

Development and Validation of a Portable GC-MOS System for High-Sensitivity and Selective Analysis of VOC

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Summary:

This study presents the development, setup, and validation of a miniaturized gas chromatography system using a metal oxide semiconductor gas sensor as detector and a pre-concentrator based on metal-organic frameworks as injector. The system addresses the need for cost-effective, portable solutions for monitoring chemical markers in complex gas phases. It was first validated in the lab showing a good separation of an aldehyde mix, which is used as calibration standard for fat oxidation markers. To evaluate real-world performance, the system was deployed in a quality control lab at a chocolate factory. The device successfully detected hexanal, a critical biomarker used to determine the freshness of nuts. Additionally, initial results indicate the suitability in bio-medical application, too.

Keywords: gas chromatography, MOS-detector, Metal-Organic Framework

Introduction

High-precision gas analysis has historically been restricted to specialized laboratories due to the substantial costs and technical complexity of traditional analytical equipment. In various sectors ranging from food quality control to medical diagnostics, there is an urgent need for the rapid and reliable detection of chemical markers in the gas phase. One of the major challenges of sensor systems in real world applications is the poor selectivity whereas the sensitivity of metal oxide semiconductor (MOS) gas sensors is typically sufficient. To overcome this limitation a gas chromatography (GC) column has been utilized for separation of VOCs prior to the MOS sensor [1, 2]. However, sampling and injection of samples into the system still remain a challenge, in particular for inexpensive portable devices, as well as poor quality of the chromatograms [3] resulting from large dead volumes in the system affecting the acceptance of such systems so far.

In this study, a GC-MOS system using a metal-organic framework (MOF) for pre-concentration and sharp sample injection into a GC column is presented and field validated in the food industry to determine the freshness of hazelnuts.

Technological realization

Fig. 1 shows the schematic of the GC-MOS system consisting of a pre-concentrator as injector, a GC column, and MOS sensor as detector. The

entire system design is optimized for a small bill of materials and low integration and operating costs. As pre-concentrator and sampling unit a MOF, i.e., UiO-66-BDC, on a ceramic heater substrate (Umweltsensortechnik, Germany) was used, similar to the solid phase micro extraction of common GC-MS approach. A membrane pump (NMP03KPCD, knf Neuberger, Germany) enables a constant sampling flow of the headspace of a sample onto the pre-concentrator. A portion of the desorbed volatiles are injected into the separation column (3-meter oxygen-resistant FS-5MS Supreme with 250 µm ID) column by a 3/2 cartridge valve (Bürkert, Germany). The column is temperature controlled by a custom-built GC-oven between 30 °C and 100 °C. During analysis and for flushing the column, a second pump is used downstream the MOS sensors which provides a negative pressure in the sensor chamber. This pressure gradient over the separation column leads to a constant flow through the column. As detector a MOS sensor, i.e., GGS1530 (Umweltsensortechnik, UST), is employed. The setup allows for an optional make-up flow in the sensor chamber for, e.g., rapid flushing. The GC system was optimized in terms of low dead volume allowing for sharp peaks.

Measurement

For validation of the GC-MOS system, a test measurement with an aldehyde mixture was performed. Five aldehydes, i.e., propanal up to

heptanal with 25 ppm each were solved into rapeseed oil. The expected headspace concentrations are 40.7, 11.5, 2.9, 0.9, and 0.2 ppm, respectively.

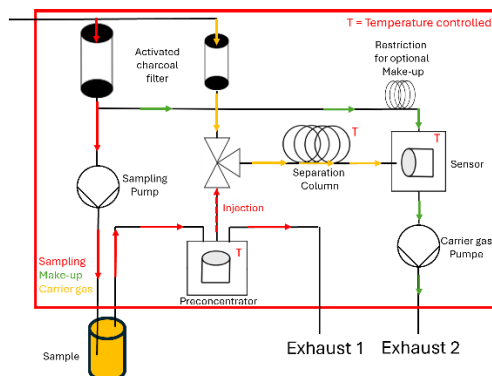


Figure 1: Schematic of the GC-MOS system.

A droplet (~1 mL) of this mixture was put onto a tissue and put into a 50 mL borosilicate sampling vial. The sampling flow was 200 mL/min which lowers the estimated headspace concentration. The headspace was sampled for 150 s at 20 °C. After this period the pre-concentrator was heated to 200 °C for 30 s for desorption and the first 3 s of this period were injected onto the column. The column and the sensor were temperature controlled at 35 °C and 400 °C, respectively.

Results

Figure 2 shows the sensor response and the derivative of the sensor response of the GGS1530 at 400 °C for five consecutive injections of the aldehyde mix. It was observed that the intensity of propanal and butanal decline strongly with each consecutive measurement. This is due to the considerably higher volatility compared to other aldehydes in the mixture. The peak for hexanal and heptanal was quite stable over the measurement. Each aldehyde was well detected by the GC-MOS system.

In addition to the aldehyde mix, the fat oxidation, and with that the decay of freshness, of real hazelnuts harvested in 2023, 2024, and 2025 were tested. The derivative of the sensor response in Fig. 3 shows a clear peak for hexanal and octanal increasing with the age of the nuts – both known as biomarkers for the oxidation process.

Discussion

The results of the aldehyde mix proved that the GC-MOS system can separate each substance of the mixture within 180 s cycle time. This is a reasonable duration for the quality control in the food industry. The system was further validated with real food samples, i.e., hazelnuts. The unambiguous determination of hexanal as biomarker proves the suitability of the system for food quality applications.

Preliminary experiments (data not shown in this abstract) with propofol (intravenous anaesthetic) and pentanal (biomarker for oxidative stress and lung injury) have shown, that concentrations in the single digit ppb range can be detected reliably. This underscores the systems versatility and potential for medical applications.

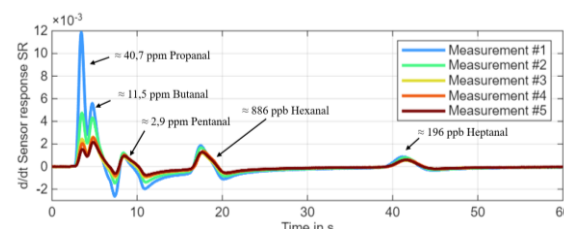


Figure 2: Derivative of the sensor response at 400 °C for five consecutive injections of the aldehyde mix.

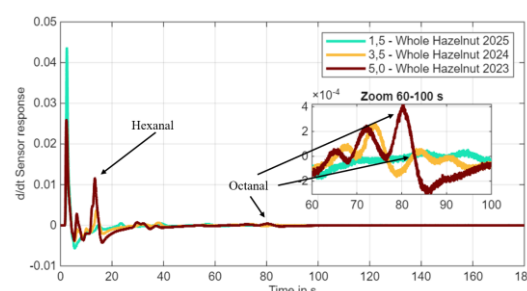


Figure 3: Derivative of the sensor response at 400 °C for hazelnuts (different harvest years) with some degree of oxidation due to age.

With different pump and make-up-flow setups a better separation is achievable. Further optimization of system parameters (i.e., sampling temperature and adsorption time of the pre-concentrator) will allow for measuring much lower concentrations. However, the presented results are meant to be a proof of principle.

Acknowledgement

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