical curve is obtained by performing measurements without the use of fiber optic cables to guide the light. These results indicate, as expected, a compromise between the light intensity that reaches the detector and the information gathered. For example, the 5.0 mm optical path provides low intensity signals at wavelengths longer than 2200 nm when optical fibers are used to guide the light, while the cell without these fibers provides information between 2200 and 2500 nm, which can be important for use in a multivariate calibration model for simultaneous determination of BTEX.

The salting out effect is widely used in headspace SPME, as the salinity of the sample solution reduces the solubility of the volatile organic compounds in water, therefore enriching the gaseous phase. This effect was also applied in the present work, as a means of increasing the detectivity of the method. Preliminary experiments employing aqueous reference solutions containing 0.5 mol L^{-1} NaCl were carried out in 85-mL flasks without avoiding headspace. Analytical curves obtained with 0–400 mg L^{-1} toluene reference solutions revealed that the hydrocarbon is preferably lost to the air, instead of being absorbed by the PDMS solid phase, as the slope of the curve obtained in water was higher than those obtained in NaCl solution. Furthermore, it was verified that solid sodium chloride must be added directly to the measuring flask in order to minimize the loss of volatile organic compound to air during sample preparation and/or handling. Fig. 3 depicts three analytical curves obtained in water and in 0.5 mol L^{-1} NaCl solutions with and without headspace, indicating that the sensitivity of the method can be improved if headspace is avoided during the measurements. Table 2 lists the absorbance values obtained in measurements carried out with a 400 mg L^{-1} toluene solution whose NaCl concentration was varied from 0 to 2.0 mol L^{-1}. As can be noted, the signal intensities increase when the salt concentration is increased, also indicating that the use of NaCl concentrations higher than 2.0 mol L^{-1} can still improve the detectivity of the method. However, 2.0 mol L^{-1} NaCl was employed throughout, as it is high enough to demonstrate the improvement obtained with the exploitation of the salting-out effect.

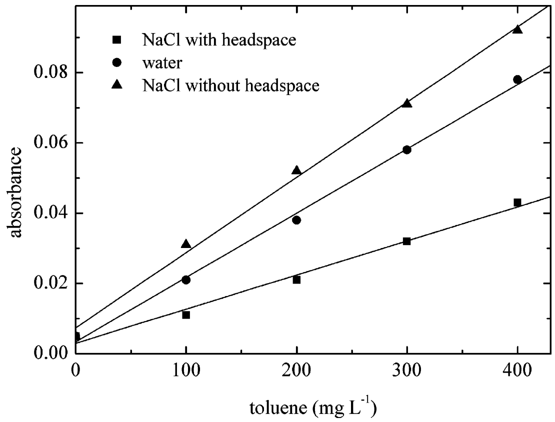

Fig. 3. Analytical curves for toluene obtained after its extraction from water and from 0.5 mol L^{-1} NaCl solutions with and without headspace (3.2 mm diameter and 2.0 mm high PDMS disk, extraction for 60 min, 1675 nm).

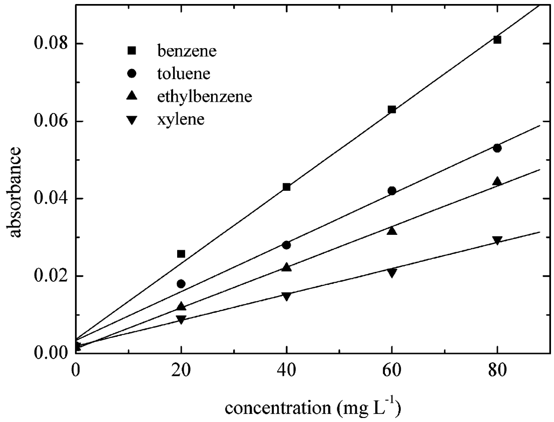

Fig. 4. Analytical curves for benzene, toluene, ethylbenzene and xylene (3.2 mm diameter and 5.0 mm high PDMS disk, extraction for 60 min, 1675 nm).

Fig. 4 shows the analytical curves obtained for the BTEX compounds over the 0–80 mg L^{-1} range, using solutions made 2.0 mol L^{-1} in NaCl. A 5-mm long disk was employed for extraction and a cell without optical fibers was employed for absorbance measurements. Detection limits (calculated as being three times the standard deviation of the blank divided by the sensitivity of the analytical curve) of 0.6, 0.7, 0.6 and 0.7 mg L^{-1} were obtained in these conditions for benzene, toluene, ethylbenzene and xylenes, respectively, while values of 1.1, 1.8, 1.1 and 1.1 mg L^{-1} were obtained when reference solutions were prepared in water. However, limits of detection of 0.080, 0.12, 0.14 and 0.27 mg L^{-1} were obtained for benzene, toluene, ethylbenzene and xylene, respectively, for extractions performed in 2.0 mol L^{-1} NaCl, with a 5.0 mm disk and measurements employing the cell with fiber optics cables to guide the light, which were similar to those described in literatures [15,18,19].

The precision of the proposed system was evaluated by constructing three analytical curves for toluene, providing average slopes and linear coefficients with relative standard deviations of 5.8 and 3.8%, respectively.

Table 2
Absorbance values for measurements performed at 1675 nm with a 400 mg L^{-1} toluene solution as a function of NaCl concentration (extraction time of 60 min, cell with optical fiber)

NaCl (mol L^{-1})	0	0.1	0.3	0.5	1.0	2.0
Absorbance	0.134	0.167	0.219	0.300	0.469	0.665

4. Conclusions

The results obtained in the present work indicate that the salting out effect can be employed to improve detectivity in the NIR determination of aromatic hydrocarbons in water, using a silicone rod as sensing phase. However, the extraction step must be performed in the absence of any headspace, as the volatile hydrocarbons are preferably transferred to the air instead of to the silicone sensing phase. Finally, the detection limits achieved for BTEX compounds were similar to those obtained with Mid Infrared or evanescent field optical sensors, presenting as advantage the use of simpler instrumentation.

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M. Fernanda Pimentel is an associate professor in the Department of Chemical Engineering of Federal University of Pernambuco (UFPE) since 1998. She received her PhD in chemistry from UFPE in 1996, working with simultaneous multicomponent determinations employing multivariate calibration and ICP OES. Her currently research interests are on near infrared spectroscopy and chemometrics.