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New Tool to Unravel Interactions Between Gas and Sensitive Surface Through the Simultaneous Characterization of Gas Uptake and Electrical Properties of the Material

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Summary:

This work presents an innovative setup for simultaneous determination of (i) electrical gas sensor response and (ii) uptake of target gas on sensitive material. Operating under atmospheric pressure and at 298K, it is designed to work under a wide range of gas flow, relative humidity and target gas concentration. Results validate the system effectiveness in monitoring gas uptake and sensor responses simultaneously, offering insights into physicochemical processes related to gas detection.

Keywords: Gas sensors, Conductive polymers, Ammonia detection, Polyaniline, Uptake quantification

Introduction and Objectives

The last decade has seen a growing interest for air quality, leading to different approaches developed to monitor air pollution such as sensors. Among them, resistive sensors based on metal oxides or conductive polymers are promising for gas detection because of: (i) low limit of detection, (ii) high tunability and (iii) low manufacturing cost [1]. While these gas sensors can achieve high sensitivity (ppt range), selectivity and high processability with low energy consumption [2], drivers of their performances are still not fully understood [3]. Addressing accurately gas-sensor interactions could enhance the understanding of the physicochemical parameters responsible for the limitations of the sensors and facilitate their optimization. Indeed, this work proposes the development of a new experimental setup for monitoring simultaneously sensors electrical response and gas uptake on the sensitive material: the Cell for Atmospheric Pressure Uptake and SensINg Experiment (CAPUSINE). For the first time, quantification of the amount of gas (ammonia, NH_3) taken up by a sensitive surface based on conductive polymer (doped polyaniline, dPAni), is monitored under atmospheric pressure conditions, at 298 K, simultaneously with the sensor response. The developed setup is easily applicable to others sensing materials and gaseous species thanks to the ability of the analyser to monitor different types of gas, making CAPUSINE a powerful tool to unravel interactions between gases and sensors.

Material and Method

CAPUSINE, consists in four main parts: (i) gas generation system, (ii) sample cell, (iii) online reference analyser and (iv) gas sensors under study.

Gas generation system: Three drivers of the gas intake are controlled: (i) relative humidity (%) known to significantly affect sensor response, (ii) target gas concentration (ppm) and the (iii) gas flow ($mL.min^{-1}$) which impacts the residence time of the gas in the sample cell.

Sample cell for uptake: The sample cell consists of a ¼ inch steel tube, featuring sieve with a mesh size of 250 µm to prevent drift of powder samples. Interestingly, specific surface area (SSA, $m^2.g^{-1}$) of the sensor material serves as a normalization factor for comparing results across different experiments. To limit gas interactions with walls, treated steel (Sulfinert®, Silcotek) is used. The geometry of the cell ensures thorough exposure of the material to the gas flow.

Analyser: SIFT-MS is used to monitor concentration profiles of the target gas. SIFT-MS has low intake flow (30 $mL.min^{-1}$), a high temporal resolution (1 measurement every 3 seconds) and an ability to monitor a wide variety of gaseous species without water interference.

Gas sensors: To corelate the uptake of target gas on sensor electrical response, CAPUSINE is also equipped with two gas sensors based on the same material (Fig. 1): one upstream, located in the sample chamber, and a second downstream, located just before the analyser (Fig. 1). The two sensors could allow for the corelation of uptake and sensor response and their measurements are also compared to the analyser.

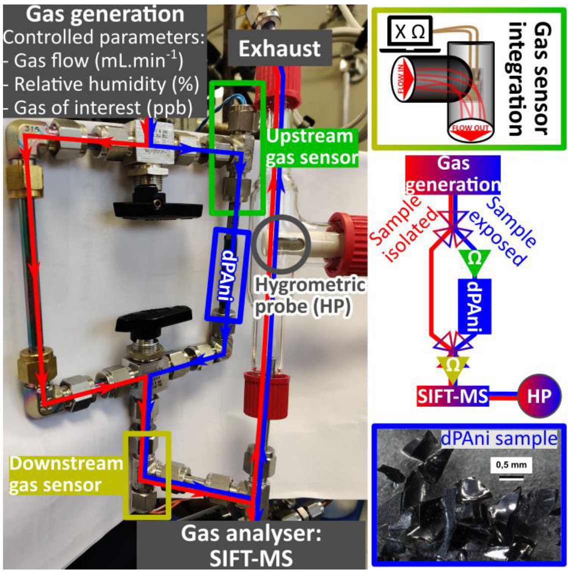

Fig. 1. CAPUSINE general description

Results and Discussions

A typical experiment conducted in CAPUSINE follows 4 steps (Fig. 2). (i) A fresh dPAni sample (50 mg corresponding to 1.5 × 10^{-3} m^2) is placed in the sample cell and equilibrated with controlled relative humidity. (ii) The gas flow is redirected to the bypass, isolating the sample, and the gas of interest (NH_3) is set to targeted concentration (2 ppm). (iii) Upon stabilization of pollutant concentration, the flow is switched to expose dPAni, initiating NH_3 uptake. (iv) After reaching equilibrium ([NH_3] is equal to the beginning of step (iii)), the flow returns to the bypass for NH_3 concentration control. The amount of NH_3 taken up can be retrieved from integration of the area under NH_3 profile from SIFT-MS (light blue area in Fig. 2).

To validate that the observed uptake is solely attributed to dPAni, two experiments were conducted. First, without any sensor neither dPAni, no change in NH_3 signal occurred upon switching from bypass to the empty sample cell. Second, with sensors but no dPAni, sensors took up, up to 8 × 10^{14} NH_3 molecules in the first 10 minutes, i.e. 1 % of NH_3 molecules in the flow over this period of time and less than 3 % of the NH_3 taken up by dPAni (Fig. 2). Hence, sensors used interacts negligibly with NH_3, confirming that the observed uptake in Fig. 2 is mainly due to NH_3 uptake on dPAni.

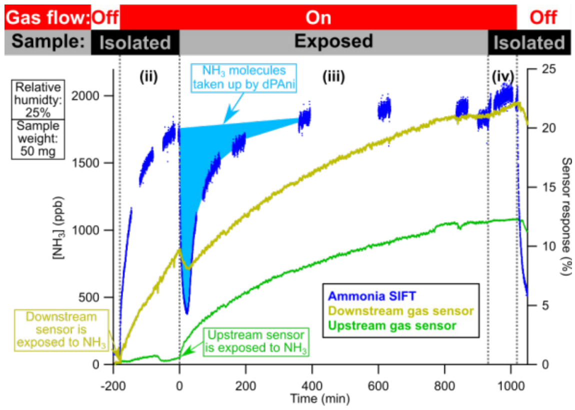

Fig. 2. CAPUSINE experiment with dPAni and NH_3, at 25% RH.

As, dPAni is exposed to 2 ppm of NH_3, it uptakes 2.7 × 10^{16} molecules.cm^{-2} (Fig. 2). Notably, the downstream sensor response mirrors the uptake monitored by SIFT-MS. The same experiment can be repeated at different levels of humidity to observe the impact of water on uptake and sensor response. The impact of interfering gas species can also be explored by replacing or mixing NH_3 with another gas.

To conclude, CAPUSINE experimental setup enables simultaneous monitoring of gas uptake and sensor response within a realistic environment, with control over the relative humidity and gas phase composition. This innovative setup is expected to be able to provides deep insights into the physicochemical processes contributing to the response of resistive gas sensor. Thanks to its versatility, CAPUSINE is anticipated to be applicable to a wide range of solid gas sensors and capable of accommodating various gaseous mixtures.

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