

Chemical Gas Sensor Based on a Flexible Capacitive Microwave Transducer Associated with a Sensitive Carbon Composite Polymer Film [†]

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[†] Presented at the Eurosensors 2017 Conference, Paris, France, 3–6 September 2017.

Published: 18 August 2017

Abstract: This communication presents results on the feasibility of an inkjet printed chemical gas sensor suitable for a real time multi-sensing platform. The prototype targets volatile organic compounds (VOCs) such as ethanol vapor to monitor environmental pollution. The designed and ink-jet printed microwave sensor is presented. Preliminary results have shown the influence of ethanol vapor on the electrical properties of the sensor at microwave frequency range. The sensor's sensitivity to ethanol vapor has been estimated to −2.48 kHz/ppm. The final aim of this work is to develop a low cost sensor for Internet of Things (IoT) applications.

Keywords: chemical gas sensor; electromagnetic transduction; inkjet printing; flexible substrate; carbon materials; microwave device; resonator

1. Introduction

Atmospheric environmental pollution caused 7 million deaths in the year 2012 as stated by the World Health Organization (WHO) [1] (pp. 3–4). This is related, among other things, to the complexity and diversification of pollutants, known as a source of irritation and decreased lung capacity. Some of these pollutants are even considered carcinogenic, among them VOCs, which is why they were chosen as target compounds. In this context, we propose a low cost chemical sensor for real-time monitoring and quantification, combining an electromagnetic transduction resonator with a sensitive layer, in the frequency range up to 6 GHz. In addition, the device will be integrated in an IoT and embedded system solution in the future, offering interesting prospects for the proliferation of detection and control communicating wireless networks sites. The device is fabricated on a flexible paper substrate by inkjet printing technology [2] (pp. 876–886), [3–5] (pp. 425–435). We present the theoretical study, the results of experimental characterizations, before concluding and opening on perspectives of the sensor based on two bandpass resonators.

2. Theoretical Study

The studied platform is based on two capacitive resonators in order to provide differential detection. One resonator is functionalized with a sensitive material and constitutes the sensitive channel, while the other is considered as a reference channel and compensates the variations not related to the sensitive material. Each channel consists of two parallel networks of 50 electrodes each, spaced with a gap (L_2) of 300 μm . The geometry and configuration of this prototype is shown in Figure 1

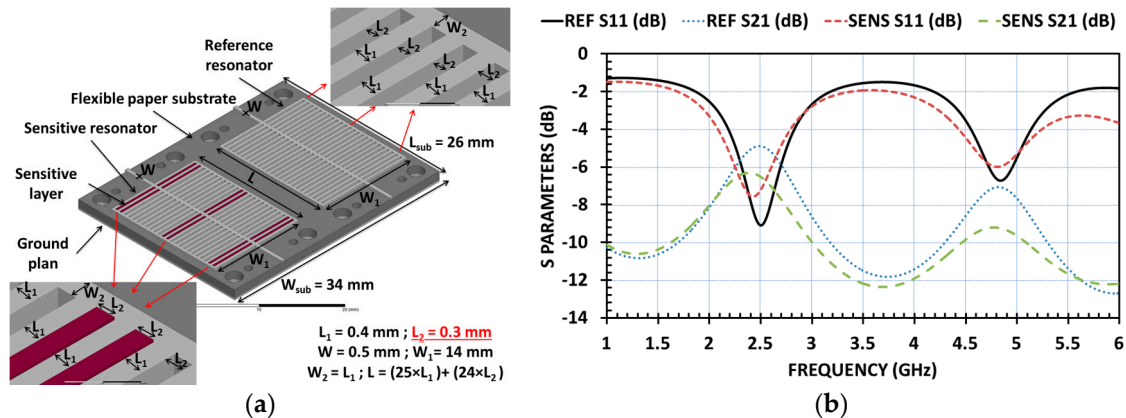


Figure 1. (a) Overview of the device geometry and (b) Simulated S parameters.

The working principle is based on the perturbation of the electromagnetic field due to the presence of vapor. The target species are absorbed on the sensitive layer and modify the electrical properties of the materials thus the electrical behavior of the device [2,3] (pp. 876–886). Using ANSYS-HFSS software which delivers 3-D full-wave simulation for RF components, we perform the simulation with paper as substrate (permittivity 3.05 and dielectric loss 0.089 at 4.3 GHz), silver nanoparticles as metallized lines, conductive polymer poly (3,4-ethylenedioxythiophene) polystyrene sulfonate with multi walled carbon nanotubes (PEDOT: PSS-MWCNTs) as sensitive layer. The material were considered as homogeneous in a first approximation, their parameters were measured and presented previously, in [3,6,7]. Figure 1b shows the S parameters of the simulated device. Each resonator has two resonant modes in the frequency range up to 6 GHz, around 2.49 and 4.83 GHz, respectively. The simulation of the addition of 5 layers of the sensitive material induces a frequency shift close to -100 MHz for each mode.

The distribution of the electric field E for both resonant modes of the reference channel is shown in Figure 2. To ensure good sensitivity, the 5-layers sensitive film was placed on the three zones where the E field is more intense, enhancing a capacitive disturbance effect for the first and second modes.

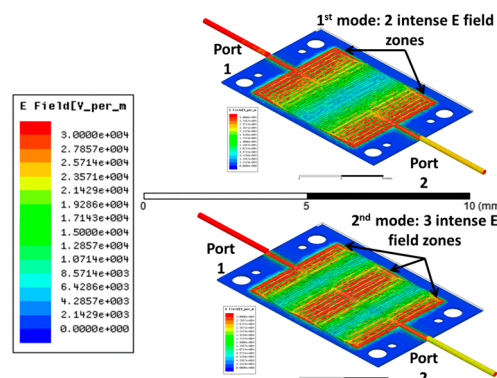


Figure 2. Distribution of the electric field E for both resonant modes.

3. Experimental Study and Discussion

The sensor was fabricated on flexible paper substrate using inkjet printing technology (Dimatix inkjet printer 2800 series). We used an Epson photo paper with a thickness equal to 260 μm for the substrate, a silver nanoparticles solution ink (JSB 25 HV) for the metallized geometries and a composite polymer solution doped with 1 to 1.2% of multi-wall carbon nanotubes ink (Poly-Ink HC) for the sensitive film. Figure 3a shows the manufactured device with 5 layers of the sensitive material as indicated in part 2, and Figure 3b shows the experimental S parameters measured over the frequency range from 1 to 6 GHz with the vector network analyzer (VNA) MS2026B, calibrated with 4001 points. We will focus on the first mode only, within the reduced frequency range (2–4 GHz). The behavior of this first mode for both resonators under ethanol vapor is presented in the next part. Further studies are planned in order to remove the ripples disturbing the second mode.

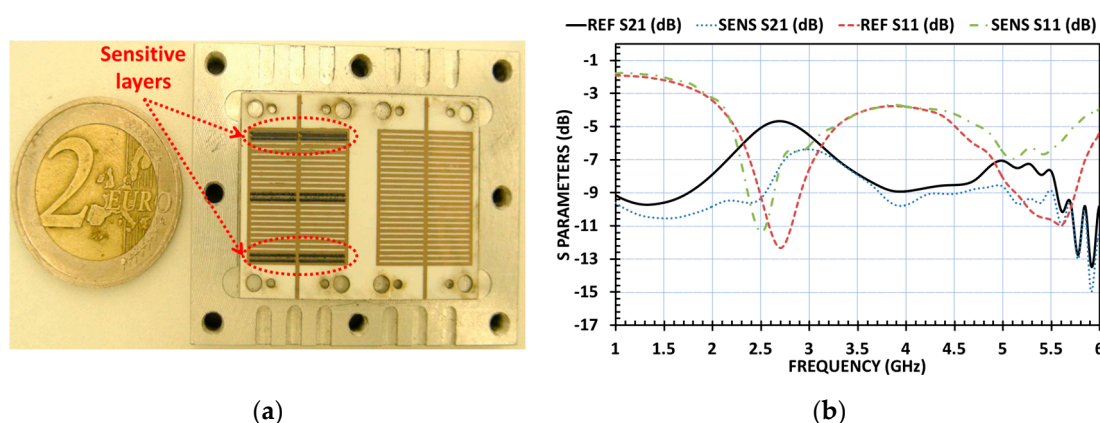


Figure 3. (a) Experimental device on paper mounted on the test cell basement; (b) Experimental S parameters.

The experimental detection configuration is mainly based on a vapor generator (CALIBRAGE PUL 110), which is used to generate and control the concentrations of the target vapor from a liquid contained in a heated bottle. The vapor particles are transported by nitrogen as a carrier gas at a constant flow rate of 0.112 L/min [3,8]. A sequence of target vapor concentrations (C) was delivered on the two resonators of a device placed in the low cost test cell. Ethanol was used as model target vapor, using the sequence: 0, 500, 0, 500, 0, 1000, 0, 1000, 0, 2000 ppm for 10 min each concentration step, carried out after an initial rinsing step under nitrogen for 450 min to ensure stability at room temperature. The 0 ppm concentration corresponds to nitrogen exposure only. The relative permittivity of the ethanol is about 24.5 at 25 $^{\circ}\text{C}$, compared to the permittivity of nitrogen which is close to 1, that may cause a change in the environment of the sensor and will thus modify the electrical properties of the sensitive layer, resulting in an additional variation of the sensing channel compared to the reference one [9]. All detections are carried out under the same conditions, at a fixed temperature equal to 26 $^{\circ}\text{C}$ and a humidity of 32% RH.

At this point we recall that we focus on the behavior of the first resonance mode of the device. The effects of the interaction of the ethanol vapor with the electromagnetic fields of the resonators are thus observed in the reduced frequency range between 2 and 4 GHz that is recorded with the same VNA calibrated with 4001 points. Figure 4a illustrates the effect of the ethanol vapor on the resonant frequencies. These measurements correspond to the real time dynamic detection of different ethanol vapor concentrations (from 0 to 2000 ppm). While the frequency related to the minimum insertion losses of the reference resonator (FrS21r) increases slightly with the ethanol concentration, the frequency of the sensitive resonator (FrS21s) decreases significantly at the same time. The steady state frequencies at the end of each 10 minutes step as function of the concentration are reported on Figure 4b. On both figures are superimposed the resulting differential signal, by subtracting the response of the reference from that of the sensitive line: $\Delta F_{S21} = \text{FrS21s} - \text{FrS21r}$. This leads to the estimation of a high level sensitivity (−2.48 kHz/ppm) which is promising for future applications of the sensor.

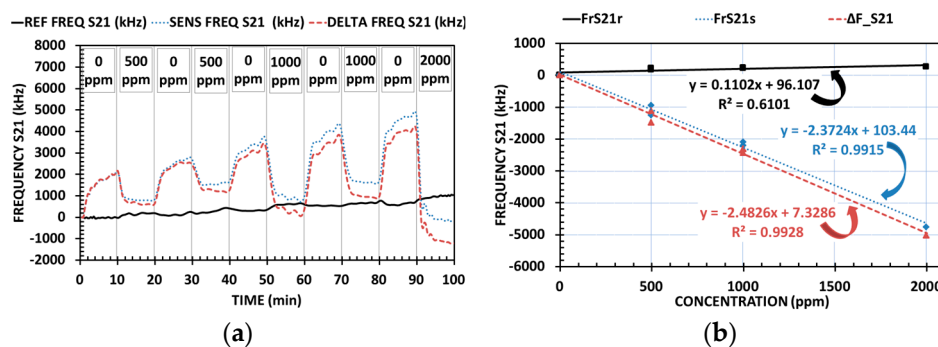


Figure 4. (a) Real-time frequency variation with different ethanol vapor concentrations; (b) Resulting experimental steady state frequency shift (monitored at 10 min) and sensitivity.

4. Conclusions

The feasibility of a fully inkjet printed chemical gas sensor based on a flexible capacitive microwave transducer associated with a sensitive carbon composite polymer film has been demonstrated by the theoretical study based on finite element tool for electromagnetic simulations and by relevant experimental study. The sensitivity might be further improved by increasing the thickness of the sensitive material, or by its further functionalization. Future studies will aim to improve the understanding of the physical phenomena that are involved. However, the high effective surface area of the proposed device could be compatible with other sensitive materials, within a large range of electrical conductivity. It is thus very interesting for the development of various chemical gas sensors based on an integrated microwave device.

Acknowledgments: We would like to thank the National Research Agency for its support (CAMUS project, ANR-13-BS03-0010) and the French Embassy of Singapore (Merlion project), as well as the RENATECH national network for its technological support.

Conflicts of Interest: The authors declare no conflict of interest.

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