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Characterization of a thick film MOS gas sensor as detector of short trace gas pulses

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Summary:
This study introduces a thick film metal oxide semiconductor (MOS) sensor as a promising alternative to traditional gas chromatography (GC) detectors. A gas mixing apparatus was used to simulate gas elution from a GC column with a custom measurement chamber for optimal coupling of sensor and gas flow. The experiments achieved precise quantification of ethanol at concentrations in the ppb range, utilizing a MOS sensor temperature profile for sensitive peak detection. The results show that MOS sensors offer a cost-effective and reliable substitute for traditional GC detectors in analytical applications.

Keywords: MOS sensors; gas chromatography; ethanol; differential surface reduction (DSR)

Motivation

Many applications, e.g. for quality control of food, today rely on either human sensory solutions or laboratory analytic devices, i.e. gas chromatography mass spectrometry (GC-MS). These solutions lack either an objective analysis or entail high cost without real-time capability. In the pursuit of mobile gas analytic devices, MOS sensors combined with gas chromatography utilize the sensors excellent sensitivity in combination with the columns selectivity and emerge as a promising approach [1]. This work investigates the capabilities of a thick film MOS sensor (custom GGS 1530 T without Gaze, UST Umweltsensortechnik GmbH, Geratal, Germany) as a GC-detector, which offers significant advantages such as high sensitivity to VOCs, small size and low cost. A temperature profile is employed to enhance the sensitivity of the MOS sensor [2] based on the Sauerwald-Baur model [3].

Setup and Methodology

A gas mixing apparatus (GMA) is used to simulate peaks eluting from a GC column [4]. Two gas lines enable a constant flow of 500 ml/min at 50 % relative humidity. A predilution line injects 10 ml/min of an adjustable ethanol concentration with a valve (Type 6624, Bürkert GmbH & Co. KG, Ingelfingen, Germany) into the total flow. The GMA is connected to a measuring chamber that guides the flow into the housing of the sensor, minimizing dead volumes. The schematic of the setup is shown in Fig. 1.

The MOS sensor is run in a temperature profile with a sampling period of 10 ms using custom electronics [5]. The temperature profile includes three high temperatures (400 °C) and three low temperature stages (150 °C, 200 °C, 250 °C), with a total duration of 15 min. During each five minutes interval, while the sensor is at low temperature, the gas valve is opened for 5 s between 50-55 s. Ethanol is injected at concentrations of 10 ppb, 30 ppb, 100 ppb and 300 ppb. Temperature profile and sensor response of the MOS sensor are shown in Fig. 2.

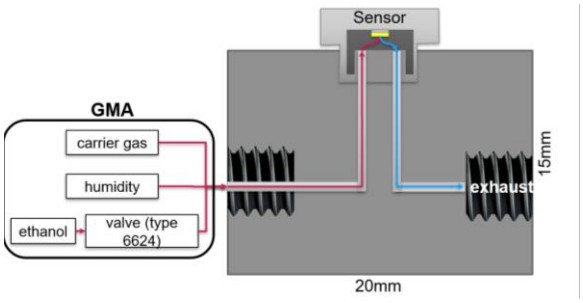

Fig. 1: Schematic of the setup including the section view of the sensor housing.

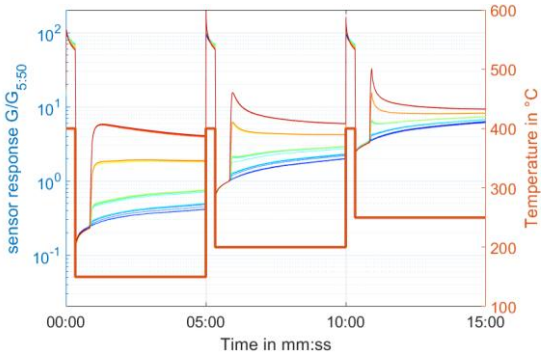

Fig. 2: Temperature cycle and sensor response $\frac{G}{G_{5:50}}$ of the MOS sensor.

Results

The ethanol injections result in different sensor responses in terms of shape and relative conductance change depending on the temperature plateau (cf. Fig. 2). Even the lowest injected dose of 50 ppb·s (10 ppb for 5 s) is easily distinguishable from the baseline. Fig. 3 shows 12 repeated injections for each dose, showing highly reproducible results in the sensor response with a change of conductivity of up to one order of magnitude for a dose of 1500 ppb·s (300 ppb for 5 s).

The sensor response for the lowest doses 50 ppb·s and 150 ppb·s approximately follows the baseline after the injection with a small offset. This has previously been observed in [6] with the differential surface reduction (DSR) method for MOS sensors under a similar temperature profile and confirms the Sauerwald-Baur model for low concentrations.

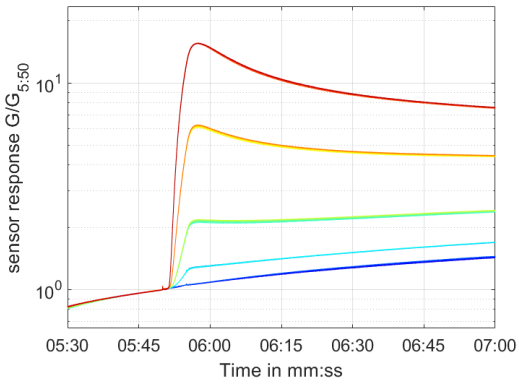

Fig. 3: Sensor response for 60 injections with the doses of 0/50/150/500/1500 ppb·s between 5:30 min and 7:00 min at the 200 °C plateau.

Fig. 4 shows the change of the sensor response vs. dose and a linear fit. Start and end values to calculate the change in sensor response are determined by the inflection points of the sensor response in the opening interval of the valve. The change of sensor response shows excellent linearity over the tested range.

It is obvious that different models are required for different injections dose ranges. Higher doses will strongly deplete ionosorbed oxygen on the sensitive layer according to the Sauerwald-Baur model. This can be observed by a decrease in the sensor response after detection of higher doses. Peaks that deplete the sensitive layer of oxygen could negatively influence the detection of following peaks, i.e. reduce the response. This is a challenge that needs to be addressed in the realization of portable GC-MOS systems. The MOS sensor nonetheless excels in reliably detecting even the smallest doses of substance.

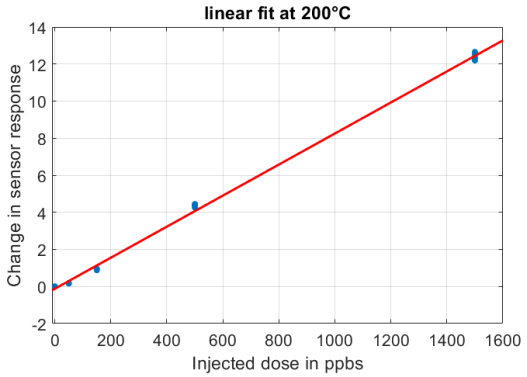

Fig. 4: Linear fit of sensor response change for different injections. $p_1 = 0.0084$; $p_2 = -0.122$; $rsquare = 0.998$ *corresponding to* $\approx 23\,ppb \cdot s$.

Conclusion

In conclusion, the MOS sensor exhibits highly sensitive and reproducible results as detector for gas peaks in temperature programmed operation. This demonstrates the capability and potential for MOS sensors as detector in gas chromatography systems.

Further studies will include the quantification ability of the MOS sensor with coeluting substances, i.e. multiple GC peaks.

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