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Gas preconcentrator made by rolling up a printed hotplate on foil

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Abstract

This paper presents the design and implementation of a foil gas preconcentrator (FGP) on polyimide (PI) substrate. One novelty of the paper is that our device is made on flexible foil by using printing whereas all preconcentrators seen in literature are mainly based on rigid substrates and are micro-machined using cleanroom processes. Printing allows the additive and localized deposition of materials at low temperature on large area and can be applied to both the patterning of the heating element and the integration of the gas absorbent material. The benefits are the easy and flexible processing of cost-effective and lightweight preconcentrators for a variety of target gases. The tubular shape of the FGP is obtained by rolling up and sealing the inkjet printed gold hotplate on foil, which is then filled with the gas absorbent material (Carbopack B and Tenax). The diameter of the inlet/outlet of FGP is adjustable leading to high flow rates, up to 1.5 L/min, much larger than their silicon counterpart. The concept was validated using two target gases (Benzene and Acetophenone) at concentrations down to 250 ppb.

Keywords

Micro-hotplates; Inkjet printing; Polymeric foil; Gas preconcentration; Pollution monitoring

1. Introduction

Since a decade, different designs of silicon-based preconcentrators have been reported in literature based typically on two preponderant approaches: a close device (micro-channel device sealed with cover) and open shape device (micro-hotplate-type device) [1, 2]. However all these devices were microfabricated in a cleanroom environment resulting in a high component cost and time consuming process. Meanwhile our group also reported on micro-hotplates on flexible polyimide (PI) foil applied to gas sensors operating up to 300 °C [3], which were also produced in cleanroom facility. We have also recently published our work on low cost inkjet printed - micro-hotplates on PEN (Polyethylene naphthalate) foil for gas sensors operating at low temperature (<100 °C) [4]. Here we report on high operating temperature printed micro-hotplates for gas preconcentration based on the patterning of gold nanoparticles-based heater on PI foil. We propose a cost-effective process to develop a tubular gas preconcentrator by rolling up a printed micro-hotplate on foil before filling it with an adsorbent. Our foil gas preconcentrator allows operating at higher flow rates than the silicon devices previously reported.

2. Design and fabrication

The micro-hotplate is made by inkjet printing (Dimatix DMP 2800 printer) a gold ink (from Harima) on 50 µm-thick PI foil (Kapton® EN from Dupont). The heater meander is 500 µm-wide and 100 mm long with a gap of 300 µm between its arms. The size of the hotplate *i.e.* the active area of the FGP was fixed to 10x8 mm². After a sintering at 250 °C during two hours, the electrical resistance of the heater was 50 Ω. The foil hotplate is then rolled and sealed with a high temperature adhesive for obtaining the tubular shape of the preconcentrator (Figure 1).

Before sealing the fluidic capillaries, the chosen adsorbent is inserted within the tube in granular form and blocked at the edges with fiberglass. Well established adsorbents, Carbopack B and Tenax TA (from Sigma Aldrich), were used in this work for demonstrating the concept.

Our device is designed with a large heater size for two reasons:

- ✕ On one hand, to obtain a long enough tubular preconcentrator to maximize the adsorption time by increasing the residence time of the target gas when going across the adsorbent material.
- ✕ On another hand, to be sure that the heater meander covers all the diameter of the FGP in order to avoid any cold area during the heating up of the device for gas desorption. The latter could reduce considerably

the preconcentration factor by acting as a second “cold” adsorption site, which would trap again the gas during the desorption phase.

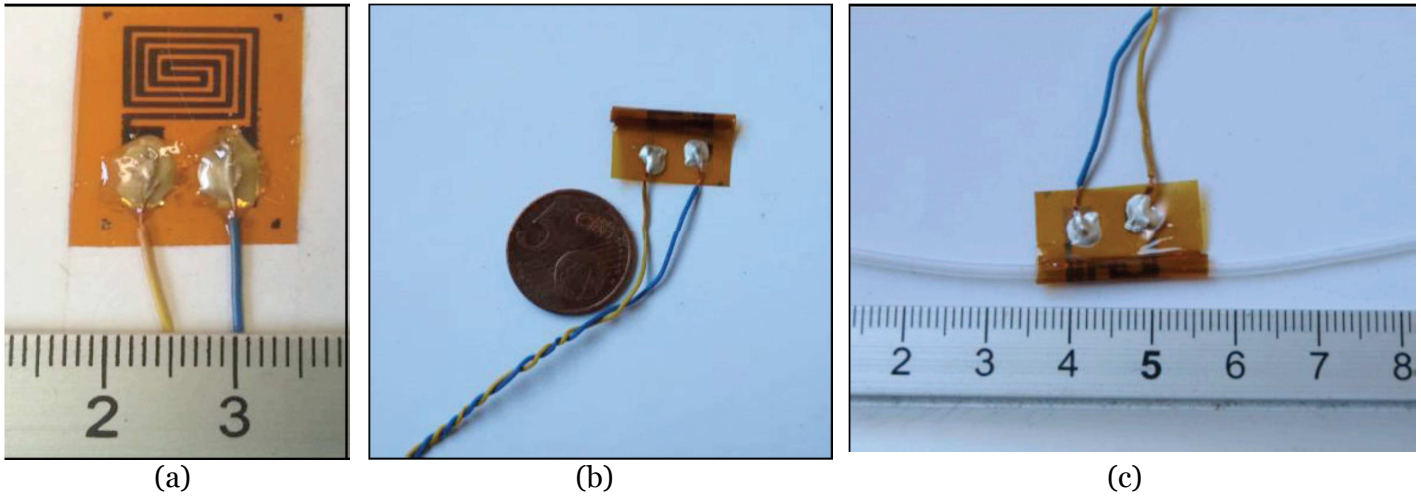


Figure 1: Implementation process of our tubular FGP: images of (a) A printed hotplate with electrical wires (b) The rolled up micro-hotplate compared to 5 euro cents and (c) The final FGP with fluidic interconnects.

3. Characterization and optimization

The design of the printed micro-hotplate was supported by FEM simulations using COMSOL and validated by thermal mapping experiments using micro-thermocouples. The gap between lines was narrowed from the edge to the center for better heat distribution across the active area of FGP and low power consumption. Our FGP allows reaching 350 °C with a power of 1700 mW, with a temperature gradient from the center to edge of less than 50 °C (Figure 2).

After the rolling up and sealing of the fluidic interconnects, the pressure drop was measured at flow rates from 1L/h to 100 L/h. This characterization showed that the FGP is usable at high flow, up to 100 L/h with low pressure drop, down to 100 mbars (Figure 3). In comparison, our previously reported silicon gas preconcentrators (SGP) [2] could not exceed 4 L/h without damaging the device. The operating flow rate of the FGP is mainly limited by the currently used fluidic interconnections which was the cause of the experimentally observed pressure drop. However, the inlet and outlet of FGP could be adjusted during rolling up of micro-hotplate for allowing larger fluidic interconnect in regards to the requirements of the application.

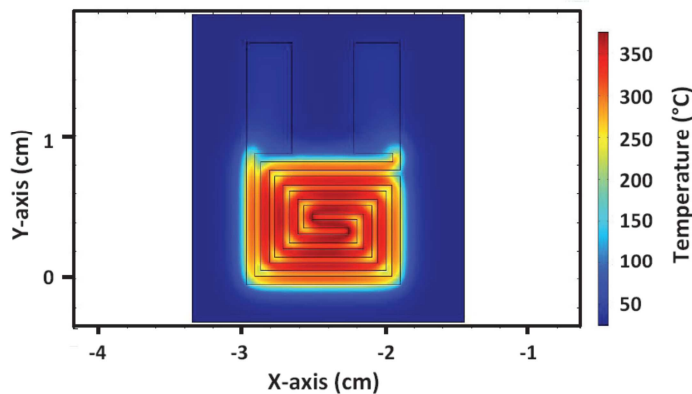


Figure 2: Thermographic simulation obtained on COMSOL 4.2 of the micro-hotplate operating at a power of 1700 mW.

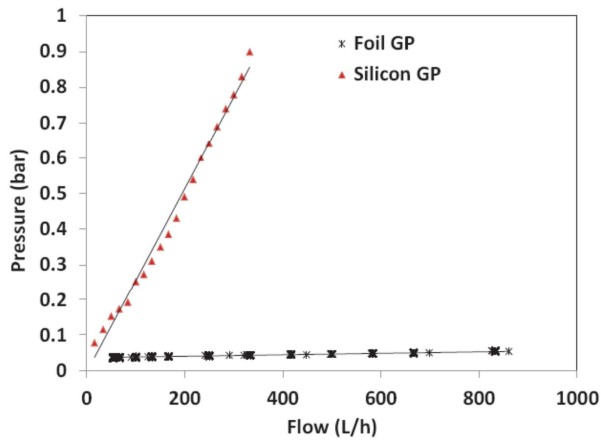


Figure 3: Flow (mL/min) versus Pressure (bar) obtained with a Foil GP filled with 1mg of Carpack B and a silicon GP filled with 1mg of carbon nanopowders.

4. Results on gas preconcentration

This study has been done in the frame work of the EU project SNIFFER which aimed at detecting explosives, illicit drugs, and volatile organic compounds for a variety of border security scenarios like smuggling prohibited goods. In this paper, we demonstrate the proper operation of the rolled-up preconcentrators on foil for aromatic compounds (benzene) and cigarettes additives (acetophenone). Carpack B was chosen as adsorbent because of its high affinity towards aromatic compounds whereas Tenax adsorbent was selected for its low desorption temperature towards sticky compounds like acetophenone.

As basic principle of operation for gas preconcentrators, a high adsorption flow rate, a low desorption flow rate, a relatively long adsorption time, and a very short desorption time are desired for good preconcentration performance. In this purpose, experiments were conducted with an adsorption flow rate and duration of 80L/h and 10 min, respectively, while the desorption flow rate and duration were set to 5 L/h and 10 min, respectively. As the desorption time is fixed by the desorption rate, the heating rate was fixed at the maximum value achievable in our experimental setup, up to 40 °C/s. Thanks to the use of high adsorption flow rates, experiments showed a preconcentration factor-PF (the ratio between the concentration after and before preconcentration), higher than the ones we observed for our silicon devices reported in [2] (Figure 4 left). Indeed, the PF obtained with our FGP is about 68 when it was exposed to 250 ppb of benzene.

However, the performances for the couple Tenax-Acetophenone are lower than for Carpack-Benzene because of low adsorption capacity of Tenax compared to Carpack and also the spreading of the desorption peak due to the high stickiness of acetophenone molecules which slows down the desorption rate (Figure 4 right).

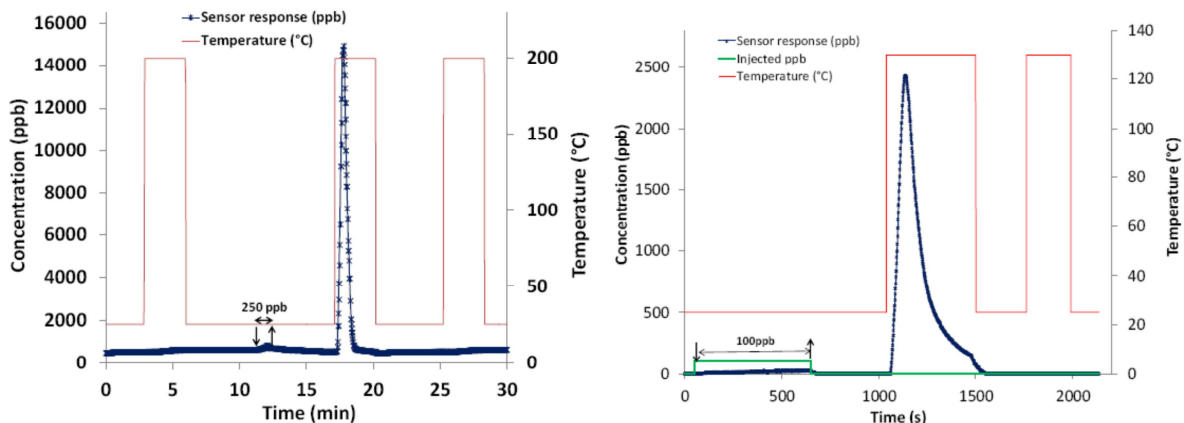


Figure 4: Sensor response for a FGP filled with 1mg of Carpack B when exposed to 250 ppb@1 min of benzene at 667 mL/min and desorbed at 200 °C with a flow of 66mL/min (left) and for a FGP filled with 1mg of Tenax TA when exposed to 100 ppb@10 min at 333 mL/min of acetophenone and desorbed at 130 °C with a flow of 33 mL/min (right).

5. Conclusions

We have successfully demonstrated the fabrication and testing of a versatile FGP based on gold heater printed on a polyimide foil. Successful operation of the devices was demonstrated for the preconcentration of benzene and acetophenone with a PF higher than silicon preconcentrators tested under the same conditions thanks to high flow rates allowed by our rolled up preconcentrator.

We are now working on the maximization of this PF by increasing the inlet/outlet diameter of the FGP during the rolling up for a higher flow rate.

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References

- [1] W.R. Collin, G. Serrano, L.K. Wright, H. Chang, N. Nuño, E.T. Zellers Microfabricated gas chromatograph for rapid trace-level determinations of gas-phase explosive marker compounds, *Analytical Chemistry*, 86 (2014), pp. 655-663
- [2] M. Camara, P. Breuil, D. Briand, N.F. de Rooij, C. Pijolat A micro gas preconcentrator with improved performance for pollution monitoring and explosives detection *Anal Chim Acta*, 688 (2011), pp. 175-182
- [3] J. Courbat, D. Briand, L. Yue, S. Raible, N.F. de Rooij Drop-coated metal-oxide gas sensor on polyimide foil with reduced power consumption for wireless applications *Sensors and Actuators B: Chemical*, 161 (2012), pp. 862-868
- [4] E. Danesh, F. Molina-Lopez, M. Camara, A. Bontempi, A. Vásquez Quintero, D. Teysieux, L. Thiery, D. Briand, N.F. de Rooij, K.C. Persaud Development of a new generation of ammonia sensors on printed polymeric hotplates *Analytical Chemistry*, 86 (2014), pp. 8951-8958