Development of a microfluidic preconcentration device for sub-ppb level detection of BTEX

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Abstract

Nowadays, indoor air pollution has become a major concern for society due to its significant impact on human health leading to 3.8 million premature deaths every year. It has been estimated that people spend about 90% of their time in closed environments where the concentration of pollutants can be from 2 to 10 times higher than outdoors. Among the broad variety of contaminants identified in indoor air, benzene, toluene, ethylbenzene and xylenes (BTEX) require particular attention since BTEX exposure has been associated with the development of numerous pathologies including asthma, kidney damage or neurological problems. In the case of benzene, more serious effects such as leukaemia had led the European Union to establish in 2013 a threshold limit value of 5 μg m-3 (1.6 ppb) for this pollutant in public buildings. In France, this limit was decreased to 2 μg m-3 (0.6 ppb) in 2016. Hence, in order to check if indoor air quality (IAQ) is in accordance with the recent legislation, on-site rapid and sensitive analysis is required.
Our research group recently developed a gas chromatograph (GC) laboratory prototype enable to perform BTEX analysis in 10 min and exhibiting detection limits between 1–3 ppb for the different compounds. This work is focused on the development of a preconcentrator (PC) to improve BTEX sensitivity to sub-ppb levels. In this study, an aluminium PC with dimensions of 40 mm × 40 mm × 12.3 mm and weight of 54.9 g was manufactured and integrated into the abovementioned GC laboratory prototype. It consists of a system of microchannels connected to a microfluidic cavity of 4.6 mm × 7.4 mm where 5.8 mg of Basolite C300 are placed. The heating system consists of three heating cartridges (Watlow, St. Louis, MO, USA) of 70 W each allowing to reach a temperature ramp of about 150 ∘C/min.

Analytical performances of the device were demonstrated in terms of adsorption capacity and repeatability showing satisfactory results. Linearity of the detector response was confirmed by correlation coefficients (R2) ranging between 0.9608 and 0.9946, obtained when

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conducting several injections of different sample volumes in the range 0–80 mL for a fixed gaseous concentration of BTEX. In environmental monitoring, a compromise between sensitivity and reasonable time resolution is required, therefore, a sample volume of 20 mL was selected for the next experiments. Using a 20 mL sampling volume, BTEX analysis was conducted in 19 min, which is acceptable to establish concentration–time profiles. Prototype calibration was performed in the range 2.5-100 ppb showing that the peak area increases linearly with the injected concentrations and demonstrating that the preconcentrator operates in a very satisfactory way as confirmed by the obtained correlation coefficients ranging from 0.9777–0.9959. Using a signal-to-noise ratio of 3 for the two lowest injected concentrations (2.5 and 5 ppb), detection limits of 0.20, 0.26, 0.49, 0.80 and 1.70 ppb were calculated for benzene, toluene, ethylbenzene, m/p-xylenes and o-xylene, respectively. It should be noted that lower detection limits can be achieved with this new prototype by increasing the sample volume passing through the preconcentration unit. In addition, the benzene limit of detection of 0.2 ppb obtained with the novel miniaturized GC integrating a preconcentration module is now consistent with the threshold limit value of 0.6 ppb imposed by the French regulation.

Even if these results are successful, there are still significant scopes for improvements in terms of peak resolution. The development of a miniaturized PC is currently in progress with a faster heating system, which would lead to more rapid desorption and then to thinner chromatographic peaks. In addition, the miniaturization of the PC altogether with the new heating system will reduce the energy consumption of the preconcentration unit, increasing the autonomy and improving the portability of the GC prototype. Simultaneously, a study of volatile organic compounds (VOC) adsorption on different materials is performed to extend the number of the detectable compounds.

Keywords: preconcentrator, microfluidics, miniaturized gas chromatograph, BTEX