

Volatile-organic-compound optic fiber sensor using a gold-silver vapo-chromic complex

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Abstract. We describe a new gold-silver complex based on 2,2'-bipyridine, whose formula is $\{Au_2Ag_2(C_6F_5)_4[(C_5H_4N)-(C_5H_4N)]_2\}_n$, used to detect volatile organic compounds (VOCs) such as ethanol, methanol, and acetic acid. This organometallic material is presented in the form of bright yellow powder, and suffers a change in its optical properties when it is exposed to VOCs. A new fiber optic sensor is presented based on the properties of a new vapo-chromic material. The sensor works in a reflection configuration and consists of an optic fiber pigtail (core diameter is 200 μm and cladding is 230 μm) on the cleaved end of which the vapo-chromic material, previously mixed with a commercially available solvent, Liquicoat[®], is deposited by using the dip-coating technique. Using an optical source and a photodetector, it is possible to detect and quantify the change in reflected intensity-modulated signal when the sensor is exposed to VOC inside a sealed chamber. This behavior can be related to the VOC concentration. The study of the sensor's response is made at a specific wavelength for different VOC concentrations. Limits of detection of 2.16, 1.73, and 3.73 mg/L of vapors of ethanol, methanol, and acetic acid vapors, respectively, are attained.

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Subject terms: optic fiber sensor; gold-silver vapo-chromic material; dip-coating technique.

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1 Introduction

In the last few years, investigation of sensors for volatile organic compound (VOC) detection has increased greatly, motivated by the number of applications where they can be used, such as food or chemical industry, electronic noses, or safety concerning toxic ambient conditions. So far, the devices developed are polymer-based electronics. Although

this technology has been improved in the last few decades, it presents some important limitations. Because these devices are electronic, they cannot be used in electromagnetic noisy places. Also, they show a great dependence on ambient conditions, mainly temperature. Some devices must be heated to 400°C just to operate, which implies a great waste in energy and poor mobility.

Fiber optic sensors have many characteristics that give them an advantage over conventional ones. They are compact and lightweight, in general are minimally invasive,

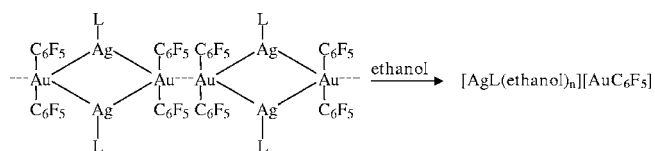


Fig. 1 Molecular structure of the new developed vapochromic material and its reaction in the presence of ethanol vapors.

immune to electromagnetic interference, can be multiplexed effectively on a single fiber network, etc. The main advantage of fiber optic sensors is tackling difficult measurement situations where conventional sensors are not well suited for use in a particular environment. Actually, many sensors have already been developed. Some are based on Fabry-Perot interferometers,¹⁻³ other sensors are obtained by depositing a material either in the core of an optical fiber⁴⁻⁷ or onto the thinnest zone of a biconically tapered optic fiber.^{8,9} There are also sensors based on the reflection of films deposited at the end of a fiber optic pigtail.^{10,11} The main working principle of fiber optic sensors is the modulation of guided light caused by the sensing material.

In this work, a new vapochromic material whose optical properties (such as color, and therefore, refraction index) change in the presence of VOC (ethanol, methanol, acetic acid, etc...), has been fixed to the end of a cleaved fiber pigtail to perform a VOCs detector sensor.

2 Materials and Instrumentation

The phenomenon of vapochromism (shifts in absorption and emission maxima characteristic for specific organic solvents¹²) has been studied since Nagel¹³ first synthesized Pd and Pt mixed salts as gases and vapor detectors. These salts have been used in the detection of volatile organic compounds (VOCs) with optical fiber sensors.^{14,15} Gold complexes¹⁶ and bis(phthalocyaninate) rare-earth compounds are also applied in the sensor field due to their vapochromic characteristics.

In previous research, we found some gold complexes with vapochromic characteristics.¹⁷⁻¹⁹ We present a new vapochromic polynuclear silver-gold complex to detect and quantify VOCs such as ethanol, methanol, and acetic acid. This compound, of molecular formula $\{Au_2Ag_2(C_6F_5)_4[(C_5H_4N)-(C_5H_4N)]_2\}_n$, changes its color (and therefore its refractive index) from bright yellow to red when it is exposed to organic solvent vapors.

Figure 1 shows the molecular structure of the sensing material synthesized in our laboratories. The mechanism, which occurs when the VOC is in contact with the vapochromic material, is well known: the polynuclear structure disappears due to the break down of first the gold-gold contacts and then the gold-silver bonds, and the solvent is coordinated with the silver center. The new anion-cation derivative formed, $\{Ag[(C_5H_4N)-(C_5H_4N)](VOC)_n\} \times [Au(C_6F_5)_2]$, has a different refractive index compared to the first one, allowing the parameters to define the reflectance changes while the VOC molecules are in contact with the vapochromic material. This organometallic complex

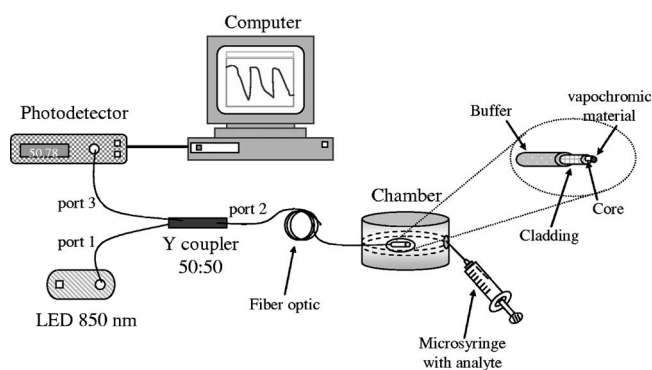


Fig. 2 Experimental setup implemented to characterize the sensor's behavior.

needs a big quantity of solvent to show a color change, although a small quantity of solvent is able to induce detectable changes in the refractive index.

It was necessary to fix the vapochromic material into the cleaved end of the fiber and Liquicoat[®] was chosen, a commercially available solvent, purchased from Sigma (St. Louis, MO, USA) and used as supplied. This product is a metal alkoxide colloidal solution containing 7% silica and is normally used for the deposition of antireflective coatings on displays, but it has been used to detect chemical parameters in fiber optic sensors²⁰ and to fabricate optical waveguides.^{21,22}

The sensor is made up of a plastic cladding-silica core (PCS) multimode optic fiber pigtail (core and plastic cladding diameters 200 and 230 μ m, respectively), the end of which is cut with a Siemens S46999-M9-A8 precision fiber cleaver. The principal advantage of this fiber is the ease of removing its plastic cladding. As the optical source, it is not necessary to use a laser but a LED, so the sensor system could be fabricated at a lower cost. The core's fiber width ensures a high numeric aperture, making easy coupling light into the fiber.

The experimental setup to measure the reflected optical power is shown in Fig. 2. A Y coupler 50:50 was used to connect the system. This device has also a 200- μ m core diameter to avoid insertion losses. The sensor head is con-

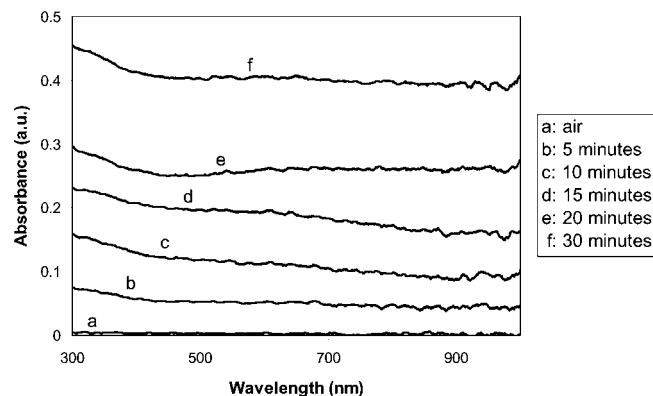


Fig. 3 Absorbance spectrum of vapochromic material when it is exposure to 1 ml of methanol after 0 (outside in air), 5-, 10-, 15-, 20-, 25-, and 30-min exposure.

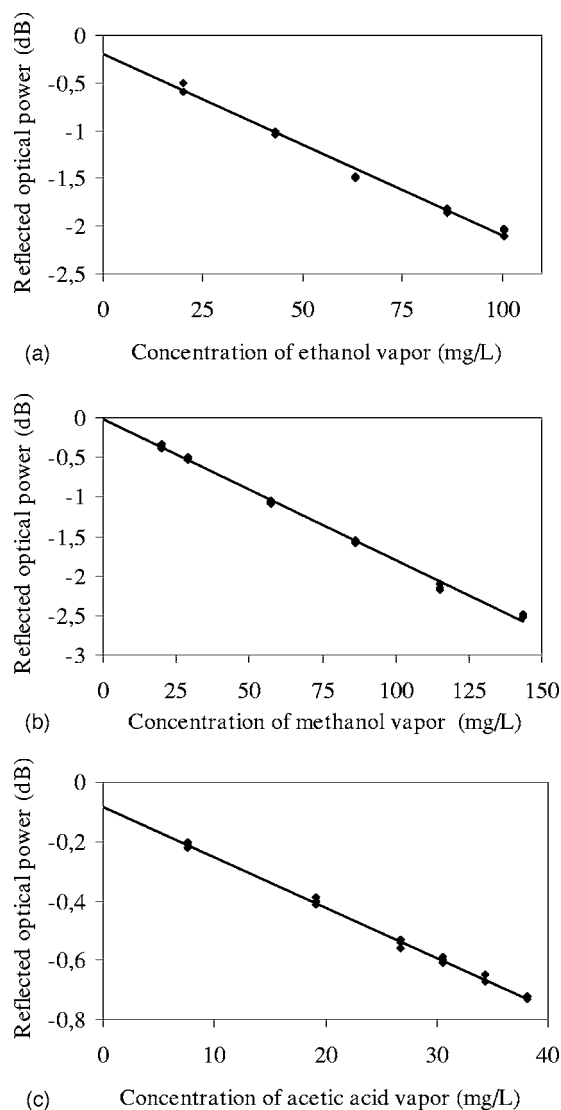


Fig. 4 Sensor's response with different VOC concentrations at 850 nm: (a) ethanol, (b) methanol, and (c) acetic acid.

nected to port 2 and introduced into the chamber (9 cm diam and 2 cm high, 275 cm³ volume), and closed hermetically. The other two ports are connected to the LED source (port 1) and to the photodetector, 675RE from RI-FOCS Corporation (Camarillo, CA, USA) (port 3).

3 Experimental Results and Discussion

To produce VOC sensitive films, different coatings based on Liquicoat[®] solution and vapo-chromic material were deposited on microscopic glass slides, varying the volume ratio of Liquicoat[®] and vapo-chromic material. The coatings were made using a concentration of 1 mg of vapo-chromic material per 10 μ L of Liquicoat[®], after several experimental attempts with different ratios. This ratio was the optimum for obtaining coatings without cracks and with enough vapo-chromic material for a proper optical response.

Ten films were deposited at the end of the fiber using the dip-coating technique. This technique has to be carried out with great precision, therefore a mobile platform is used at 1-cm/s velocity, obtaining coatings with a uniform profile

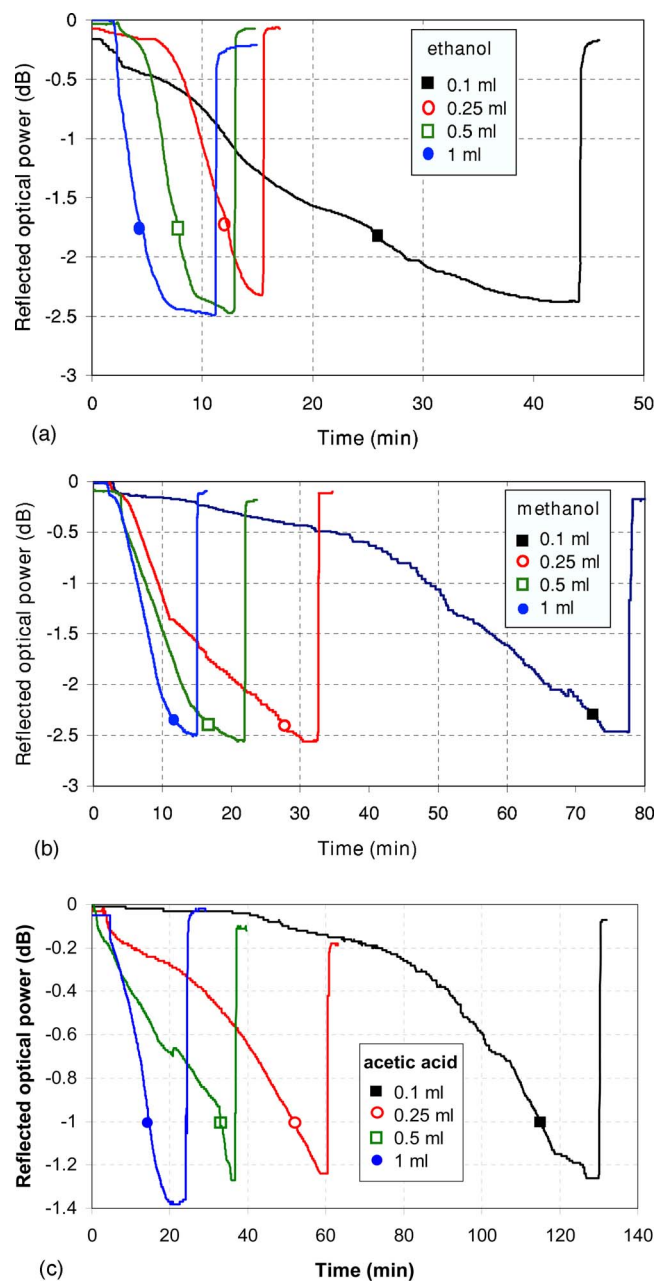


Fig. 5 Behavior of the sensor versus different volumes of the three VOCs when the atmosphere is saturated with vapor: 1, 0.5, 0.25, and 0.1 ml of (a) ethanol, (b) methanol, and (c) acetic acid.

and yielding to an acceptable reproducibility. The solvent needs one day to evaporate; after that, the vapo-chromic complex stays perfectly adhered to the end of the fiber.

3.1 Absorbance Spectra Analysis

The absorbance spectra analysis is carried out using the same reflection setup shown in Fig. 2, but with a white light source connected to port 1 and a CCD spectrometer, PC2000 connected to port 3, instead of a LED and a photodetector, respectively. The absorbance is shown in absorbance units, defining it as:

Table 1 Analytical performance characteristics for the method when the atmosphere is not saturated with vapor. S_0 is the standard deviation of the blank, and RSD is the relative standard deviation for an intermediate point of the calibration curve.

<i>Nonsaturation situation</i>	Ethanol	Methanol	Acetic acid
Slope	0.0191	0.0178	0.0170
Intercept	0.1944	0.0241	0.081
Correlation coefficient	0.9945	0.9980	0.9913
Linear range (mg/l)	2.16 to 100.50	1.73 to 143.60	3.73 to 38.20
s_0	0.013	0.011	0.023
Detection limit (mg/l)	2.16	1.73	3.73
Quantification limit (mg/l)	7.18	5.76	12.44
RSD %	0.671	1.437	1.741

$$A = \frac{S_\lambda - D_\lambda}{R_\lambda - D_\lambda},$$

where S_λ is the optical power received from the sample, D_λ is the optical power of the dark reference signal, and R_λ is the optical power monitored from the reference signal, all of them at wavelength of λ .

An absorbance spectra analysis was carried out to check whether or not the material used is sensitive to VOCs. Figure 3 shows the wide spectral region where the optical signal changes. The absorbance spectrums are almost flat from 400 nm, and exhibit a smooth curve in the 300 to 400-nm band. This means that there is a change in the refractive index, but no remarkable color change is achieved (remember that a big quantity of solvent is required to achieve this). This gives the system an important degree of flexibility and the possibility of fabricating different sensors at distinct working wavelengths and implementing a sensor network using, for example, a wavelength division multiplexing scheme in a straightforward way.

3.2 Reflected Optical Power Analysis

Although the results presented here are obtained with a 850-nm LED source, it is necessary to comment that other optical sources have also been tested with different coherent lengths and at different working wavelengths. The higher changes were obtained using a simple LED source at 850 nm, and the absorbance spectra shows that there is an optimal detectable change in absorbance at this wavelength. Therefore, all the results presented here are referred to this source.

Using the setup of Fig. 2, when ethanol, methanol, or acetic acid liquid are introduced into the chamber and the vaporization starts, the optical power decreases until it reaches a stable value. Once the chamber opens, the vapors quickly vanish and the reflected optical power returns to its initial values.

The variation in reflected optical power is bigger with greater volumes of VOC introduced, until the atmosphere inside the chamber is saturated with vapor. Then, this variation remains constant, although greater volumes of VOC

were introduced. On one hand, when there was no saturation inside the chamber, it was possible to detect and quantify different concentrations of ethanol, methanol, and acetic acid vapors. On the other hand, when there was saturation vapor inside the chamber, it was possible to relate the fall slopes of the reflected optical power obtained at the photodetector with the evaporation speed of the liquid, and with the volume of liquid introduced.

Therefore, the experimental work has been divided into two sections: one in a nonsaturated situation, and the other with saturation. In the first section it is demonstrated that it is possible to detect and quantify different concentrations of ethanol, methanol, and acetic acid vapors. In the saturation situation, only different liquid volumes, not vapor concentration, are determined because the vapor concentration will always be the same inside the chamber.

It is also shown that the sensor's response time is between 20 to 30 min, which is slower than the results obtained with other vapochromic complexes. The solvent employed to fix the vapochromic material to the end-cleaved fiber, is shown to be transparent to chemical agents once it is dried,²⁰ so time response is an intrinsic characteristic of the vapochromic complex.

3.2.1 Measurements in nonsaturation situation

Taking into account the vapor pressure of the liquids (43.51-, 92.5-, and 12-mm Hg for ethanol, methanol, and acetic acid, respectively), the volume of the chamber (275 cm³), and the temperature (20°C), it is possible to calculate the minimum liquid volume, which produces saturation of the chamber atmosphere: 38.2 μ L for ethanol, 56.4 μ L for methanol, and 10.3 μ L for acetic acid. By introducing lower volumes of VOCs than these values, different vapor concentrations are determined and quantified.

To evaluate this methodology, standard linear calibration graphs were done (see Fig. 4). The measurements were repeated three times to determine error. Analytical performance characteristics were evaluated using the International Union of Pure and Applied Chemistry (IUPAC) method²³ and are summarized in Table 1.

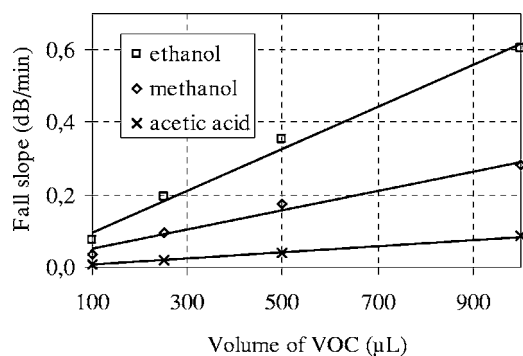


Fig. 6 Fall slopes of the sensor's response when exposed at different volumes of methanol, ethanol, and acetic acid.

3.2.2 Measurements in saturation situation

If greater volumes than the previously mentioned values are introduced into the chamber, the optical power decrease is the same, but the fall slopes of the output reflected optical power will be different, as shown in Fig. 5. In that way, a saturation situation inside the chamber has been reached and the vapor concentration is the same. It is easy to understand from these experimental graphics that greater volumes of VOC introduced into the chamber produce bigger fall slopes in the sensor response.

These slopes let us distinguish between the different VOCs studied due to their different kinetics, as can be inferred from Fig. 6, where the fall slopes of the optical sensor response at different volatile organic compound volumes are presented. As can be seen, the response time of the optical fiber sensor to ethanol, methanol, and acetic acid is clearly different. This fact, with the help of appropriated software, could be further taken into consideration for distinguishing between different VOCs, and could be useful in optoelectronic noses, just to mention an example.

Standard linear calibration graphs were drawn and all the features are summarized in Table 2.

3.2.3 Influence of environmental conditions

How the changes in temperature and humidity can influence the sensor response has been studied by means of a climatic chamber. This setup is similar to Fig. 2 but introduces the sensor head inside the climatic chamber. Here, the conditions of humidity and temperature are changed using a computer, where three cycles of four hours each have been programmed. Within each cycle, the temperature has stayed constant, at 20, 40, and 60°C, respectively, and the humidity has been varied from 15 to 85%, returning to 15% and beginning a new cycle with another temperature. The results show that there are no significant influences of these two variables in the sensor response, as the reflected optical power changes less than 0.1 dB when the temperature and humidity are varied in the range studied.

4 Conclusions

A low-cost volatile-organic-compound fiber-optic sensor is developed. The sensor can detect low concentrations of vapors of ethanol, methanol, and acetic acid. Its low cost, easy implementation, and the possibility of multiplexing into either a telecommunication or a sensor network make

Table 2 Analytical performance characteristics for the method when the atmosphere is saturated with vapor. S_0 is the standard deviation of the blank, and RSD is the relative standard deviation for an intermediate point of the calibration curve.

Saturation situation	Ethanol	Methanol	Acetic acid
Slope	0.0006	0.0003	9×10^{-5}
Intercept	0.0383	0.0238	0.0009
Correlation coefficient	0.9957	0.9908	0.9981
Linear range (μ l)	68.61 to 1000	54.99 to 1000	118.79 to 1000
s_0	0.014	0.011	0.023
Detection limit (μ l)	68.61	54.99	118.79
Quantification limit (μ l)	228.69	183.30	395.96
RSD %	1.762	1.583	1.952

this sensor suitable for use in environmental applications, optoelectronic noses, or chemical industries.

It is important to emphasise that the sensor has been used continuously in the laboratory for more than three months and neither degradation in the material or variation in its behavior has been observed.

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