



## Lossy mode resonance optical fiber sensor to detect organic vapors

C. Elosúa<sup>a,\*</sup>, I. Vidondo<sup>a</sup>, F.J. Arregui<sup>a</sup>, C. Barriain<sup>a</sup>, A. Luquin<sup>b</sup>, Mariano Laguna<sup>b</sup>, I.R. Matías<sup>a</sup>

<sup>a</sup> Departamento de Ingeniería Eléctrica y Electrónica, Universidad Pública de Navarra, Campus de Arrosadía s/n, E-31006 Pamplona, Spain

<sup>b</sup> Instituto de Síntesis Química y Catálisis Homogénea-CSIC, Universidad de Zaragoza, E-50009 Zaragoza, Spain

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### ABSTRACT

A transmission sensor able to detect Volatile Organic Compounds (VOCs) has been developed using optical fiber with Plastic Cladding (PCS). Specifically, 1.5 cm of the cladding was removed in order to deposit an organometallic compound whose chemical structure is  $[\text{Au}_2\text{Ag}_2(\text{C}_6\text{F}_5)_4(\text{C}_6\text{H}_5\text{C}\equiv\text{CC}_6\text{H}_5)_2]_n$  along this section. This complex reacts reversely in presence of organic vapors such as alcohols, therefore, it is used as sensing material. The compound was altered to show a negative charge, so it can be deposited combined with a positive charged polymer by means of the Layer-by-Layer (LbL) method. In this manner, as the nanolayers were deposited, Lossy Mode Resonances (LMRs) were induced and shifted. The polymer nanolayers doped with the organometallic material accelerated the LMRs appearance with respect to the nanolayers without additive and so, it enhanced the spectral shift. Once the construction process was completed, two LMRs were observed, choosing the second one to study the sensor behavior when it was placed at 663.57 nm. The sensor was exposed to different concentrations of ethanol, methanol and isopropanol vapors, showing sensitivities of 0.417, 0.520 and 263 nm ppm<sup>-1</sup>, respectively. In the case of methanol, the second LMR peak shows a remarkable blue shift of 100 nm. The interference with water vapors is minor to 1 nm below 60%, whereas the effect of temperature is insignificant between 20 °C and 60 °C.

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### 1. Introduction

Applications related with the detection of VOCs can take advantage of the interesting features that optical fiber sensors offer. Electromagnetic immunity, remote sensing and the absence of electrical signal, just to mention the most relevant, make this kind of sensors an option to be taken into account [1]. Many sensors have been developed based on intensity modulation, but this transduction can be affected by undesired power fluctuations. In this context, devices based on spectral shifts of electromagnetic resonances such as LMR are a promising alternative [2].

There are several applications related with VOCs: controlling the maturing process of food and beverages [3], or monitoring their concentration due to their toxicity [4] are some of them. Different working environments and even dwellings can be affected by VOCs: it is known as Sick Building Syndrome [5]. Although electronic sensors are currently available to detect some VOCs, their electrical nature [6] or the need of biasing makes their use questionable in certain places with an explosion risk or with

high electromagnetic noise. The features mentioned above make this background a potential market niche for optical fiber sensors [7].

Organometallic materials have been used in previous works to prepare VOC sensors [8–12]. These compounds show reversible changes in their optical properties (refraction index, optical absorption, etc.) in presence of VOCs. Different types of fiber and experimental configurations have been studied, but all the devices were based on intensity modulation [7]. As mentioned before, this transduction, although simple, depends on optical source power fluctuations or artifacts induced by undesired fiber bending [13–15]. Therefore, optical fiber sensors based on spectral shifts can overcome this kind of inconveniences. It has been demonstrated that thin coatings deposited along an optical waveguide can induce distinct resonances [16]: surface plasmon resonances (SPRs) [17], lossy mode resonances (LMRs) and long-range surface exciton polariton [18]. The optical properties of the thin film, such as its refractive index and its thickness, define the type of resonance induced and the wavelength where it takes place. LMR is growing in interest because both TM and TE polarizations can be used, which is not the case of SPR. Resonances can be induced and tuned depending the thickness of the coating [19]. On the other hand, SPRs have to be produced with metals, but LMRs can be induced with a wider variety of materials. In any case, the material has to meet

\* Corresponding author. Tel.: +34 948169328; fax: +34 948169720.  
E-mail address: [cesar.elosua@unavarra.es](mailto:cesar.elosua@unavarra.es) (C. Elosúa).

some specifications to induce LMR: its real part of the permittivity has to be positive and higher in magnitude than its own imaginary part as well as the real part of the permittivity of the waveguide material [19,20]. Optical fiber sensors based on LMR have been reported using metal oxide coatings [20,21] and polymeric films [2].

The presence of metallic atoms in the sensing material makes it possible to induce LMRs if it is deposited along an optical fiber. Furthermore, the change on its optical properties in presence of VOCs can also vary the spectral location of the LMR. In this manner, sensors based on this kind of resonances can be developed using this material.

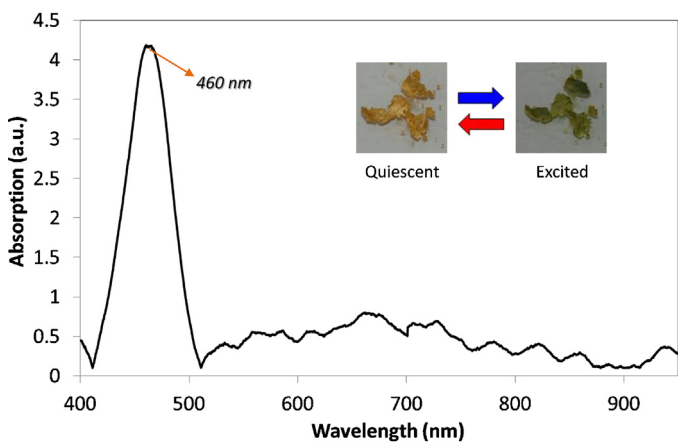
We present an optical fiber sensor to detect VOCs. The sensing material  $[\text{Au}_2\text{Ag}_2(\text{C}_6\text{F}_5)_4(\text{C}_6\text{H}_5\text{C}\equiv\text{CC}_6\text{H}_5)_2]_n$  is deposited following the LbL method. The construction process has been optimized, so only one polymer is used to develop the sensors. This optimization simplifies the device fabrication, and therefore, the time that it takes to develop it. The sensor has been exposed to different alcohols individually, calibrating its response for increasing and decreasing concentrations in order to check the hysteresis. The effect of both temperature and relative humidity has also been studied.

## 2. Materials and methods

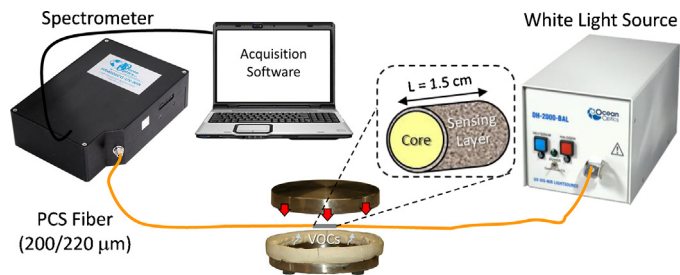
### 2.1. Sensing material and chemical reagents

The organometallic compound obeys to the formula  $[\text{Au}_2\text{Ag}_2(\text{C}_6\text{F}_5)_4(\text{C}_6\text{H}_5\text{C}\equiv\text{CC}_6\text{H}_5)_2]_n$ , with a polymeric structure by Au–Au interactions [22]. The diphenylacetylene molecules act as ligands, enhancing the stability of the molecule and also defining its optical properties such as the luminescence and the optical absorbance [10]. When VOCs molecules are present, they broke firstly the Au–Au interactions, and secondly, they get coordinated to the Ag atoms, breaking the Au–Ag bonds [23]. This reaction modifies the molecule morphology and so, its optical properties. The optical absorbance of the sensing material is plotted in Fig. 1, from 400 nm to 900 nm: the absorption peak located at 460 nm is remarkable. More details about this complex are described in [24].

The polymer used to fabricate the sensing layer was poly (allylamine hydrochloride) (PAH) (Mw ~56,000); it was purchased from Sigma–Aldrich Inc. and used without further purification. Ultra-pure deionized water (18.2 MΩ) was employed to prepare aqueous



**Fig. 1.** Absorption spectrum of the organometallic material; inset, a detail of the compound color change in presence of ethanol vapors. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of the article.)



**Fig. 2.** Experimental set up used to study both the sensor construction process and its response to distinct alcohols vapors. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of the article.)

solutions of the polymer (10 mM), adjusting them at pH 12 by a NaOH mixture. Potassium hydroxide from Sigma–Aldrich Inc. was used to prepare 1 M aqueous solution. Sodium dodecyl sulfate (SDS) from the same supplier was employed to make a 10 mM aqueous solution. All the VOCs used were obtained from Sigma–Aldrich Inc.

### 2.2. Experimental set up

A transmission configuration was used to study the construction process and also the behavior of the sensor in presence of the different VOC (Fig. 2). The sensor was connected between a white light source (DHS2000, Ocean Optics Inc.), and a CCD spectrometer (HR4000CG-UV-NIR High-resolution Miniature Fiber Optic Spectrometer, Oceanoptics Inc.). The spectral range of this spectrometer goes from 200 to 1025, but the range studied was limited between 450 and 900 nm as in this region the spectrometer sensitivity is optimal. Plastic clad silica fiber (PCS) obtained from Thorlabs Inc was used to prepare the sensor; its core/cladding diameters are 200/220 μm, respectively.

The fiber was kept straight to avoid any bending artifact along both the construction and the characterization processes. The sensor was placed into a steel chamber where the different vapors were injected in liquid state; then, the chamber was hermetically closed, minimizing any vapor leak and limiting interfering light. The chamber volume is 20 cm<sup>3</sup>, so it is assumed that the vapors get homogeneously distributed inside it. The liquid volume to inject in order to get a certain concentration was calculated by the ideal gases law. Once the measurement was recorded, the chamber was opened and the vapors evacuated with an air flow. All the experiments were performed 25 °C by a thermal jacket [11].

### 2.3. Sensor construction process

The first step to prepare the sensor consisted of removing the plastic cladding of the fiber by means of a flame burner. The length of this segment was 1.5 cm to minimize the optical losses [25]. Any cladding or organic remains were removed by cleaning the fiber gently with ethanol. In order to induce a superficial electrostatic charge, the cladding-removed segment was immersed in the KOH solution for 10 min [2,26].

The sensing material deposition is achieved by the LbL method. This procedure is based on the electrostatic attraction of oppositely electrically charged polymers (polycation and polyanion), so that they get assembled in a nanometric scale. The hydrophobic nature of this organometallic compound makes impossible to prepare an aqueous solution to be used in any LbL sequence [27]. In previous works, it was first diluted with ethanol and then mixed with water, forming a suspension [10,11]. Looking for slowing the precipitation process, the ethanol solution is added to a

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