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Metal Oxide Semiconductor Gas Sensor Systems for Food Freshness Detection: A Review of Recent Studies

Metalloxid-Halbleiter Gassensorsysteme zur Frischeerkennung von Lebensmitteln: Ein Überblick über aktuelle Studien

Hamza Ali Imran[1,2], *Bora Ersöz*[1], *Richard Fix*[1], *Christian Bur*[2], *Andreas Schütze*[2]
[1]*Bosch Sensortec GmbH, Kusterdingen, Germany*
[2]*Saarland University, Lab for Measurement Technology, Saarbrücken, Germany*
Contact: *h.imran@LMT.uni-saarland.de*

Introduction

Throughout history, humans have relied on their sense of smell, known as the Olfactory sense, to detect food freshness. This sense is crucial for our health and safety. It consists of multiple receptors sensitive to certain gases released by decomposing food [1]. The "best-before" and "use-by" dates significantly influence consumer food choices, yet many consumers are unaware of their distinct meanings. The "best-before" references the date after which the food quality starts to degrade while "use-by" means the date after which the food becomes unsafe for consumption. The confusion in these terms is often the reason for the premature disposal of food [2]. In the European Union (EU), about 58 million tons of food waste (131 kg per inhabitant) are created yearly [3]. The EU places a high importance on the solution of this problem. The recently initiated project, SERENADE (HORIZON-MSCA-DN-2021 (Marie Skłodowska-Curie Doctoral Networks) program under Grant Agreement No. 101072846) [4], specifically addresses the last stages of the food supply chain aiming to reduce food waste at the household, supermarket, and retailer levels [4].

Metal oxide semiconductors (MOS) gas sensors have effectively been used for VOC monitoring in various applications. These applications include environmental monitoring, where MOS targets compounds like BTEX (benzene, toluene, ethylbenzene, and xylene) and trichloroethylene, which are measured from ppb to ppm levels. For Indoor Air Quality (IAQ), the sensors detect total VOC concentrations and among others toxic VOCs such as benzene and formaldehyde, with concentration ranges from sub-ppb to several ppm. In fire detection, they respond to acetic acid and inorganic gases including CO and NO_x, detecting concentrations up to 10 ppm. In industrial settings, MOS helps monitor workplace safety and emission control by detecting toxic pollutants and BTEX compounds, even at levels as high as 100 ppm. They are also valuable in health-related applications, such as detecting acetone and isoprene for diabetes diagnosis, and in odor monitoring, where they can sense organosulfur compounds and hydrogen sufide (H_2S) at sub-ppt levels. However, each application faces challenges due to interferents like CO, NO_x, O_3, and humidity, which must be accounted for to ensure accurate readings [5]. The food spoilage process produces different VOC and also other gases including CO_2 and H_2S and the pattern of these emissions changes over the entire spoilage process making MOS suitable for the detection and quantification of their spoilage process [6]. The development of sensor technology for Artificial Olfaction began in 1982 with the introduction of the first Gas Sensor Array (GSA) [7]. Designed to mimic the mammalian olfactory system, these devices, commonly known as electronic noses (e-noses). Electronic noses offer non-destructive testing, are user-friendly, compact, field-testable, allow for rapid analysis, and are cost-effective to operate [8]. Figure 1 shows a comparison of human olfactory sensory system and MOS systems. These systems are similar in several steps from detection of odors or gases (human nose vs MOS sensors), data acquisition (neural signals vs dynamic operation like Electrical Impedance Spectroscopy (EIS)), processing (brain vs machine learning), and prediction, for example, freshness/spoilage for both systems. This analogy highlights that sensor technology is inspired by biological processes.

Over time, multiple studies have attempted to use these sensors for food spoilage detection. This contribution reviews relevant studies in the literature, highlighting the application of GSAs, where a variety of MOS sensors—non-specific to particular VOCs or gases—are collectively exposed to food samples, and the changes in their resistance are recorded. Pattern recognition techniques are then employed to analyze this data. However, controlling the selectivity and sensitivity of a MOS for a particular gas is a challenging task. Therefore, alternative methods called Dynamic Operations (DOp) have been adopted but in other applications. Using DOp we can create a Virtual Sensor Array (VSA) scenario or increase both the sensitivity and selectivity of the sensors to different target gases. A renowned DOp method is Temperature Cycled Operation (TCO) [9]. Another less explored method is EIS [10]. Both of these methods are discussed in contribution.

The remaining sections of this contribution discuss food freshness studies synopsis and review, followed by that it covered the working of dynamic operations including Temperature Cycled Operation and Electrical Impedance Spectroscopy. Then it concludes with the argumentative support of adaptation, of DOp for food freshness detection application.

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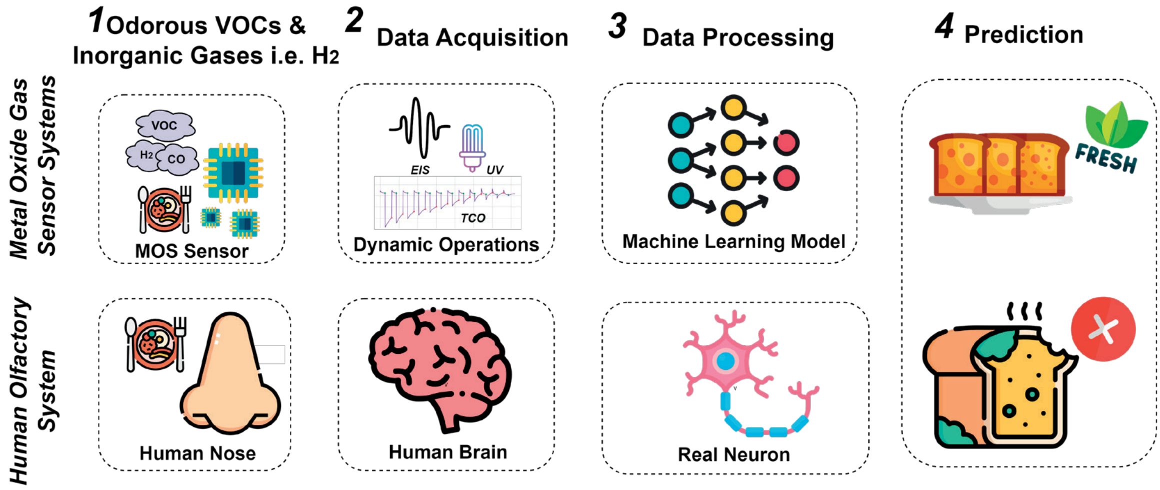

Figure 1: A Comparison between nature and technology: This visual highlights the parallel processes between the human olfactory system and metal oxide gas sensors, showcasing how both systems detect, analyze, and interpret odors—whether through neurons or machine learning models.

Food Freshness Detection Studies - Synopsis

This section presents a review of recent studies on food freshness detection with MOS sensors. The research conducted by Sanaeifar et al. [11] assesses the capability of an e-nose system, to forecast various banana quality indicators such as total soluble solids (TSS), titratable acidity (TA), pH, and firmness at different stages of shelf-life. The study used partial least squares (PLS), multiple linear regression (MLR), and support vector regression (SVR) approaches to find correlations between the e-nose responses and quality metrics. The results illustrate that Support Vector Regression (SVR) models have superior predictive capabilities, suggesting that the e-nose system is a dependable instrument for assessing the chemical and physical characteristics of bananas.

Masi et al. investigated the raspberry deterioration process over five days at 23 ℃ room temperature [6]. Eleven distinct substances that are commonly released by food were included in random gas mixtures that were used to calibrate the sensors. A Partial Least Square Regression (PLSR) model was trained using the data from these calibrations to forecast the concentrations of these volatile organic compounds. Principal Component Analysis (PCA) was used to reduce the dimensionality. The Root Mean Squared Error (RMSE) of the model, which was tested for ethanol detection using a hold-out test and 10-fold cross-validation, was roughly 223.9 ppb. This study is among the first to include the dynamic operation of TCO in the context of food spoiling.

Tang et al. [12] used a GSA to determine freshness in chicken meat. The aim was to develop a rapid, non-destructive, and accurate method for the evaluation of freshness in chicken flesh by measuring total volatile basic nitrogen (TVB-N) contents. This measure works as a measure for the release of ammonia and amines due to microbial activity. This study investigated chicken meat kept at 4 ℃ for 5 days. Along with sensor measurements, the levels of TVB-N with chemical methods were also conducted. Initially, PCA was implemented for data preprocessing but lacked discriminative power. Double-layered Cascaded Serial Stochastic Resonance (DCSSR) approach was adopted to improve the Signal-to-Noise-Ratio (SNR) and required better differentiation performance in meat freshness classification. They derived a predictive model associating TVB-N levels with the SNR eigenvalues retrieved from DCSSR analysis. This model yielded an impressive accuracy of 93.3% concerning the freshness of meat.

Raudiene et al. proposed a prototype e-nose system built with GSA [13]. This system was designed to detect the freshness of chicken meat. The study conducted a comparison between the data obtained by an e-nose and traditional chemical measures. The main focus of the comparison was on Volatile Fatty Acids (VFA), which are indicative of meat decomposition. The e-nose developed had a correlation of (R^2 = 0.89) with conventional techniques, indicating its potential for practical use in evaluating the freshness of meat. This system claims to provide an uncomplicated, cost-effective, and expeditious approach to assessing the quality of chicken meat.

A study by Han et al. [14] proposed assessing the benefits of employing an e-nose in conjunction with an electronic tongue (e-tongue) for detecting the freshness of fish meat. A three-layer Neural Network was designed to qualitatively distinguish the freshness of seafood. PCA was employed to decrease the size of the input based on GSA consisting of nine sensors, all of which were obtained from Figaro Inc., Japan. Regarding the e-nose instance, a discrimination rate of 80.0 % was reached. Their performance significantly improved when they utilized both the e-tongue and e-nose simultaneously. The Total Viable Count (TVC) which is a measure used to determine the total number of viable microorganisms was quantified using biological analysis, and a correlation coefficient of 0.91 was observed between TVC and both the ee-nose and e-tongue.

Guohua et al. [15] researched to evaluate the efficacy of utilizing an e-nose for detecting the freshness of grass carp (Ctenopharyngodon idellus), a widely eaten fish in the Chinese market. They utilized an e-nose equipped with eight sensors to differentiate between the states of the fish, specifically fresh, somewhat fresh, and rotten. The findings obtained from the e-nose were compared with the assessments of TVC. Achieving an overall prediction accuracy of 87.5 %, predictive modeling was built using nonlinear regression based on the maxima of the signal-to-noise ratio (SNR).

Zhang et al.'s study [16] proposed a novel MOS sensor that used a (001)TiO2/Ti3C2Tx (MXene) heterostructure to create a UV-enhanced ammonia gas sensor. The sensor leverages the (001) crystal plane of TiO2, which has a high affinity for ammonia. The sensitivity of the sensor is greatly increased by adding UV light, which improves electron-hole pair separation at the Schottky junction between TiO2 and Ti3C2Tx. This sensor is intended to identify ammonia (NH3), a crucial marker of food deterioration, especially in foods high in protein. They tested it on meat from shrimp, fish, and pigs. When the sensor is exposed to UV light, its performance is 34 times higher than when Ti3C2Tx is used alone. It has a detection limit of 156 ppt and fast response/recovery times of 10 and 5 seconds, respectively. A proof of concept project was created utilizing a sensor and a microcontroller equipped with three-level LED indicators. Ammonia concentration thresholds of 1 ppm and 5 ppm were selected to denote the start and end of meat deterioration, respectively.

Xiong et al. [17] used a sensor array consisting of MQ135, MQ136, MQ137, and MQ138 from Zhengzhou Weisheng and TGS2602 from Figaro. They converted the sensor array data into images by applying multiple transformations and used transfer learning to transfer learned popular convolutional neural networks including GoogleNet, AlexNet, and ResNet. The important point here is that the models were originally trained on the ImageNet dataset, which is the dataset of image classification. In principle, transfer learning should only be applied to domains of a similar nature which is not the case in this study. However, they reported the best classification accuracy of 99.70% with ResNet.

The study [18] investigates the application of electronic nose (e-nose) and electronic tongue (e-tongue) systems for detecting pesticide residues in fruits including cape gooseberries, apples, plums, and strawberries. The e-nose system, consisting of 16 metal oxide gas sensors from Figaro, detects VOCs emitted by the fruits. Data analysis was performed using PCA and Linear Discriminant Analysis (LDA), coupled with various classical machine learning algorithms. The e-nose system demonstrated high effectiveness in classifying pesticide-contaminated fruits achieving an accuracy of 85.7 % for cape gooseberry, 90.5 % for apple, 76.2 % for strawberry, and for plum it was 95.2 %

The MAU-9 e-nose system is used in the Rasekh et al. study to distinguish between two types of essential oils [19]: fruit oil (mango, orange, and lemon) and herb oil (thyme, tarragon, and mint). A variety of Figaro MOS gas sensors enable the identification of particular VOCs like alcohols, ammonia, and methane. PCA, LDA, QDA, and SVM were among the algorithms used to examine the data produced by the e-nose. All oils were perfectly classified by LDA, QDA and SVM, which also showed precision (up to 99%).

A novel odor sensing system is proposed in study [20], which investigates a non-destructive method to test the quality of beef. The study measured the smells of beef at various cooking temperatures (room temperature, boiling, and frying) and storage times (one to eight days). This 16-channel MOS sensor array was used to track odor changes. Gas chromatography-mass spectrometry (GC-MS) was then used to identify particular compounds, like acetoin and pyrazines, that may alter with storage and temperature. The study used machine learning techniques, such as dimensionality reduction techniques like PCA and UMAP, to process the sensor data and classify beef odors. Supervised UMAP greatly improved classification, reaching over 99.5% accuracy, while unsupervised methods only achieved moderate accuracy. The results suggest that this system could reliably monitor beef freshness and safety, offering a quicker and non-invasive alternative to traditional microbial testing.

Dynamic Operation

This section shares details on TCO and EIS dynamic operations. This enables us to create multiple profiles from a single sensor and also tweak the selectivity and sensitivity of the MOS sensors.

Temperature Cycled Operation

TCO is a well-studied technique that could significantly enhance the selectivity and sensitivity of metal oxide semiconductor sensors. This process entails regularly altering the temperature of the sensor, resulting in non-equilibrium conditions on the sensor surface. The surface temperature exerts a substantial influence on the chemical interaction occurring between the sensor and the gas it is being exposed to. For example, methane CH_4 exhibits more stability and hence necessitates larger activation energies to initiate a response in a sensor. In contrast, gases such as CO or H_2 will respond at comparatively lower temperatures. The primary advantage of varying the sensor's temperature over a wide range is that, at some point during the thermal cycle, the sensor will reach an optimal temperature with maximum sensitivity for the target gas or VOC. Additionally, dynamic temperature cycling, characterized by rapid temperature transitions, creates non-equilibrium surface states that are unattainable under static, constant-temperature conditions. By precisely tuning the operational parameters, the sensor's sensitivity can be significantly enhanced. For example, initially operating the sensor at a high temperature promotes a high surface coverage of ionosorbed oxygen. Rapid cooling

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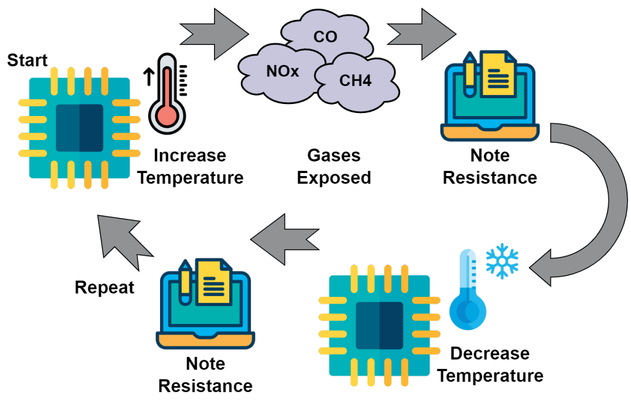

Figure 2: This illustration depicts the temperature cycled operation applied to MOX sensors for VOC detection. The process entails regular alteration of the sensor's temperature, resulting in non-equilibrium conditions on the sensor surface. The surface temperature plays a crucial role in modulating the chemical interactions between the sensor and the gases it encounters. Resistance values are recorded at both elevated and reduced temperatures, with each temperature stage enabling distinct gas adsorption and desorption behaviors

from this state results in a surface at a lower temperature with an excess of reactive oxygen ions, a state that cannot be achieved through static operation. In the absence of a target gas, the ionosorbed oxygen will gradually desorb to establish equilibrium, leaving only a minimal amount of oxygen ions at low temperatures. However, when a gas interacts with the adsorbed oxygen, it accelerates the relaxation process, leading to faster equilibration. [5]. Figure 2 shows all steps of TCO.

A study by Baur et al. makes use of exactly this methodology for IAQ application. The researchers used the SPG30 MOX sensor from Sensirion AG, Stäfa, Switzerland, which has four sensitive layers. The sensor may be digitally configured to alter temperature by 25 ℃ from 100 ℃ to 425 ℃. The TCO cycle has ten 5-second steps at 400 ℃. After that, 7-second low-temperature stages are performed at 100 ℃, 125 ℃, 150 ℃, 175 ℃, 200 ℃, 275 ℃, 300 ℃, 325 ℃, 350 ℃, and 375 ℃. Thus, the temperature cycle lasts 120 seconds. Gas concentrations were accurately controlled using a gas mixing device (GMA) during calibration. Six VOCs from different chemical groups were used for calibration. Four typical (VOCs) were used for calibration: ethanol, formaldehyde, acetone, and toluene. The calibration also employed hydrogen and carbon monoxide as interfering gases. Initial sensor data, including gas-sensitive layer resistance, is logged and converted into logarithmic conductance. In feature extraction, each temperature cycle is segmented and conductance features of mean and slope are calculated. The Partial Least Square Regression (PLSR) model trains and validates each target gas to make predictions. Recursive feature elimination selects relevant features, whereas cross-validation optimizes PLSR components and features. The sensor is calibrated regularly to correct for deviations and maintain accuracy. Merging the original calibration data with recent recalibration data improves model stability. Field testing lets VOCs out in a test room at regulated levels. Sensor data from these tests is compared to GC-MS, GC-PID, and GC-RCP measurements [21].

Electrical Impedance Spectroscopy

Other than TCO there is another method for increasing selectivity is to assess and analyze the complex sensor impedance response of the MOS when excited with AC current of different frequencies. This technique is called as Electrical Impedance Spectroscopy (EIS) [22]. One of the underlying impacts is the change in capacitance at the grain boundaries, which is induced by gas, specifically oxygen. The capacitance characteristics of the sensor layer are also influenced by the dielectric properties of the chemical species present in the sensing layer; measuring them using EIS thus increases selectivity [23]. Hence, there is a possibility to create a Virtual Sensor Array using the EIS technique. Compared to TCO, EIS is much less explored for VOC detection and quantization. For EIS the system is excited with an alternating perturbation and its response is measured in the form of a spectrum over wide frequency spectra. Two plots named Nyquist and Bode are than plotted to see the response of the system. The Nyquist plot is a plot where the real part of the impedance of the system is plotted on the axis and on the Y axis the imaginary part is plotted. Bode plot, presents two separate plots for impedance magnitude and phase over the change in frequency. Figure 3 represents the stages in EIS evaluation.

The study [23], [24] experimented with combining both dynamic operation techniques, namely TCO and EIS, to enhance sensor selectivity and enable self-monitoring. This approach helped detect sensor damage by comparing predictions. For practical implementation, a low-cost EIS solution was developed, using an FPGA for digital broad-spectrum stimulation and a high-speed ADC for recording sensor responses. Pseudo-random Maximum Length Sequence (MLS) signals were used for excitation, and the Fast Fourier Transform (FFT) was employed to calculate the impedance spectrum quickly, within 50 milliseconds, for the range of 100 kHz to 100 MHz. Experiments demonstrated effective discrimination of gases including CH_4, CO, and C_2H_4, relevant to underground fire detection, with improved selectivity and reliable self-monitoring through the combined TCO and EIS data.

Conclusion

Finally, most of the fresh-food detection study nowadays is based on static sensor operations, which have been laid with substantial challenges in selectivity and sensitivity. Metal oxide semiconductor (MOS) sensors have been used in this area, but more sophisticated dynamic operation techniques like TCO and EIS are highly under-appreciated. However, they have been shown to give significant benefits

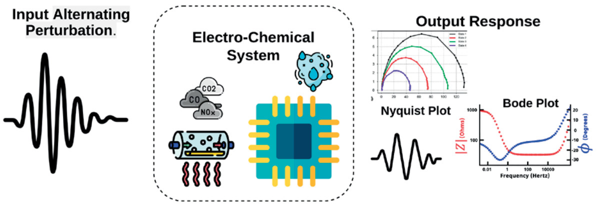

Figure 3: Illustration of EIS Process: An alternating perturbation is applied to an electrochemical system, such as a gas sensor exposed to gases like CO, NOx, and CO2. The system's response is captured in the form of Nyquist and Bode plots, providing insights into its impedance characteristics and the underlying electrochemical processes at different frequency ranges.

in other application domains such as Indoor Air Quality (IAQ) monitoring by improving sensor performance due to the gesture of increasing selectivity and sensitivity and creating virtual arrays. Dynamic operations add often expression in higher dimensions of sensor data which is beneficial for AI/ML algorithms. This increased complexity is the key to developing better AI models that can find much finer patterns in the data and make a more accurate prediction of food freshness. Moreover, limitations in the state-of-the-art also include issues such as sensor drift, interference from non-target gases (e.g., CO, NOx, O3, and humidity), and a lack of stability over prolonged periods that hinder widespread adoption. Additionally, while TCO and EIS show promise, their complexity and the need for frequent calibration present hurdles for practical deployment. Machine learning integration also encounters obstacles, including variability in sensor responses and constraints in model generalizability. In fact, most of the studies focus on untargeted measurements, i.e. analyse VOC-pattern without identifying them. Nor a selective measurement of VOCs.

In SERENADE we first, identify the marker substances, and second, build sensor systems able to measure these specific VOCs (targeted approach) by using dynamic operation strategies like TCO and EIS

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