

Preliminary Study on the Poisoning Effect of Different Materials on MOX Sensors

Beatrice Julia Lotesoriere¹, Stefano Robbiani², Ana Maria Tischer¹, Lucia Corrà¹, Emanuele Zanni², Anna Gianfranceschi², Lucia Giuffrida², Laura Capelli¹, Raffaele Dellacà²

¹ Politecnico di Milano, Department of Chemistry, Materials and Chemical Engineering "Giulio Natta", Piazza Leonardo da Vinci 32, Milan, Italy

² Politecnico di Milano, Department of Electronics, Information, and Bioengineering, Via Giuseppe Colombo 40, 20133 Milan, Italy

Corresponding author: lucia.corra@polimi.it

Summary:

Additive fabrication techniques have opened new possibilities for the realization of sensors chambers of different shapes, to optimize performances. The objective of this work is to carry out a first experimental study to evaluate the poisoning effect of some new materials on commonly use MOX sensors. Effects of PEEK, 3D Print Resin and Silicone on TGS2610 and TGS2611 is investigated. Gas sensors response to different calibrants is recorded before and after sensors' exposition to the materials, resulting in reduction of the response for Silicone and Resin, while PEEK do not affect sensors' response.

Keywords: chemical sensors, contamination, 3D Print, Resin, Silicone, Siloxanes, PEEK

Background, Motivation and Objective

Several studies show how the geometry and the fluid dynamics of gas sensor chamber affect the sensor responses, especially in terms of response time, amplitude, and stability [1]. Nowadays, additive fabrication techniques like 3D printing have become more precise and affordable, and have opened up new possibilities for the realization of sensor chambers of different shapes, with the purpose of obtaining optimized performances according to the application. However, whenever introducing a new material, besides considering safety issues, ease of processability and cost, it is important to verify its compatibility with odour analysis. Some evaluations regarding the inert behaviour and recovery of VOCs for different sampling materials have been included in the recent revision of the European Standard about dynamic olfactometry (EN 13725:2022) [2]. In the specific case of e-nose applications, it is of fundamental importance to verify that the VOCs that are released by all the materials [3] used for the manufacturing of the sampling line and analysis system do not have a poisoning effect on the gas sensors.

In this context, the objective of this work is to carry out a first experimental study to evaluate the poisoning effect of some materials that are commonly used in biomedical devices, and that could potentially be used to design fit-for-purpose sampling systems and sensors' chambers for e-nose applications.

Description of the New Method or System

A dedicated setup was developed to specifically investigate the poisoning of some commercial MOX sensors (i.e., 5 TGS2610 and 5 TGS2611, Figaro Inc., Osaka, Japan) towards some materials that could be used for the realization of e-nose systems for biomedical applications, including polyetheretherketone (PEEK), Resin (Biomed Clear, Formlabs, Massachusetts, USA), "Boiled" Resin (pre-treated by boiling in distilled water for 2 h at 100 °C), and Silicon. Even though the poisoning effect of siloxanes on MOX sensors has already been reported in literature [4], it was decided to include it in the study as a reference, because it is often used for the realization of biomedical components such as face masks [3]. The experimental part consisted of three different phases. First, gas sensors were tested before poisoning to evaluate the sensors' responses towards different calibrants (i.e., butanol 20 ppm, isobutylene 20 ppm, 4-heptanone 20 ppm, methane 750 ppm) without any potential interferent. To do this, all the gas sensors were installed in a glass sensors' chamber equipped with a temperature and humidity sensor (SHT40, Sensirion, Switzerland) to monitor stability of environmental conditions. The gaseous calibrants were stored in 18 L Nalophan™ bags and conditioned for 3 h before analysis in room conditions at 22 °C and 40%RH. The tests were carried out alternating air and gaseous samples to stabilize

and restore the sensors' baseline before and after each analysis controlled by 2 shut-off valves: 15 min of air sample analysis, 15 min of calibrant sample analysis, and 15 min of air sample analysis. The samples were injected at a constant flowrate of 1 l/min into the chamber by a lung-pump system of two flux drums (Fig. 1a). After that, gas sensors were placed in pairs (i.e., one TGS2610 and one TGS2611) in 5 flasks of 50 mL StonyLab®: 4 flasks with a piece of one of the 4 poisoning materials considered with an equivalent surface area of 11 cm² and a flask with no poisoning material to be used as a blank. To accelerate the VOCs release, the flasks containing a pair of sensors each were placed in a ventilated oven (AEG BSK798280B) at 80 °C for 15 days (Fig.1b). Finally, the effect of the contaminants on sensor responses was evaluated by repeating the same analyses carried out before poisoning at the same controlled conditions (i.e., 40% RH, 22°C).

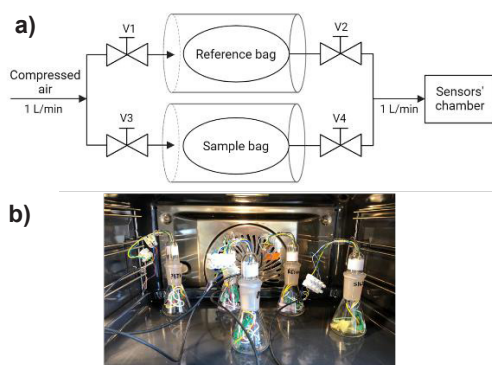


Fig. 1. a) Sample delivery system to the sensors' chamber; b) Sensors poisoning in the oven

Results

Sensors' outputs were compared with respect to the sensors' responses obtained before poisoning and with respect to the readings of the blank analysis in terms of baseline shift and signal amplitude. Fig. 2a and Fig. 2b show a comparison of the sensors' responses (in terms of raw resistance values and normalized resistances ($R-R_0$), respectively) after 2 weeks of permanence in the oven. The results obtained, besides proving that after 2 weeks the response of the sensors in contact with the silicon are substantially zeroed, also show that the only materials, among the ones tested, resulting in almost no alteration compared to the blank sample, is PEEK. Boiling the resin results in an improvement compared to the resin without pre-treatment, but it still doesn't seem suitable for long-term applications. Further studies should investigate other materials and other possible pre-treatments.

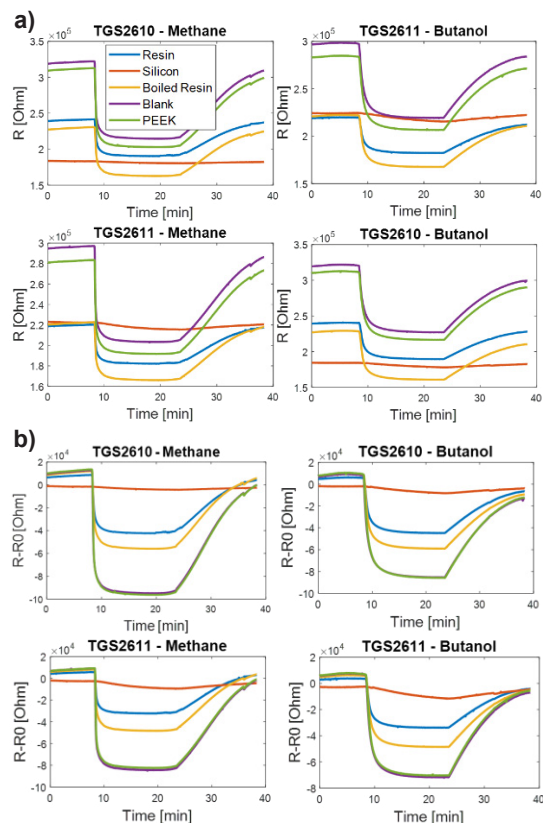


Fig. 2. Sensor responses: a) resistance values; b) normalized resistances

Funding

This study was co-funded by the National Plan for NRRP Complementary Investments (PNC) - project n. PNC0000003 - AdvANced TechnoloGies for Human-centrEd Medicine (ANTHEM). This work reflects only the authors' views and opinions, neither the Ministry for University and Research nor the European Commission can be considered responsible for them.

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