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# Reproducibility Study of Metal-Oxide Gas-Sensors Using Two Different Temperature Setup †

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**Abstract:** The use of the electronic nose as a screening device is of great interest in various types of applications, including, for example, food quality control and environmental monitoring. It is an easy-to-use device and produces a much faster response than those obtained by classical chemical and microbiological techniques. The reproducibility of nominally identical electronic noses and sensors is critical. Four identical MOX-sensors were compared using two different working methods, temperature modulation and isothermal mode. Each sensor was tested with 2 standard compounds, water and lactic acid, often identified in food matrices, potential applications of the electronic nose.

**Keywords:** Sensors reproducibility; modulation of temperature; isothermal mode; electronic nose; MOX-sensors

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## 1. Introduction

The sensors reproducibility is an important issue to ensure the reliability of the final instrument, such as electronic noses, in which individual sensors are implemented [1]. The core of the electronic nose is made by a MOX- sensors array [2], which may work in isothermal and/or temperature modulation mode. These two different types of working can affect the sensitivity of the sensor respect to the gases.

For the temperature modulation, a periodic signal is applied to the heater to periodically change the sensor temperature in order to activate and inactivate the oxidation-reduction reactions between the sensitive material and gases. As a consequence the sensor resistance changes with time in a period manner and parameters describing this curve can be extrapolated and used as input to the pattern recognition algorithm. These parameters play the same role at those attributed to the responses of individual sensors that work at a constant temperature, i.e., a fixed voltage applied to the heater [3].

The electronic noses have been used for different type of applications, for example in environmental field [4], medicine [5], security and safety [6], and food control [7–8].

For the commercial applications (medical, food, environmental) three different aspects are important to develop an effective electronic nose: the system must guarantee good performance in order to sensitivity and specificity; the database and the pattern recognition software should work on different nominally identical electronic noses with minimal adaptation work; sensors should be interchangeable with nominally identical ones in case of failure. For these reasons, in this work the reproducibility of nominally identical sensors was tested comparing the features extrapolated with sensors working in the two modes: isothermal and temperature modulation.

## 2. Material and Mmethods

In this work, we used a JLM MOX STICK (JLMInnovation Gmbh) device (Figure 1) to perform experiments. That instrument include the sensors control software and the electronic part. The experiments were made using 4 commercial sensors (TGS 2620 - Figaro Sensor) exposed to vapours from pure solution of water and lactic acid.

For the constant temperature mode, each sensor was tested with a constant voltage of 3.5 applied to the heater. For temperature modulation, the hot period lasted 10 s (4 Volts) and cold period lasted 10 s (3 Volts). We chose these voltage values to have the same average temperature for both modes during a single thermal period.

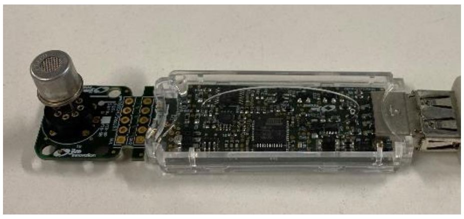


**Figure 1.** JLM MOX STICK device (JLMInnovation Gmbh) equipped with a commercial (Figaro TGS2620 ) sensor.

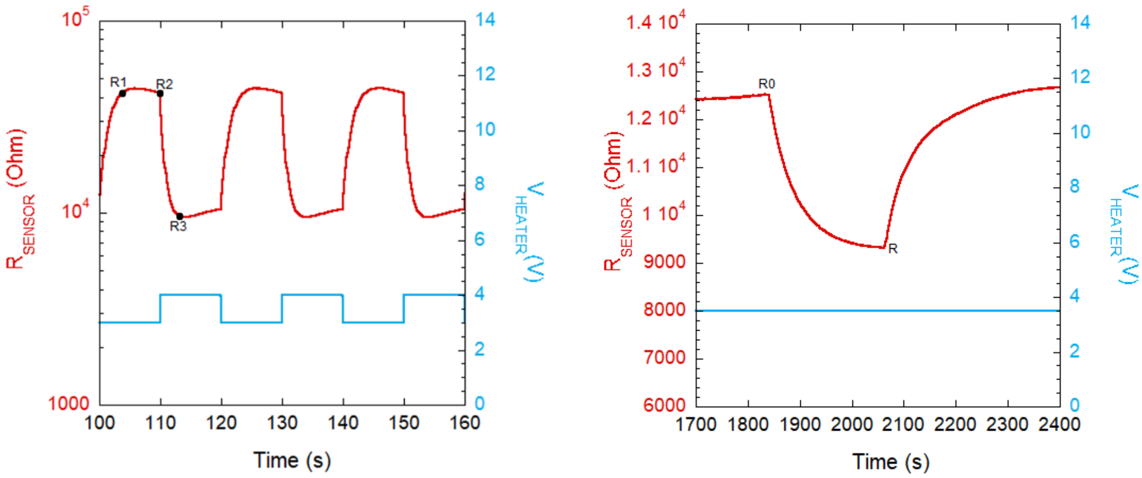
Each sensor was turned on and exposed to ambient air (environmental temperature 21 °C) for one hour in order to it stabilized before performing the planned series of measurements.

Two vials were prepared using 10 ml of water and 10 ml of lactic acid. Both were sealed with parafilm and it's left in the room for one hour to create the headspace. A hole was then created in the parafilm and the sensor was insert in. Measurement times were as follows: 10 minutes in contact with the compound vapors and 10 minutes recovery in air. Each substance was replicated 3 times.

The following features were used for the elaboration on temperature modulation dataset and to describe the resistance versus time curve. Using this method of measurement it is possible to extrapolate several features to be used to analyse the data, unlike the isothermal method that allows the processing of a single parameter R/R0. The value of R/R0 was calculated using the minimum resistance value reached by the gas in contact with the sensor (R), divided by the starting value of the sensor in air (R0). For the isothermal method was extracted 2 different significant feature:

- Ratio-CH = ratio among the sensor resistance identified at the end of the cold half-period and at the beginning of the next hot half-period, i.e., Ratio-CH = R2/R3.
- DeltaR-C = change of sensor resistance measured during the cold half- period, i.e., DeltaR-C = R1 − R2;

All these information are shown in the Figure 2.

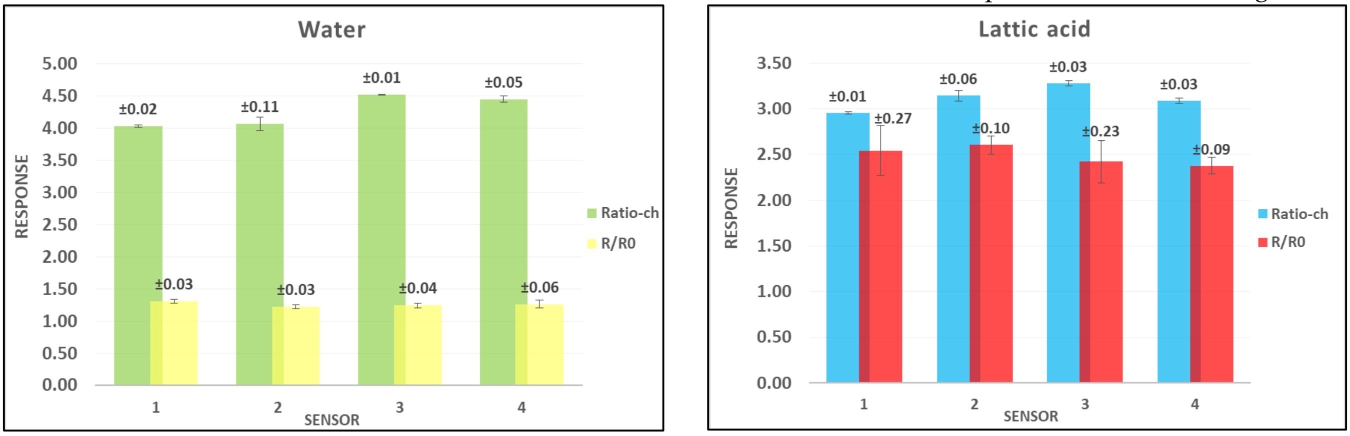



(**a**) (**b**)

**Figure 2.** (**a**) The Light blue line represent the modulation of the heater voltage between 3 and 4 volt. The red curve is an example of sensor resistance measured during exposure to water vapors. DeltaR-C = R1 − R2; Ratio-CH = R2/R3. (**b**) The isothermal example showing the voltage applied to the heater with light blue line, while in red curve there is an example sensor resistance measured during exposure to water vapors (response calculated as R/R0).

## 3. Results and Discussion

Based on our previous experience of food quality control applications [7] we chose the most significant features (Ratio-CH and R/R0) to compare the results obtained from each individual sensor. The statistics of the recorded responses are shown in Figure 3.




**(a)** **(b)**

**Figure 3.** (**a**) Comparison between Ratio-CH (green color) and R/R0 (yellow color) response to water vapors. (**b**) Response obtained to lactic acid vapors. Light blue rectangles identified Ratio-CH, while red rectangles concern R/R0.

The value of each single response is the average of the single values obtained during the measurement session (replicate for 3 time). The graphs also show the standard deviation corresponding to each result.

The first thing to note is that for both compounds analyzed and for both methods used, the sensors give a reproducible response. Evaluating in detail the response obtained of each methods we can see that the standard deviation (absolute value) for temperature modulation is always lower than that obtained using the isothermal method. This is further enhanced normalizing the standard deviation to the average of the response intensity. Indeed, concerning water, the value change from 0.5% to 2.6% for Ratio- CH, wile to R/R0 from 2.25% to 4.65%. Is possible to note the same situation in the measure of lactic acid (Ratio-CH 0.5%–3.9%, R/R0 3.80%–10.74%).

During the experience gained using the temperature modulation method, we realized that one of the features that bring a lot of information in the construction of the PCA (Principal Component Analysis) plot is the DeltaR-C and therefore we decided to analyze it [5–7].

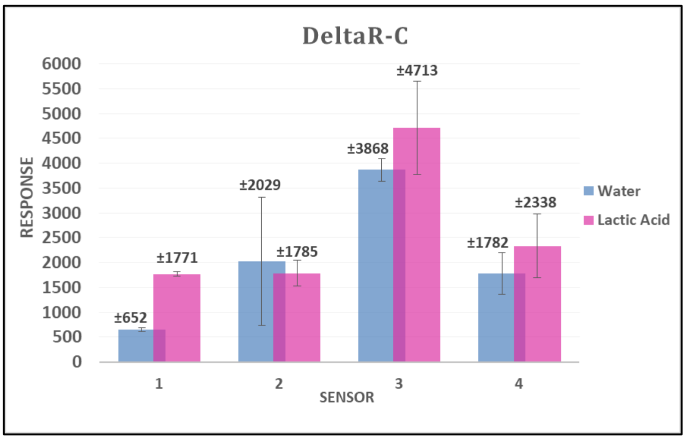



**Figure 4.** Sensors responses measured during exposure to water and lactic acid vapors.

As can be seen in Figure 4, the reproducibility of the sensors towards the analyzed compounds is relatively poor respect to what observed concerning the Ratio-CH feature. There is an appreciable variance from sensor to sensor and within individual measurements. We find a standard deviation that ranges from 5.3% to 63% (water) and from 2.5% to 27.6% (Lactic acid). But in any cases it is able to give a most important information combined with other extracted features [5–7]. This means that the reproducibility of sensors depends on the given feature analyzed.

## 4. Conclusion

In conclusion, the experiments carried out with temperature modulation show that some extracted features are more stable than others, even if from the PCA it is possible to deduce that also the latter ones provide important information.

For the constant temperature the normalized feature may be more or less repeatable than those extrapolated from the other mode depending on individual features, though the response extrapolated from this working mode benefit from the normalization to the reference air while this not occurs for the temperature modulation mode.

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