

MOS Sensors Characterization for Fermentation Monitoring Using GC-SOMSA

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Summary:

Headspace monitoring is a promising strategy for fermentation control, with ammonia representing a key fermentation marker. In this work, MOS sensor characterization for headspace measurement of aqueous ammonia solutions (pH > 10) was carried out using gas chromatography mass spectrometry (GC-MS) coupled with a sensor array. All four sensors exhibited a power law response to ammonia concentration, with one (SGP40, Sensirion) achieving a limit of detection below 1 ppm. These results establish a foundation for the characterisation of MOS sensor for fermentation processes.

Keywords: GS-SOMSA, MOS, fermentation, ammonia.

Background, Motivation and Objective

Monitoring of fermentation processes in the headspace has become increasingly attractive to standardize products characteristics, detect faults and optimize the process [1]. Fermentation refers to the enzymatic conversion of organic substrates by the action of microorganisms into a variety of metabolites [2]. Among the various metabolites, ammonia is widely reported, particularly as a product of the fermentation of protein-rich foods such as cheese, natto, and miso [2]. Metal oxide semiconductor (MOS) sensors can be an inexpensive and compact technology to monitor a large range of metabolites generated throughout the fermentation processes including ammonia [3]. However, due to their low selectivity and lack of specificity, a sensor characterization towards the target analytes needs to be carried out to identify suitable sensors and their operating modes as well as other potential system components for sampling and separation [4]. To this end, the gas chromatography selective odorant measurement sensor array (GS-SOMSA) has been developed [4,5] focusing on volatile organic compounds (VOCs). In this work the methodology is enhanced to the characterization of ammonia to enable a more comprehensive sensors characterization.

Materials and Methods

The GC-SOMSA system described by [5] consisted of a gas chromatograph (Trace 1610 gas chromatograph; Thermo Scientific, USA) coupled in parallel with a mass spectrometer (ISQ

7610 Single Quadrupole Mass Spectrometer; Thermo Scientific, USA) and a sensor chamber was used. For direct ammonia quantification without derivatization, the SPME-GC-MS method proposed by Zidouhia et al., 2025 was adapted to our work [6]. Aqueous ammonia was prepared in triplicate at different concentrations from ammonium hydroxide solution (Sigma-Aldrich, USA). The pH of the ammonia solutions (2 mL) were adjusted with 200 µL NaOH solution (1 M) to pH ≥10 [6]. The concentrations tested ranged from 0.5 ppm to 1000 ppm. Ammonia was extracted for 32 min at 40°C by a acrylate SPME fiber (, Sigma-Aldrich, USA) and analyzed by GC-MS using a DB-5 (Agilent Technologies, USA) capillary GC column. The sensor chamber housed an SGP40 gas sensor featuring four sensing elements. The sensor signals were evaluated by an in-house software. The heights of the ammonia signals were used for calibration plots evaluation and limit of detection estimation. The estimated limit of detection was obtained by comparing the peak height of water blank (adjusted at pH >10 with NaOH) with the four sensing elements when compared to the lowest concentrations of ammonia tested.

Results

The characteristic m/z-ion 16 was used for monitoring ammonia in GC-MS analyses. It was tentatively identified using the NIST database (match >90%). The retention time determined by GC-MS was used to identify ammonia in the SOMSA-chromatograms of the four sensors.

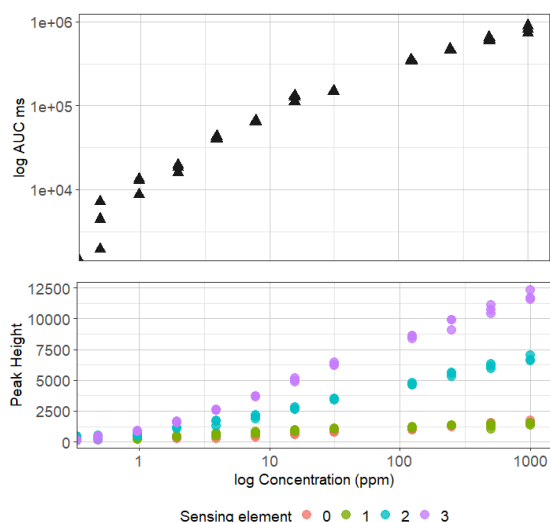


Fig. 1. Calibration plot of MS signal (logarithm of AUC for ms vs logarithm) of ammonia concentration and calibration plot for sensor response (peak height vs logarithm of ammonia concentration)

Fig. 1 shows first the calibration plot of MS results obtained using the AUC of m/z of 16. These preliminary results were used as reference to compare with the calibration curves for sensors responses. From the sensors responses, which are proportional to the logarithm of the sensor conductance [5] the peak height was derived. Subsequently, a calibration plot was constructed by plotting the peak height against the logarithm of ammonia concentration. As displayed in Fig. 1 the sensor response can be all estimated by a power law with R^2 as follow per sensing element 0 (0.9563), 1 (0.9354), 2 (0.9873) and 3 (0.9922). Following peak height extraction, the mean peak height amongst replicates of the water blank was compared to the mean ammonia peak at low concentrations (0.5, 1 and 2 ppm) as displayed in Fig. 2.

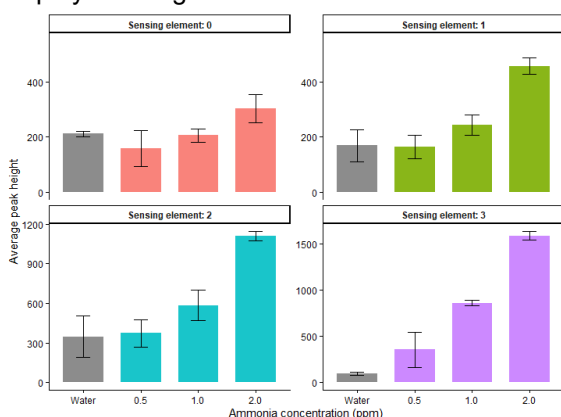


Fig. 2. Average peak height vs concentration of ammonia (0.5, 1 and 2) and water blank (grey) for the 4 sensing elements: 0: red, 1: green, 2: light blue and 3: purple

As shown in the histogram, at 1 ppm only sensing element 3 (purple) exhibited a peak height exceeding that of the water blank. At 2 ppm,

sensing elements 1 and 2 (green and light blue respectively) displayed higher peak height than the water blank, while sensing element 0 (red) didn't show a significant response below 2 ppm. The differing estimated limits of detection across the four sensing elements suggest that their selection should be tailored to the expected concentration range, as some sensing elements may be more suitable for specific applications. A method for GC-SOMSA measurements of ammonia was established. By the choice of the sampling technique and GC column it is expected that VOC key markers from fermentation can be measured with the same apparatus. This would enable a highly functional approach to characterize additional fermentation markers.

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