



Remote optical fibre microsensor for monitoring BTEX in confined industrial atmospheres

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ABSTRACT

A portable optical fibre sensor has been developed for remote monitoring of benzene, toluene, ethylbenzene, p-xylene, m-xylene and o-xylene (BTEX). Firstly, the analyser was tested for calibration and its analytical performance for BTEX monitoring compared with a more classical analytical method, namely gas chromatography coupled to a flame ionization detector (GC-FID). The developed remote sensor shows several analytical advantages such as, high analytical sensitivity and accuracy, good linearity and stability of the analytical signal and short analytical time. Secondly, the optical fibre based sensor was applied to air monitoring for detection and quantification of BTEX in a confined industrial environment. The analytical signal measurement was performed by wireless at 20 m of distance from the local of analysis. Besides, the reported sensor showed a high degree of portability, compact design and high analytical performance for remote BTEX monitoring, *in situ* and in real-time.

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1. Introduction

Volatile organic compounds (VOCs) are an important class of indoor air pollutants and even at a trace amounts, these compounds have a high potential hazard to human health due to their carcinogenic nature. In industrial environments, VOCs namely benzene, toluene, ethylbenzene, p-xylene, m-xylene and o-xylene (BTEX) could arise not only from industrial activities but also from outdoor sources (such as vehicle pollution) and from building materials (such as cleaning products, paints and adhesives/sealants). The level of exposure to VOCs in an industrial environment is substantially higher than in non-industrial environments. Human exposure to VOCs is a function of both time and concentration and symptoms of overexposure include fatigue, headache, nausea, dizziness, skin and eye irritation and central nervous system damage. The workplaces with low air quality could negatively affect worker's efficiency and productivity. Therefore, obtaining reliable monitoring data, *in situ* and in real-time, of VOCs in indoor industrial atmospheres to prevent exceeding safety levels and assuring an acceptable air quality becomes an important aim both in environmental sciences and in sensors technology.

Fibre optical sensors show very appropriate analytical characteristics and attractive advantages, especially when compared to the more classical analytical methods and chemical sensors, for pollutants monitoring. They show no electromagnetic interferences, high durability, small size, low maintenance cost, short analytical time and simple design [1]. Furthermore, optical fibre sensors provide the possibility for fast, accurate and safe detection of toxic compounds besides showing high potential for remote measurements in inaccessible and harsh environments, in a continuous mode operation. Many research groups [2–5] focused their attention on optical fibre sensors as a tool for monitoring the concentration of VOCs. Although some miniaturized devices have been reported for VOCs detection, such as for example, a microfluidic device mesoporous silicate adsorbent [6], they have not shown yet an adequate analytical selectivity or sensitivity.

This work aims at the development of a new and portable design for an optical fibre sensor for remote monitoring of BTEX in confined environments with both high selectivity and sensitivity besides a capability for transmission of data by wireless.

2. Experimental

2.1. Optical fibre preparation

The sensor head consists on a monomode optical fibre (OF) pigtail coated with a nanometric fluorosiloxane polymer film. An optical coupler (OC) 50:50 was utilized with OF core and cladding diameters of 9/125 μm , respectively. The OF was mechanically

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uncladded and cleaved on a length of 20 mm with a Cleaver V6 (from Future Instrument) precision fibre cleaver. The sensitive film of poly[methyl(3,3,3-trifluoropropyl)siloxane] (PMTFPS) was deposited on the cleaved OF end by spray technique, using a coating solution of PMTFPS at 0.01% in dichloromethane. Finally the sensitized optical fibre section was cured at 70 °C overnight and then introduced through a Teflon plug inside the 7.2 cm long analytical tube (AT). The film thickness was estimated as 2 nm by Rutherford backscattering spectrometry (RBS).

2.2. Preparation of calibrants for BTEX determination

Aromatic volatiles standard mix solution of benzene, toluene, ethylbenzene, p-xylene, m-xylene and o-xylene, 100 µg/mL of each compound in methanol, was prepared from analytical grade reagents obtained from Supelco (Cat no. 47504).

2.3. Analytical details and experimental apparatus for calibration process

Fig. 1 shows the analytical apparatus and experimental lay out used for the calibration of the OF sensor. A standard mixture of BTEX was injected with a gastight micro-syringe (Hamilton) at the top of a glass cell (injection cell: IC). The temperature at the injection cell was controlled by a coiled tape heater (TH1) from Cole Parmer and kept at 150 °C. The aromatic compounds were injected as liquids, and after vapourization the aromatic vapours were carried by a continuous stream (25 mL min⁻¹) of reagent grade air (Linde Minican, Linde Sogás), controlled with a flowmeter (R), from Sigma, to the glass tube (GT). The GT containing a fused silica fibre coated with a film of polydimethylsiloxane (PDMS) was surrounded by a second tape heater (TH2) and constitutes the adsorption/desorption system component. Three way valves (valve 1: V1 and valve 2: V2) were used to connect the three main components of the analytical system: injection/sampling, adsorption/desorption and detection. At the adsorption step the V1 is open for allowing the flow in the IC–GT direction, while V2 is closed for the GT–AT flow but open for air outflow; in this way during the adsorption phase a constant flow of air is introduced through the adsorption component but no air flows into the detection component.

The analyte molecules are thermally desorbed by increasing the temperature of the tape heater (TH2), which surrounds the glass tube, by means of a temperature program controlled by a software home-made. The tape heater starts at 25 °C with a program rate of 30 °C min⁻¹ until 75 °C and 10 °C min⁻¹ until 150 °C. At this desorption phase the V2 is open on GT–AT direction, allowing the flow of analyte to the detection component of the analytical device. The exposure of the fluorosiloxane polymer film to BTEX vapours inside the analytical tube leads to changes on the reflected light power,

which is measured with a photodiode as the analytical signal. The light source used was a laser diode (1 mW, λ = 1550 nm) set at CW (continuous waveform) regarding the operational mode frequency. The choice of a working wavelength of 1550 nm was based on a previous observed increase in the analytical signal when increasing the wavelength from 1310 nm to 1550 nm [8]. The laser and the photodetector were integrated in an electronic device home-made and data acquisition was performed by a computer with software also home-made.

2.4. Analytical methodology, including experimental apparatus for sampling at confined atmospheres

Sampling took place at a confined environment in a Portuguese solvent industry and the details of the analytical apparatus are shown in Fig. 2. The air sample is continuously vacuum pumped (VP) from the industrial atmosphere at a flow rate of 0.2 L min⁻¹ (during 25 min) controlled by a mass flowmeter (FM). A second flowmeter (R) was connected to the analytical tube in order to detect any gas leak. The aromatic compounds (sampling from the confined environment) are adsorbed onto the PDMS fibre placed inside the GT during the adsorption step.

The experimental lay out used for *in situ* BTEX monitoring was similar to the one used for the calibration, except for the injection system, which is absent in this analytical system, and for a new component which includes a vacuum pump for air sampling. During the gas sampling step, the valve V1 is open for allowing the air sample to contact the PDMS fibre enclosed in the glass tube, before exiting through V2 which is in position open for air outflow and closed for GT–AT flow.

During the desorption step, the analytes are thermal adsorbed by increasing the TH2 temperature, using the same temperature programme as for the sensor calibration. At this stage the position of values V1 and V2 are changed: V1 is closed for air sampling and open to a flow of air (A) pure, while V2 is open in the direction of GT–AT flow; these changes in the position of the three way valves allow the desorption of the analytes from the PDMS fibre and the flow of the analytes into the OF detection system. The analytical tube was maintained at room temperature during both sampling and calibration processes. The light power guided through the OF is reflected when the analyte molecules are present at the fibre/fluorosiloxane film interface, thus causing a change in its refractive index leading to variations of the reflected optical power, which is monitored by a laptop with home-made software.

The sensor system elements excluding the air container, flowmeter, vacuum pump and laptop were appropriately located in a home-made box (height: 12.0 cm, width: 20.3 cm and length: 29.1 cm), providing the system with a very compact and portable design.

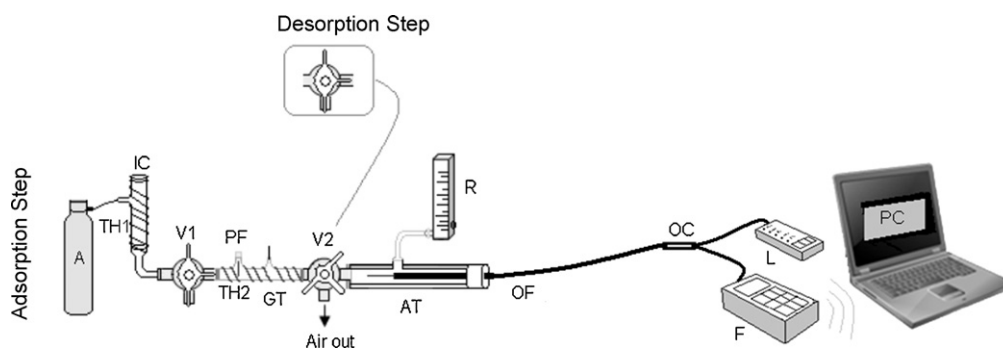


Fig. 1. Experimental apparatus used for the calibration (A: air, IC: injection cell, V1 and V2: three way valves, TH1 and TH2: tape heater, PF: PDMS fibre, GT: glass tube, AT: analytical tube, R: flowmeter, OF: optical fibre, OC: optical coupler, L: laser, F: photodetector, PC: laptop with home-made software).

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