Electronic Noses for Cyber-Physical Systems: Preliminary Results on TiO2 Thin Film as a Humidity Sensor

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Abstract. Cyber-physical systems (CPS) use algorithms to interpret sensor's responses and model the physical environment they are connected to. Among the many application domains where CPS are used, the health-related field is one of the most promising areas that can benefit from CPS. This paper considers the development of sensors to be integrated into an electronic nose, e-nose, that will be responsible for collecting the information to be fed to the cyber component of a CPS. The work presented addresses the challenge of detecting Volatile Organic Compounds (VOCs) in the air at low concentrations with the main goal of diagnosing diseases through breath samples, using an electronic nose (e-nose) concept. Since the identification process of several VOCs can be highly sensitive to the level of humidity in the breath samples, the incorporation of a humidity sensor in the e-nose is proposed as a way of providing information that can be used to guarantee the accuracy of the results to be obtained. To this effect, a TiO_2-based sensor was tested as a solution, and preliminary results are presented. Results demonstrated clearly that it is necessary to maintain the e-nose at a controlled humidity to ensure that the responses from the different e-nose sensors are correct and reliable.

Keywords: e-nose; Cyber-physical systems; CPS; sensor; VOC; TiO2; humidity.

1 Introduction

Cyber-physical systems (CPS) are becoming a mature technology where the physical world is integrated with the cyber (computational) world [1]. As illustrated in Fig. 1, sensors play a fundamental role in CPS, being responsible for collecting data to be transmitted to the cyber system [2]. The collected data is then processed and analyzed in the cyberworld through the cloud and serve as a stimulus to control actuators.

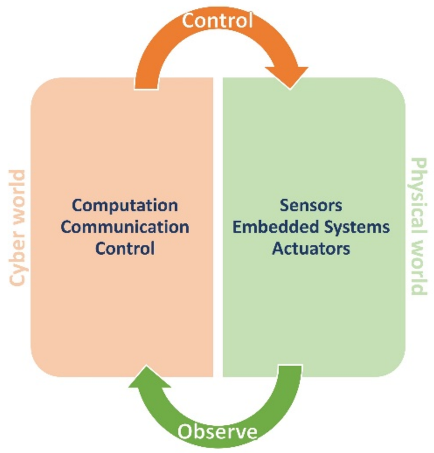

Fig. 1. Cyber-physical Systems overall structure. *Adapted from* [2]

CPSs have been proposed in the literature for a wide range of applications scenarios, such as smart manufacturing, emergency response, critical infrastructure (i.e. distribution of daily life supplies such as water or electricity), healthcare and medicine (i.e., monitoring heath conditions) or Intelligent Transportation (i.e. improving coordination in traffic management), among others [3]. In the particular case of healthcare, the patient's critical data may be collected by sensors and sent through either wired or wireless communication to the cloud, where it is processed, and relevant information regarding the patient's health status is evaluated. This information may be sent back to the CPS actuators to conduct the needed control measures. Given the specificity of the data to be collected healthcare CPS face specific challenges such as the need for granting the interoperability of the sub-systems that feed the patient health condition (e.g., simple/large image data *such as blood sugar level/ magnetic resonance imaging*, personal health history), as well as the accuracy of the evaluation results and the need for securing the protection of the data privacy.

In the process of collecting information regarding patient's health status, one major concern is the need to employ noninvasive techniques. A solution is the use of volatile organic compounds (VOCs) sensors. VOCs are compounds that have a high vapor pressure and low water solubility. In health-related applications, VOCs are usually organic substances that can be found in the air at small concentrations, i.e., parts per trillion (ppt) or parts per million (ppm) [4–7]. Because of this, our main goal is the develop an array of sensors for the detection of the existence of VOCs, associated with the presence of prostate cancer (PCa) through breath samples, as demonstrated in [7]. To collect samples, the patient could blow into a balloon with the sensor array. As such, my PhD work consists of preparing a system that can diagnose PCa by detecting those VOCs.

However, the breath samples are also composed of water vapor at different concentrations that influence the electrical response of the different sensors. So, this work focuses on a humidity sensor, based on TiO_2 thin films, to measure the relative humidity existing in the exhaled air to answer if affordable sensor arrays can be an alternative to powerful yet expensive sensors, the sensitivity to humidity of a metal oxide sensor will be studied.

Besides this introduction, the challenges associated with the use of e-noses, as well as the need for using machine learning techniques for subsequently identifying significant patterns and trends in the information to be used in CPS are addressed in Section 2. Section 3 is devoted to the literature review, and then the work developed is presented in Section 4. Finally, results are discussed in Section 5, whereas conclusions and future work are presented in Section 6.

2 VOC Identification with AI-Powered Medical CPSs

Cyber-Physical Systems consider the integration of physical processes with computation and communication. In the case of Medical Cyber-Physical Systems (MCPS), they may comprise robotics and sensor systems with data analysis to create feedback systems that may interact with patients, physicians, or other healthcare professionals. The physical component of CPS for healthcare considers the use of biomedical sensors that are responsible for collecting the relevant physiological data to be fed to the computation and communication system [8].

Over the last decade, systems comprising an array of electronic chemical sensors and an appropriate pattern-recognition module have been proposed as a way of providing an inexpensive way to analyse either gas or aqueous solutions. In the case where gas samples are analysed, these systems are usually referred to as electronic noses (e-noses) [9,10]. In MCPS, e-noses can be used for diagnosis, monitoring, or phenotyping diseases, with the advantages of being non-invasive and inexpensive.

In diagnosis, e-nose sensors aim at identifying VOCs that can be detected in the exhaled breath. These volatiles may be produced either by cellular metabolic processes or inhaled/absorbed from exogenous sources [11] and the identification of an augmented concentration may lead to the identification of a specific disease. The PhD thesis encompassing this article considers the development of sensors to be integrated into an e-nose system for PCa detection through breath samples.

Another challenge associated with MCPS arises from the need to analyse very large datasets provided by the sensorial system. To overcome this problem, the Principal Component Analysis (PCA) [9] will be used in the pattern recognition module of the e-nose. This will enable the reduction of the dimensions of a dataset while preserving most of the variance in the first two components it generates [12–14]. However, the use of AI techniques for subsequently obtaining more relevant information is envisaged [15–21].

3 Literature Review

The evaluation of the concentration of VOCs has raised a relevant interest in the research community since it can serve to protect humans (e.g., in the detection of hazardous substances such as ammonia) [22–25] and to diagnose diseases, as their existence can correlate to some well-identified process within the body [26–33]. In the case of human protection, the Occupational Safety and Health Administration (OSHA) [34] as well as the European Agency for Safety and Health at Work have established the exposure limits to those VOCs. To diagnose diseases, sometimes just monitoring one VOC can serve as a good indicator [29]. This usually happens when high concentrations (in the ppm) can only be found when the patient is afflicted by a specific disease. However, the precision and range of diseases that VOCs can be used to diagnose can be increased and expanded, respectively, by detecting combinations of VOCs in such cases where the disease can be identified by a unique set of VOCs [6,7,35].

When identifying VOCs, the most common method used is Gas chromatography–mass spectrometry (GC–MS) [9,36]. This method can accurately detect multiple VOCs [26,36–38]. However, it is expensive, and the samples can suffer thermal degradation [39]. As such, other sensor technologies are being explored as alternatives. Among these, the most common are metal-oxide (MO/MOX) or metal-oxide semiconductor (MOS/SMO), field-effect transistor (FET), Conducting Polymers (CP), and other nanomaterials [9,10,40].

MO and FET are affordable while having high operation temperatures [40–43]. Conducting Polymers and nanomaterials have low limits of detection at room temperature, while lacking mechanical strength [44–46]. Electrochemical cells have low power consumption while suffering from poor selectivity [47,48]. Currently, with these different strengths and weaknesses, no technology has been proven to be the most effective for VOC detection, and, as such, each one of them keeps being developed.

Another relevant approach for VOCs detection is the use of e-noses [9,10,49]. These consist of a sensor array that collects data and a pattern recognition unit that extracts relevant features from the multivariable analysis and extrapolates on them to make predictions [50]. This technology has been used for medical applications [14], and it is appropriate for VOC detection as it can enhance the sensors to reach low detection ranges in the presence of a complex matrix [10,51].

Due to the large dimension of the data generated, methods for selecting the most relevant actors are usually needed. Over the last years, PCA has been the most popular algorithm since it can create areas associated with a class and correlate one or more components with the desired substance. The application of PCA in VOC detection has led to very promising results [26,27,37,38].

As breath samples are practical to take, their study is of high interest. However, they have a high variance in relative humidity and temperature [52], both these factors affect the response of most MO and CP based sensors [53–55].

4 Work Developed

Over the last years, the analysis of exhaled breath has gained interest in the medical community. It yields the possibility for diagnosing different diseases by analysing the concentration, type, and other characteristics of different gases containing trace amounts of both VOCs and some non-volatile components, usually between one ppt and one ppm. The development of e-noses yields the possibility for identifying when one or more gas concentrations exceed a pre-defined range, indicating the presence of specific diseases.

The advantages of using e-noses for diagnosis concern their rapid identification capability, ease of use, portability, and avoidance of discomfort. However, the accuracy and reliability of the diagnosis may be hindered by the e-nose sensor's drift, which may be caused by the variance of the breath sample's relative humidity. To overcome this effect, the incorporation of a humidity sensor into the sensor array could be advantageous. In the literature, TiO_2 sensors have been demonstrated to produce good results in sensing humidity [56–59]. To explore their accuracy using the Impedance Spectra (IS) response, this sensor was chosen to experiment with.

4.1 Humidity Sensor Implementation and Results

TiO_2 is a notorious material for relative humidity (RH) sensors, as they are accurate and operate at room temperature [56–59]. Additionally, as a conducting polymer, it is a type of sensor commonly used in e-noses, making it easy to integrate into a sensor array. Due to these characteristics, a TiO_2 sensor was implemented to help the system monitor the RH and calibrate/correct the response from the other e-nose sensors. Each sensor integrates an interdigitated gold electrode on a ceramic support with a conducting polymer or metal oxide thin film, as demonstrated in [60]. The interdigitated electrodes used were IDEAU 200 from Metrohm DropSens, which have 200 microns lines and gaps.

For testing the sensor behaviour, a cylindrical vacuum chamber with a capacity of 58 dm^3 was used to produce different gas samples in a controlled manner. It consists of a glass and metal seal attached to a pulley system to raise and close the chamber itself. Additionally, it has as outputs for the vacuum pump and receiving air. The input is split and can take air from the room or from a connection to a dry air tank that includes a flask where mixtures can be inserted. Finally, it has BNC ports connected to the sensor(s) inside the chamber. This system is described in [60].

Each sensor can be sampled individually or have its own individual set of relevant features that need to be extracted. As such, measuring the Impedance Spectra response of a sensor is an appealing method when studying a sensor. There are many options for relevant features that can be used to test a sensor's performance, such as: absolute impedance, at a predetermined frequency; phase, at a predetermined frequency; poles and zeros.

As such, an experiment to test the correlation between the humidity in the chamber and the absolute impedance was conducted. The volume of ultrapure water inserted into the chamber was +0.3 ml than the last, starting at 0 ml. To measure each sample, the impedance spectra was considered, and five features were extracted. These were absolute impedance at 1 Hz, phase at 1 Hz, real and imaginary parts of the impedance at 1 Hz, and the Nyquist. In total, six samples were measured, and vacuum was made in the chamber between each sample. When sampling, three loops were measured, and the experiment was repeated twice at different room temperatures (RT). Sample 1 had a higher RT than sample 2. The Nyquist plots and the Impedance Spectra from the samples are illustrated in Fig. 2 and 3, respectively. The extracted features are illustrated in Fig. 4.

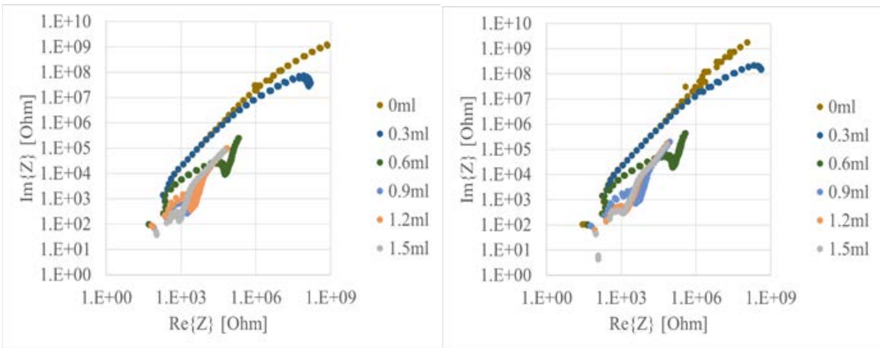

Fig. 2. Nyquist plot for samples 1 and 2 respectively.

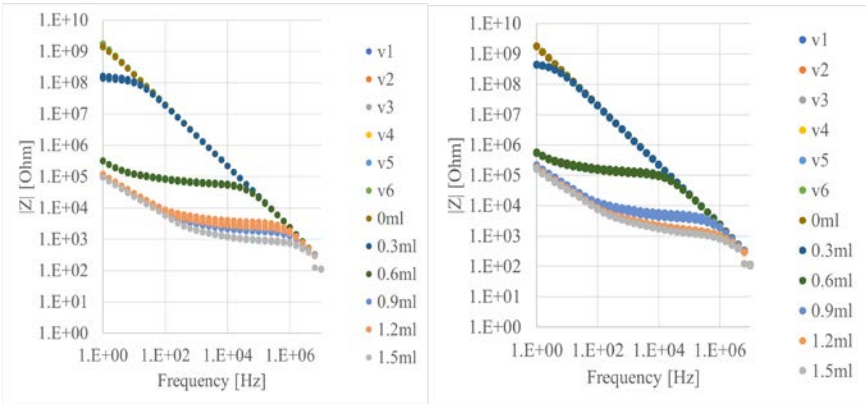

Fig. 3. Impedance Spectra, absolute impedance (|Z|) [Ohm] versus the frequency [Hz] for samples 1 and 2 respectively.

Based on these results, low-humidity samples only become conductive at low frequencies. As such the lower frequency measured was considered when studying the relation with humidity, i.e., 1 Hz.

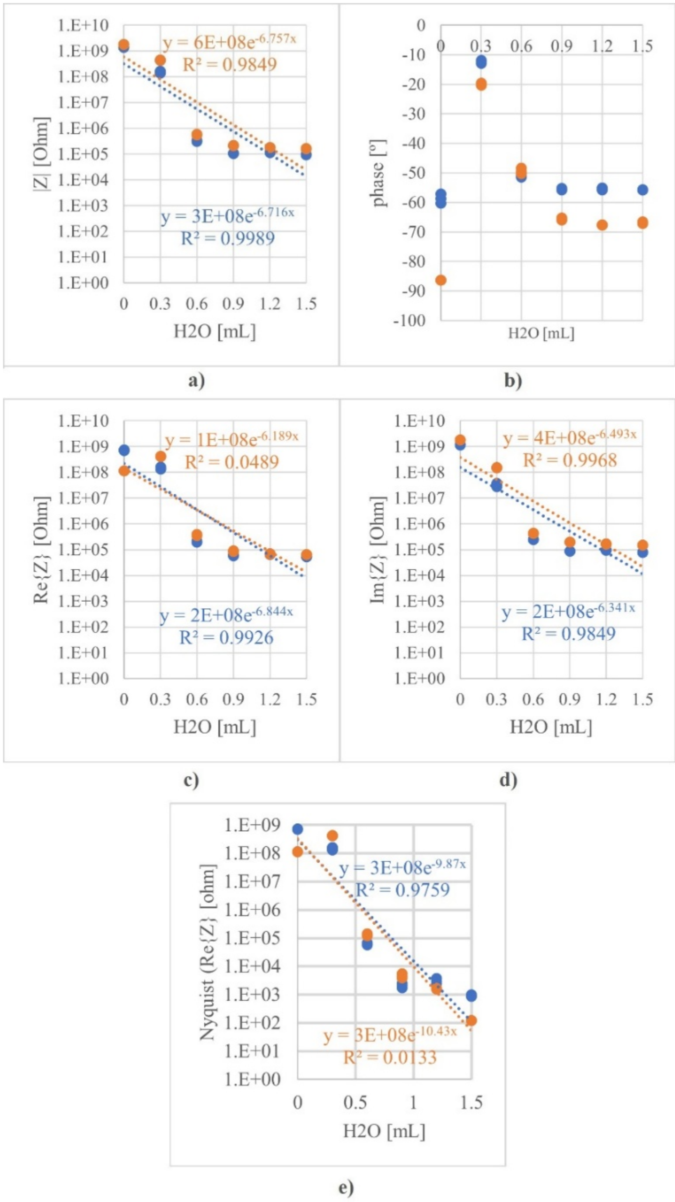

Fig. 4. Humidity response from TiO_2 film, as a function of ultrapure water inserted into the chamber [ml]. 1st sample: blue; 2nd sample: orange. a) |Z| [Ohm], b) Θ[º], c) Re{Z}[Ohm] d) Im{Z}[Ohm]. Each at 1Hz. e) Nyquist (Re{Z}[Ohm]).

To further appraise the quality of the absolute impedance and its imaginary part at low frequency, the fitting of both samples was carried out, and Fig. 5 illustrates the results.

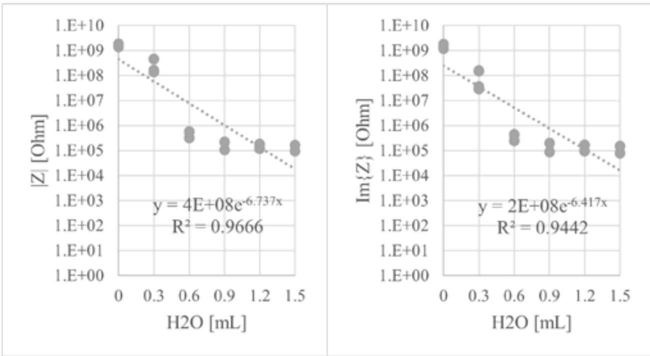

Fig. 5. |Z| and Im{Z} at, 1 Hz respectively, as function of volume of water introduced in the chamber.

5 Data Interpretation and Statistical Considerations

The absolute impedance and the imaginary part at 1 Hz showed the best results according to R^2. The current results also suggest that the phase was not related to the presence of water at 1 Hz. However, other frequencies could produce better results.

The results suggest that the presence of water increases conductivity. This is corroborated by the fact that the results with dry air and 0 ml of water are equivalent to those in the vacuum. However, the impedance plateaus at approximately 10^5 Ohms from 0.9 ml.

The results indicate a clear tendency. However, two factors still need to be controlled to achieve better results. First, the temperature inside the chamber was not monitored. Second, the samples were measured immediately after atmospheric pressure was reached inside the chamber, and as such, there was no time for the humidity to reach an equilibrium inside the chamber.

6 Conclusions and Future Work

This paper presented the preliminary experimental results for an RH sensor based on TiO_2 thin film to be used in the sensorial part of an e-nose system. In particular, the absolute impedance and its imaginary parts showed considerable sensitivity and reproducibility. Additionally, both absolute impedance and imaginary part had good fittings, with R^2 of 0.9666 and 0.9442, respectively. Although the obtained results show a good correlation between the presence of water and the conductivity of the device, the experimental conditions still need to be optimized so that more consistent results may be obtained. As such, future work will consist of repeating the experiments five times while monitoring the RH in the chamber in real-time until it reaches equilibrium. It is expected that under those conditions, enough data will be obtained to state definitively whether the TiO_2 thin film is appropriate to integrate into the sensor array.

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