

3D Printed Micro-Gas Chromatography (μ -GC) System for Improved Low-Cost VOC sensing

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Summary:

This paper describes the custom design of a 3D printed μ -GC column that is combined with commercial MOX gas sensors. A spiral-shaped column is designed in SolidWorks, the 3D printed structure is coated with OV-1, and integrated into an e-nose sensor system. 2-methyl butanol mixed with ethanol was used to test the μ -GC's ability to separate compounds based on their molecular affinity to the PDMS coating. Our preliminary results demonstrate the potential of a basic, low-cost 3D printed μ -GC based system to offer better selectivity than MOX sensors alone.

Keywords: Gas Micro chromatography, 3D printing, Metal oxide sensors, Volatile organic compounds (VOCs).

Background, Motivation an Objective

Conventional gas chromatography (GC) is known for its high accuracy and versatility in analyzing complex mixtures of chemical compounds having similar physicochemical properties. However, they are expensive, bulky and require high power, making them unsuitable for field-portable applications. Therefore, there is a clear need for economical, compact, and energy efficient alternatives that can provide comparable performance [1]. Recently, there has been an increase in effort to fabricate μ -GCs on a Si substrate using a simple Lift-off process. This approach enables the fabrication of miniature columns with precise control over dimensions and geometries, which may facilitate an efficient separation of target compounds. However, this method involves microsystems processing, which is costly and time-consuming for low volume applications. Here we are using a low-cost 3D printer (Elegoo Mars 4 Ultra) that can write micro-channels into a plastic resin in less than an hour.

One of the proposed applications of a portable μ -GC system is in plant pest control, which in turn could lead to the reduction of pesticide use. Plants emit specific volatiles when diseased or attracted by pests and one of identified compounds, 2-methyl butanol, is used for preliminary characterization of the proposed 3D printed μ -GC.

Design, Fabrication and Methodology

In this preliminary study, we have made a relatively short column length of 1.2 m (see Figure 1). The overall dimensions of the printed μ -GC

substrate are 40mm \times 40mm \times 5mm (length, width, and height) with a channel length of approximately 1.2 m, width of 0.5 mm, and depth of 0.4 mm. The channel walls were injection coated with an OV-1 stationary phase and the substrate sealed with 1.0 mm thick polyethylene terephthalate (PET) sheet. The μ -GC performance was investigated by employing Scio-Sense ENS161 metal oxide gas sensors before (S1) and after (S2) the μ -column (see Figure 2). 2-methyl butanol was chosen to assess the performance of the fabricated μ -GC column. Upon exposure to 2-methyl butanol, S2 showed delayed and reduced magnitude response curves as compared to S1 indicating that the compounds are affected by the OV-1 film. The variation in S2 response can be utilized to calibrate the μ -GC for different compound molecules.

A photograph of the entire designed column is shown in Figure 1(a) together with an insert revealing the channel dimensions and shape. Figure 1(b) shows the final OV-1 coated and fully sealed 3D printed μ -GC.

The μ -GC coating was prepared by first dissolving 0.025g of OV-1 in 5 ml of dichloromethane. The solution was then injected into the channel while the μ -GC substrate was placed on top of a hotplate set to 45°C. Afterwards, UV resin was spun coated on PET substrate and placed on the channel surface for sealing. The device was then tested for leaks before final assembly into the prototype system. Figure 2 depicts the μ -GC setup, comprising the two sensor chambers housing metal oxide sensors, the fully sealed μ -GC column, and a microcontroller (Feather TFT: ESP32-S3). In this arrangement,

the target VOCs pass first through the S1 chamber, then the μ -GC column, and finally the S2 chamber, while the microcontroller continuously monitors the resistance change of both sensors over time.

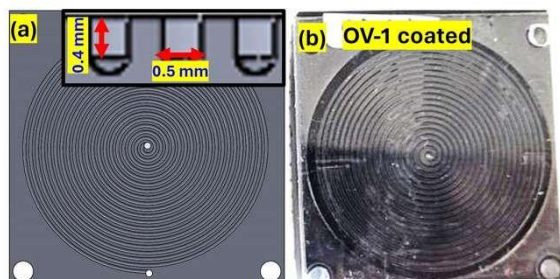


Fig. 1. (a) CAD design (b) sealed μ -GC.

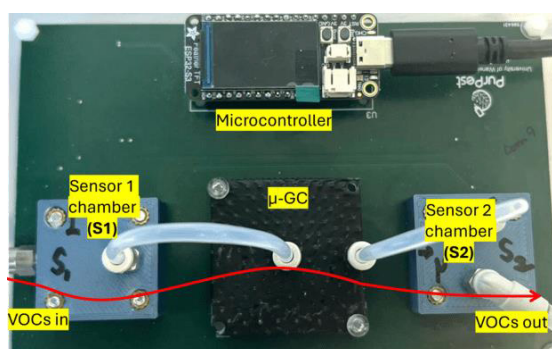


Fig. 2. μ -GC based e-nose.

Results and Discussions

Fig. 3 shows the response of sensors S1 and S2 to the mixture of 0.7 ppm pulses of 2-methyl butanol and 0.2 ppm of ethanol as a known interferant. Results indicate that OV-1 coated μ -GC column absorbs the compounds and releases them when dry air flows through the channel. This causes a delay and reduction in S2 response magnitude. As it can be seen from the enlarged image of the first cycle that the response and recovery time for S2 were delayed around 80 s and 50 s, respectively (see Figure 3(b)). On the other hand, the response magnitude for S2 was reduced by about 66% as compared to S1.

Our preliminary results suggest that this highly economical 3D printed μ -GC column holds potential for identifying different VOCs at PPM level. This cost-effective 3D printed μ -GC offers a promising method for quick onsite analysis of compound mixtures. The integration the μ -GC in front of a multi-sensor e-nose could enhance compound classification performance [2]. Further work includes detailed parametric investigations of coating thickness, optimum temperature, flow-rate, injection pulse width, and μ -column dimensions, as well as advanced processing of the time-dependent responses.

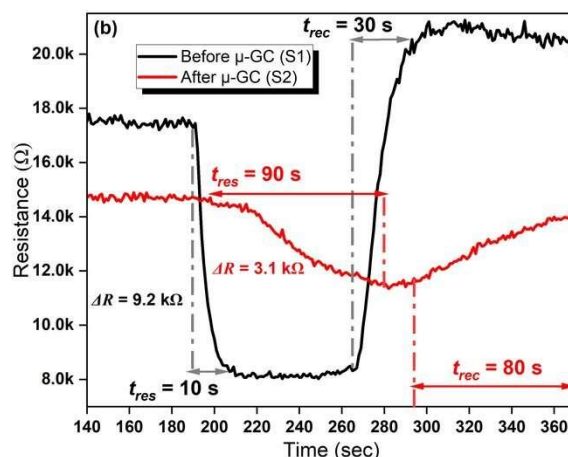
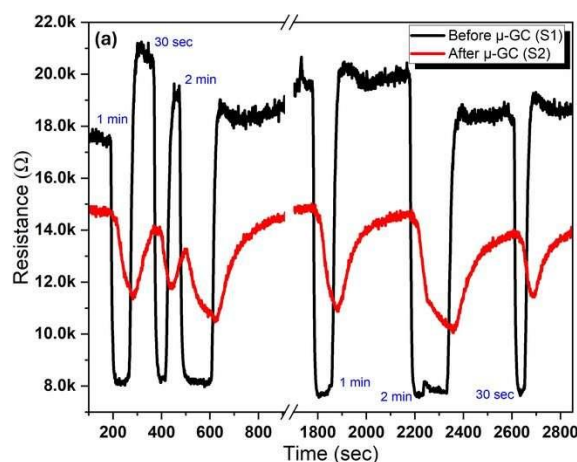


Fig. 3. Two repeats of sensors' response before and after OV-1 coated μ -GC column to 2-methyl butanol mixed with ethanol (a) at different exposure time, and (b) first cycle enlarged response curve.

References

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