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CONCEPTION AND DEVELOPMENT OF MICROFABRICATED ELEMENTS FOR MICROFLUIDIC ANALYTICAL DEVICES

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KEY WORDS

Microfluidic analytical device, VOCs analysis, MEMS, indoor air quality.

ABSTRACT

In the last few years, researches have placed indoor air quality as the 9th cause of global burden of disease [1]. In indoor environments, a wide range of VOCs are especially abundant such as BTEX (Benzene, Toluene, Ethylbenzene and Xylenes), aldehydes (acetaldehyde, hexanal) and ketones (acetone, 2-butanone). These VOCs present harmful effects on human health [2] even at low concentrations.

ICPEES and In²Air Solutions have recently developed a portable miniaturized GC for real time detection of BTEX ppb levels [3], [4]. A laboratory prototype of this device was tested and validated under controlled conditions in the laboratory [3] and during two field campaigns in schools. In the latter, BTEX concentrations were for the first time measured continuously in different occupational conditions showing that these concentrations were higher in periods of school hours. Other VOCs, more precisely hexanal and 2-butanone, were also detected indicating the necessity of analysis of a wider range of pollutants. For this purpose, our work is focused on the conception and development of a multipollutants analyzer. The operating principle of our current device is described elsewhere [3]. In order to achieve a limit of detection down to few hundreds of ppt, an additional stage of preconcentration, based on thermal desorption, will be added.

Portability and low energy consumption are of a great importance for on-site real time monitoring of indoor air quality. Therefore, our aim is to miniaturize the elements which are the bulkiest and require the highest energy supply in analytical devices. More specifically, micro-fabrication techniques were deployed so that different parts of two components, a pre-concentrator and a micro-column, were developed on wafers of small dimensions, 15 x 25 mm and 50 x 50 mm respectively. The combination of these microfabricated elements with integrated heaters and temperature sensors enables the fabrication of sensible analytical devices with very limited power consumption.

In this work, heating elements and sensors for both devices were fabricated by means of metal deposition of titanium and silver using e-beam and Joule effect evaporation, respectively. In the microcolumn, the heating system is composed of four resistances of 82 Ω which allow an increase of the temperature from 25 °C to 200 °C in 150 s whereas two other resistances of 430 Ω act as temperature sensors (see **Figure 1**). In the case of preconcentrator, a faster raise of temperature is critical to achieve a quantitatively desorption of the VOC, thus a final temperature of 250 °C has to be reached in 10s. In order to determine the most suitable configuration to

meet this goal, three different prototypes of heating systems were designed in which the resistances vary from 32 Ω to 46 Ω and from 120 Ω to 420 Ω for heating elements and temperature sensors, respectively.

Once the heating systems were fabricated, the next step was the micro-column development. A study of wet etching kinetics of Si <100> with KOH (40%) was performed in order to evaluate the suitability of this technique for microchannels fabrication. During this study, Si etching rates were calculated for 50, 60, 70 and 80 °C to be 14.7, 22.9, 39.7 and 63.1 $\mu\text{m/h}$, respectively (see **Figure 2**). These results demonstrate that Si etching rate increases exponentially as function of temperature. Additionally, Arrhenius plot was traced enabling the extraction of the activation energy of the Si etching reaction with KOH (40 %) at 57.5 kJ/mol. Afterwards, two micro-columns were etched using KOH (40 %) at 70 °C on a silicon wafer of 3 inches. For these prototypes, a serpentine configuration containing channels of 80 μm width spaced at 450 μm was selected. The total lengths of the two microcolumns is 1 m and 2 m.

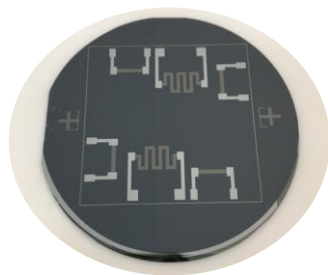


Figure 1: Heating system of the micro-column composed of four resistances of 82 Ω and two other resistances of 430 Ω which act as temperature sensors

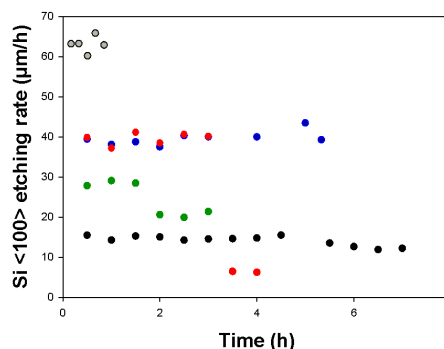


Figure 2: Si <100> etching rate (right) with KOH (40%).

In black: silicon wafer with SiO₂ and Si₃N₄ layer at 50 °C;

in green: silicon wafer with SiO₂ layer at 60 °C; **in red:** silicon wafer with SiO₂ layer at 70 °C; **in blue:** silicon wafer with SiO₂ and Si₃N₄ layer at 70 °C; **in gray:** silicon wafer with SiO₂ layer at 80 °C.

In this work, we report the conception and development of heating systems with integrated temperature sensors for two analytical components, a preconcentrator and a micro-column, using MEMS technology to serve the purposes of on-site real time monitoring of indoor air quality. The use of wet etching as an alternative and cheaper technique to fabricate micro-columns is also presented. Future challenges consist on integrating different stationary phases in the micro-column and evaluating its efficiency for VOCs separation as well as fabricating a preconcentrator integrating the heating elements and temperature sensors already developed. Finally, both components will be combined into a laboratory prototype to determine its analytical performances under controlled conditions and during field measurements.

Acknowledgements

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References

- [1] K. R. Smith *et al.* (2014). Millions Dead: How Do We Know and What Does It Mean? Methods Used in the Comparative Risk Assessment of Household Air Pollution. *Annu. Rev. Public Health*, **35**, 185-206
- [2] Arif A. A. & Shah S. M. (2007) « Association between personal exposure to volatile organic compounds and asthma among US adult population », *Int. Arch. Occup. Environ. Health*, **80**, 711-719.
- [3] Nasreddine R., Person V., Serra C. A., & Le Calvé S. (2015) Development of a novel portable miniaturized GC for near real-time low level detection of BTEX. *Sens. Actuators B Chem.*, **224**, 159-169.
- [4] Nasreddine R., Person V., Serra C. A., Schoemaeker C., & Le Calvé S. (2015) Portable novel micro-device for BTEX real-time monitoring: Assessment during a field campaign in a low consumption energy junior high school classroom. *Atmos. Environ.*, **126**, 211-217.