DOI 10.5162/EUROSENSORS2025/TP20

Rapid detection of olive oil markers using a gas chromatography sensor system

Omar Tarek Penagos Carrascal[1,3], Jan Bröckelmann[2], Tilman Sauerwald[1,3], Mark Bücking[2], Gina Zeh[1]
[1] Fraunhofer Institute for Process Engineering and Packaging IVV, Department of Sensory Analytics and Technologies, Giggenhauser Strasse 35, 85354, Freising, Germany
[2]Fraunhofer Institute for Molecular Biology and Applied Ecology IME, Auf dem Aberg 1, 57392 Schmallenberg, Germany
[3] Saarland University, Campus A5.1, 66123 Saarbrücken, Germany
omar.tarek.penagos.carrascal@ivv.fraunhofer.de

Summary:
A gas chromatography (GC) sensor system is proposed for the rapid analysis of olive oil. Alterations of olive oil were analyzed, with hexanal, *E*-2-hexenal, *E*-3-hexenyl acetate, and nonanal being identified as key marker compounds for its quality. These four key markers were successfully separated on a 3 m semi-polar GC column in 1 min. The sensor (SGP40, Sensirion AG), coupled to a polar GC column, showed good reactivity towards the four identified marker compounds, although with considerable tailing (peak tailing factor ~4.3), which can be mitigated by applying the first derivative to the sensor signal.

Keywords: Olive oil, gas chromatography, sensor system, quality control

Introduction

Olive oil is a food of great nutritional value due to its high content of mono- and polyunsaturated fatty acids, in addition to being a source of vitamins. The price of olive oil has experienced an increasing trend due to its demand and a harvest shortage. This makes it vulnerable to adulteration, whereby fractions of high-quality olive oil are mixed with lower quality oils. It is therefore crucial to detect counterfeit products to protect its market and the general public [1], [2].

Several analytical methods exist for evaluating olive oil. Gas chromatography-mass spectrometry (GC-MS) is the best known and most robust, especially for determining the origin of the oil [1]. However, instruments are expensive and must be operated by qualified personnel. To make such analyses, less expensive, and easier to perform a gas chromatography sensor system is proposed, with acceptable losses in performance.

Defining which substances are target markers and or interferents is essential as it helps the scope of the system being developed to be determined. Within this scope, the detection capability of the sensor and the resolution of the chromatography must be finely tuned to achieve the fastest and most reliable measurements.

Some of the results obtained during the conceptualization and demonstration of the proposed system are shown in this paper.

Materials and results

A baseline measurement was carried out to identify the main markers of olive oil using a commercial product as standard in a dynamic headspace-thermal desorption-GC measurement with a semi-polar Rtx-1701 GC column (L: 30 m, ID: 0.25 mm, coating: 0.25 µm). Based on this measurement, hexanal, *E*-2-hexenal, *E*-3-hexenyl acetate and nonanal were chosen as reference markers for the subsequent steps of the study.

In a second step, solid-phase microextraction (SPME) GC-MS measurements of the identified marker compounds dissolved in sunflower oil were performed. These were carried out on a semi-polar column (OV-1701, L: 3 m, ID: 0.1 mm, coating: 0.1 µm) to determine the time range that an olive oil measurement could take on a column with these characteristics. The results are shown in Figure 1. Two temperature programs were used:

- Fast program: 35 °C held for 0.35 min, then heated up to 200°C at 100°C/min with 1 mL/min helium carrier gas.
- Slow program: 35°C held for 2 min, then heated up to 100°C at 5°C/min, then up to 250°C at 25°C/min with 0.25 mL/min helium carrier gas.

These measurements showed that the separation would take between 1 min (fast program) and 7 min (slow program). The shortest difference in retention times in both programs is 11 s between *E*-3-hexenyl acetate

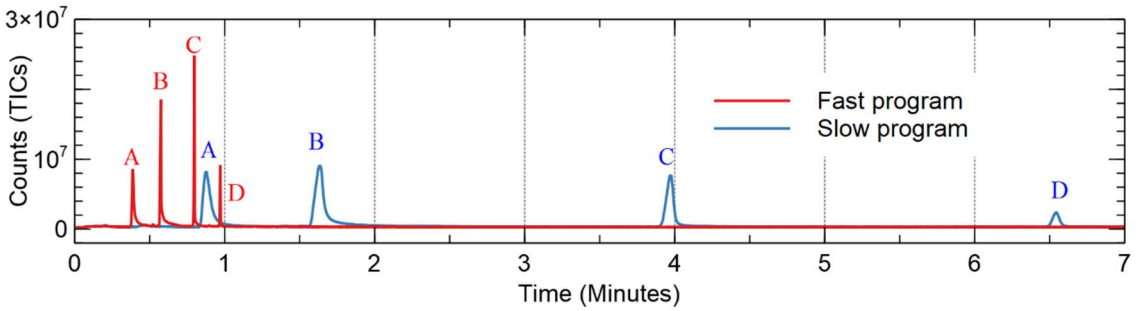

Figure 1: comparison between fast and slow programs for SPME-GC-MS with a 3 m GC column. A: hexanal, B: E 2 hexenal, C: E 3 hexenyl acetate, D: nonanal.

and nonanal for the fast program and around 45 s between hexanal and *E*-2-hexenal for the slow program.

Figure 2 shows the results of a SPME-GC-SOMSA (Selective Odorant Measurement by Sensor Array) [3], (polar column, DB-FFAP, L: 15 m, ID: 0.25 mm, coating: 0.25 µm) measurement of a spiked solution of the four previously identified markers in sunflower oil. The Sensirion SGP40 sensor showed good reactivity to the selected markers, emitting signals at similar times to those shown by the GC-MS. Considerable tailing (peak tailing factor around 4.3, peak full width at half maximum FWHM: 26-30 s) can be observed in the raw data. After performing a first derivative of the sensor signal, the peaks become more recognizable (FWHM: 3-5 s).

The peak widths of the derived signals are significantly narrower than the distance between the two closest peaks, even in the 1-minute GC run. This suggests that a GC-MOS sensor system may be able to perform quality control measurements of olive oil within one minute. However, further evaluation is needed to determine whether the derivative-based signal processing method remains effective when peaks partially overlap.

Outlook

The sampling method is currently under investigation as it has a direct impact on the analysis time and the quality of the results. To achieve fast and satisfactory measurements, a balance must be found between reduced sampling times and reduced peak intensities.

Next steps include the evaluation of sensors integrated with the optimized short GC column and method (cf. Figure 1).

References

[1] E. Kakouri, P.-K. Revelou, C. Kanakis, D. Daferera, C. S. Pappas, and P. A. Tarantilis, *Foods*, vol. 10, no. 7, Art. no. 7, Jul. 2021, doi: 10.3390/foods10071565.

[2] “Olive oil - European Commission.” Accessed: Apr. 08, 2025. [Online]. Available: https://agriculture.ec.europa.eu/data-and-analysis/markets/price-data/price-monitoring-sector/olive-oil_en

[3] M. Koehne, O. T. P. Carrascal, T. Sauerwald, and G. Zeh, *Vorträge*, pp. 230–235, Jun. 2024, doi: 10.5162/sensoren2024/C2.3.

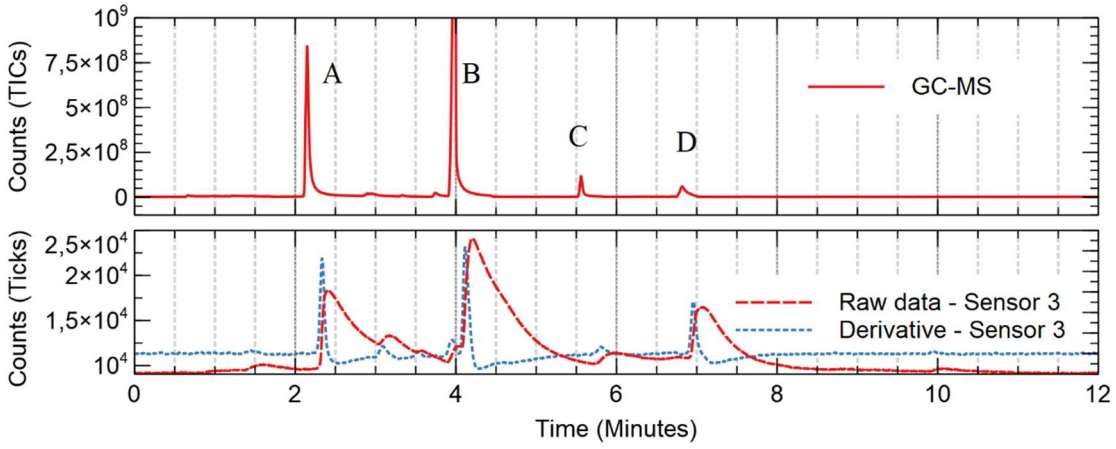

Figure 2: Measurement of the four identified markers in sunflower oil by GC-SOMSA. Top: Signal provided by GC-MS, Bottom: Signal provided by layer 3 of SGP40 (300°C) and derivative of signal. A: Hexanal (200 µg/mL), B: E-2-Hexenal (400 µg/mL), C: E-3-Hexenyl acetate (20 µg/mL) and D: Nonanal (5 µg/mL)