

PMMA-coated fiber Bragg grating sensor for measurement of Ethanol in liquid solution: manufacturing and metrological evaluation

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ABSTRACT

The sensitivity of Fiber Bragg Gratings (FBGs) to the variation of a specific parameter can be improved by choosing a suitable coating material. This paper explores the possibility of measuring the concentration of Ethanol in aqueous solutions by using poly(methyl methacrylate) (PMMA) as coating material of a single-mode FBG. The basic measurement principle relies in the absorption of liquid Ethanol which produces controlled swallowing of the coating, straining the underneath Bragg grating. The PMMA coating was deposited on the FBG by means of a specifically designed dip coating test bench, able to accurately control the thickness of the applied layer. Metrological performances of the developed PMMA-coated FBG Ethanol sensor have been assessed for Ethanol concentrations in the range of 0-38 % in distilled water at a constant temperature of 25 °C. The prototype shows good repeatability and better sensitivity when compared to a traditional FBG, especially at low Ethanol concentrations. The dynamic behavior of the sensor (which depends on the kinetics of the absorption mechanism) has been assessed as well, showing a response time of about 290 s, while the recovery time (660 s) was almost twice.

Section: RESEARCH PAPER

Keywords: FBG sensors; dip coating; Ethanol sensors; concentration evaluation in liquids; PMMA coating

Citation: Antonino Quattrocchi, Roberto Montanini, Mariangela Latino, Nicola Donato, PMMA-coated fiber Bragg grating sensor for measurement of Ethanol in liquid solution: manufacturing and metrological evaluation, Acta IMEKO, vol. 10, no. 2, article 19, June 2021, identifier: IMEKO-ACTA-10 (2021)-02-19

Section Editor: Ciro Spataro, University of Palermo, Italy

Received January 18, 2021; **In final form** April 30, 2021; **Published** June 2021

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1. INTRODUCTION

Fiber Bragg grating (FBG) sensing is a mature technology that has been used so far to develop measurement solutions for a wide range of applications. Currently, they are commonly employed for health structural monitoring, as well as to fabricate distributed sensing devices to be used in different fields such as medicine and biomedical research, automotive, aerospace, maritime and civil engineering, and many others. FBGs are largely preferred over other types of optical sensors [1] thanks to their unique advantages. They present small sizes, immunity from electromagnetic disturbances, good resistance in terms of temperature and humidity and, above all, they are wavelength encoded and chemically inert. For these reasons, they often represent the favorite choice for applications in critical environments, e.g. explosive or radioactive [2].

The sensitivity of FBG to the variation of a specific parameter can be improved by choosing a suitable coating material. The basic principle relies in the swallowing of the coating material which eventually induces a change in the grating period and in the refractive index along the fiber length. Several types of materials and deposition techniques can be used to create a fiber coating for a specific application. FBG-based humidity sensors has been realized by dip coating the fiber in a polyamide solution [3], by depositing a layer of graphene oxide on the fiber surface [4] and by embedding the FBG in a steel and carbon fiber reinforced polymer [5]. Montanini et al. [6] compared results obtained on two polymer-coated FBG humidity sensors, using poly(methyl methacrylate) (PMMA) and poly(vinyl acetate) (PVAc) as sensing materials. The calibration of the two sensors was carried out in the 20 - 70 %RH range at three different temperatures (15 °C, 30 °C and 45 °C) by a two-pressure humidity generator. The PMMA-coated sensor prototype displayed overall much better performance when compared with

the PVAc-coated one, although it manifested a higher temperature sensitivity. Instead, FBG-based cryogenic thermometers were developed using coatings of metals (copper, zinc, tin and lead) for the fiber by electroplating and casting [7], by bonding the FBG onto Teflon substrates [8] and by placing a layer of ORMOCER on the fiber surface by gravity-coating [9]. Another specific application was developed by Campopiano et al. [10]. The authors produced several types of hydrophones, using plastic-coated FBGs. They evaluated how the use of polymers, which exhibits great variability in terms of obtainable geometries and acoustic and mechanical properties, guarantees the achievement of customized performances.

An important field of application is that of the chemical industry. Cong et al. [11] proposed an optical salinity sensor based on a hydrogel-coated FBG. The detection mechanism relies on the mechanical stress induced into the coating material when water comes out of it. The main limitation of this device lies in the slow response time due to the balance between the forces acting at the intermolecular level of the coating material. Aldaba et al. [12] investigated a pH sensor consisting of a polyaniline layer polymerized on the surface of a FBG. In this case, the pH detection occurs thanks to the interaction between the optical absorption spectrum of polyaniline, which is sensitive to pH, and the Bragg grating. The device exhibited fast response, biochemical compatibility, temperature independence and long-term stability. Dinia et al. [13] proposed a FBG multifunctional pH sensor, covered with a pH-sensitive hydrogel, to monitor the pH rain on critical and prestigious monuments.

Ethanol is a widely used chemical in many fields, such as medical, food processing and automotive [13]. However, because of its high volatility and flammability, Ethanol can be dangerous for living organisms and, therefore, it is very important to have disposal effective detection systems [15]. Several sensor typologies are available, basing on transduction effect, spanning from resistive sensors [16], electrochemical [17], gas and resonant ones [18, 19].

In contrast, FBG represents an intrinsically safe device, which can be profitable used to measure Ethanol concentration either in vapor or liquid phase. Morisawa et al. [17] analyzed a plastic optical fiber sensor, coated with a layer of Novolac resin, for detecting alcohol vapors, including Ethanol. They putted into evidence that the transmitted light intensity is a function of the coating thickness, of the exposure time, of the alcohol type and of the vapor pressure. Latino et al. [21] presented some preliminary results concerning the assessment of low concentration of Ethanol (0-3 %) in aqueous vapor, using a FBG coated with thin layers of PMMA with different thicknesses. In this case, PMMA was distributed onto the FBG surface with a simple dip coating technique. Coradin et al. [22] evaluated the metrological performance of four FBGs, assembled in several configurations, for analysis of water-Ethanol mixtures. Raikar et al. [23] employed a Ge-B doped FBG to show as the wavelength shift decreases with the increase in liquid solution concentrations, moving from 0 % to 50 %. Arasu et al. [24] developed a FBG coated with a thin gold film deposited by sputtering. Their results displayed as the concentration of Ethanol in water from 0 % to 100 % is better assessed measuring the change in absorbance with the thickest gold layer. Recently, Kumar et al. [25] analyzed a graphene oxide-coated FBG for Ethanol detection in petrol. This sensor was manufactured covering a partially chemical etched FBG with a layer of an oxidize graphite powder by means of a drop casting technique. The detection of Ethanol proportion in petrol, from 0 to 100 %,

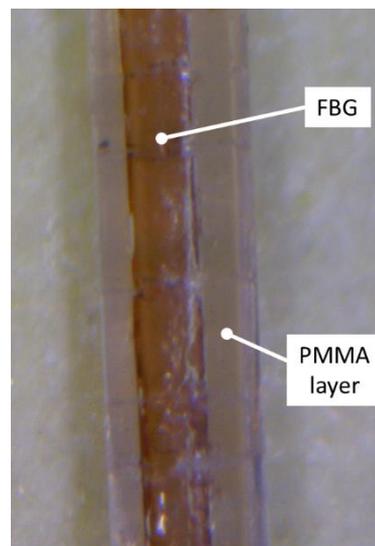


Figure 1. FBG Ethanol sensor with PMMA sensing layer.

was investigated and a calibration curve was obtained. The main result shows as the graphene oxide-coated FBG allows a significant enhancement (by a factor of ten) in the detection of Ethanol in comparison with un-coated FBG.

In this paper, which is an extended version of the one presented at the 24th IMEKO TC4 International Symposium and the 22nd International Workshop on ADC and DAC Modeling and Testing (IMEKO TC4 2020) [26], starting from our previous experience gained with PMMA-coated FBGs designed for sensing Ethanol concentration in aqueous vapor [21], we aim to explore the possibility to measure the concentration of Ethanol in liquid solutions too. The performance of the PMMA-coated FBG has been investigated by evaluating its response to Ethanol concentration at a constant temperature of 25 °C and a calibration curve was obtained and compared with that of a traditional FBG. The main metrological features of the prototype, in terms of sensitivity and response/recovery time, have also been assessed and discussed.

2. MATERIAL AND METHODS

2.1. FBG Ethanol sensor manufacturing

The FBG Ethanol sensor has been realized by recoating a single-mode silica FBG with a thin layer of PMMA. The device has an active length of the Bragg grating of about 14 mm and a total thickness of (0.44 ± 0.03) mm. This coating thickness value was chosen in order to find the best balance between sensing properties and stability ones. The FBG is of the SMF-28 type with a diameter of 250 μm , of which 9 μm of core, 125 μm of cladding and 250 μm of an acrylate coating, a reflectivity > 90 % and a full-width at half maximum (FWHM) < 0.30 nm. Figure 1 reports a magnification of the sensor structure.

The measurement principle relies on the selective absorption of Ethanol by the coating layer made of PMMA. This interaction causes the swelling of the sensing polymer and a consequent mechanical effect on the FBG, which produces a wavelength shift related with the Ethanol concentration in the liquid solution.

In order to develop the FBG Ethanol sensor, the pre-existent coating of the single-mode optical fiber was removed, then, the PMMA deposition was carried out by a controlled dip coating procedure. The FBG was dipped under controlled conditions (constant dipping/rising velocity) in a solution of 560 mg PMMA

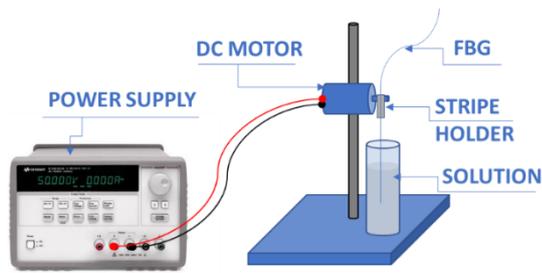


Figure 2. Block diagram of the deposition setup.

and 3 ml of Acetone. The block diagram of the deposition system is depicted in Figure 2. It consisted of a precision DC servo motor, equipped with an Agilent E3632A power supply, an adaptor, and a stripe holder where the sample was held. By means of the power modulation delivered to the servo motor, it was possible to control the rising/dipping rate of the FBG in the PMMA-Acetone solution.

Figure 3 reports the relationship between the voltage applied to the servo motor and the resulting dipping rate, showing a clear linear trend. Hence, by simply varying the rising/dipping rate, it was possible to accurately control the thickness of the coating layer.

2.2. Measurement setup

The measurement setup consisted of an optical spectrum analyzer (OSA) (Micron Optics mod. SI 720) with ± 3 pm accuracy, wired to the FBG Ethanol sensor and to a PC. A dedicated graphical user interface, developed in Labview™ environment, was implemented to display in real time and to store the data, using a National Instruments GPIB-USB-HS IEEE-488.2 communication protocol. The FBG Ethanol sensor was placed into a cylindrical metallic container, filled with the target solution of distilled water and Ethanol. The temperature of the metallic container was kept constant through a thermostatic bath, with the aim to minimize the temperature dependence of the FBG and better relate the sensor response with the Ethanol concentration. Figure 4 illustrates a block diagram of the measurement setup.

3. RESULTS

3.1. Response of the FBG Ethanol sensor

Figure 5 shows the power spectra of the FBG Ethanol sensor obtained for three different concentrations of Ethanol in water

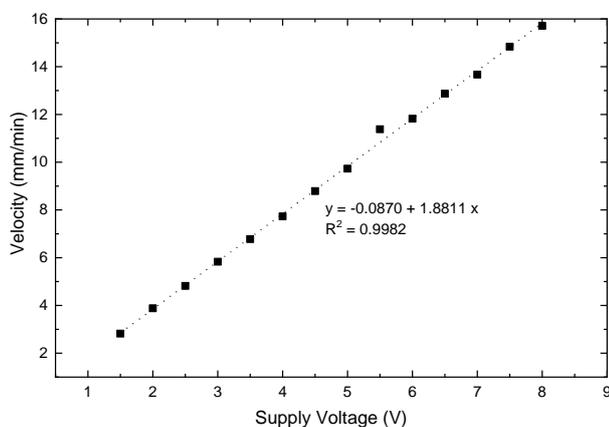


Figure 3. Relationship between the supply voltage to the DC servo motor and the resulting dipping rate.

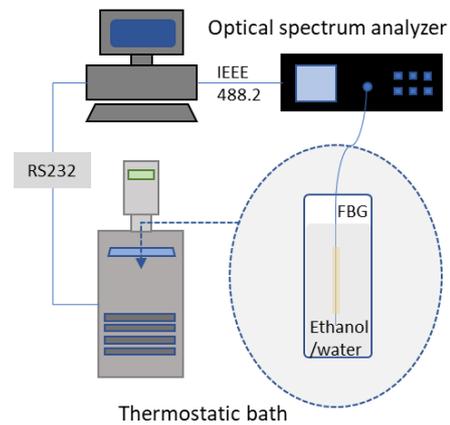


Figure 4. Block diagram of the measurement setup.

at a constant temperature of 25 °C. The peak of each curve shifts to the right as the Ethanol concentration increases, while the maximum amplitude of the power can be considered sufficiently constant. This highlights how the interaction between the PMMA coating and Ethanol results only in a shift of the wavelength of the Bragg grating, without substantial modification of the shape of the reflected signal.

Hence, the FBG sensor response at constant temperature can be defined considering the relative shift of the Bragg wavelength $\Delta\lambda$ with respect to the baseline wavelength in distilled water λ_{H_2O} (i.e. 0 % v/v of Ethanol, reference temperature T_0 of 25 °C):

$$\Delta\lambda = \lambda_R - \lambda_{H_2O}, \quad (1)$$

being λ_R the actual wavelength of the FBG sensor as it is exposed to a definite concentration of Ethanol in distilled water.

Figure 6 displays the FBG response as it is repeatedly immersed firstly in distilled water and eventually in a water/Ethanol solution of 7.89% v/v at a constant temperature of 25 °C. The obtained data highlight a large variation of the FBG wavelength: in air, the wavelength λ_0 is 1554.73 nm and it rapidly increases to 1555.99 nm (λ_{H_2O}) as the FBG Ethanol sensor was immersed in distilled water. This occurs because the PMMA layer hydrates in contact with water, and it begins to expand until a steady state condition has been reached. As the sensor is exposed to the water/Ethanol solution, Ethanol is absorbed by the PMMA layer and the swelling process restarts until a new steady state condition is gained. The described interaction between PMMA and

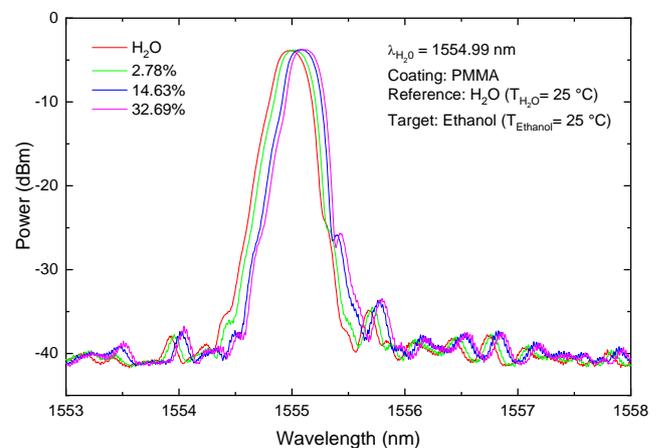


Figure 5. Power spectra of the FBG Ethanol sensor for different concentrations of Ethanol in pure water.

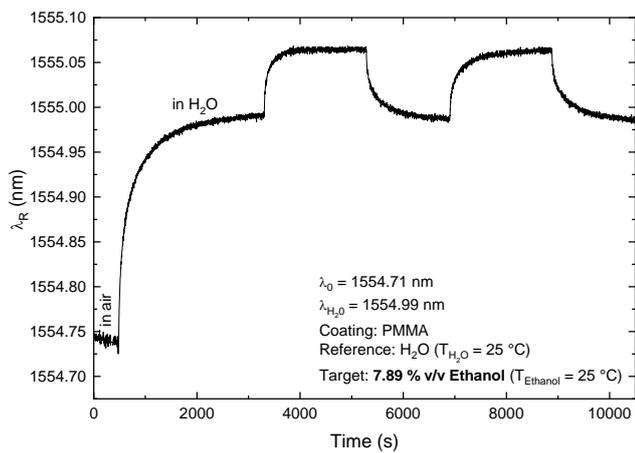


Figure 6. Different response of the FBG Ethanol sensor in air, into distilled water and exposed at a fixed Ethanol concentration (7.89 % v/v).

Ethanol is reversible, since the baseline is reached when the sensor is exposed to distilled water. Then, a proper calibration procedure allows to evaluate Ethanol concentration in the whole characterization range.

Figure 7 presents an example of data recorded in real-time, keeping the temperature constant at 25 °C and varying the Ethanol concentration from 2.78 % v/v to 5.40 % v/v, triggering the OSA on the maximum marker. Even though the FBG Ethanol sensor response is subject to a transient before reaching the steady state condition, it is evident that a greater value of λ_R is correlated to an increase of Ethanol concentration. Furthermore, the measurement process is reversible since the acquired wavelength coincides with the basic one ($\lambda_{H_2O} = 1554.99$ nm), when the FBG Ethanol sensor is placed into distilled water.

Figure 8 puts into evidence the response of the FBG Ethanol sensor by increasing Ethanol concentrations at a constant temperature of 25 °C. It can be clearly seen how an increasing in Ethanol concentration brings to a corresponding increasing of the λ_R in the whole Ethanol investigation range.

3.2. Metrological evaluation of the FBG Ethanol sensor

In this section will be evaluated the metrological performance of the FBG Ethanol sensor, such as repeatability and the calibration curve. Figure 9 reports the repeatability of the FBG Ethanol sensor, when it is exposed to three pulses of 7.89 % v/v

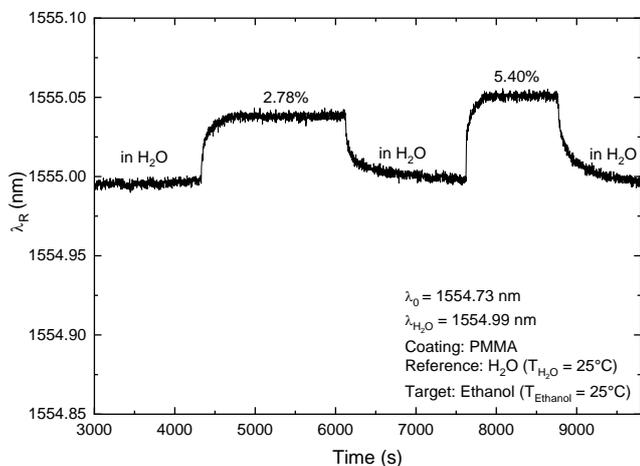


Figure 7. Wavelength shifts after exposition of the FBG Ethanol sensor to different concentrations of Ethanol.

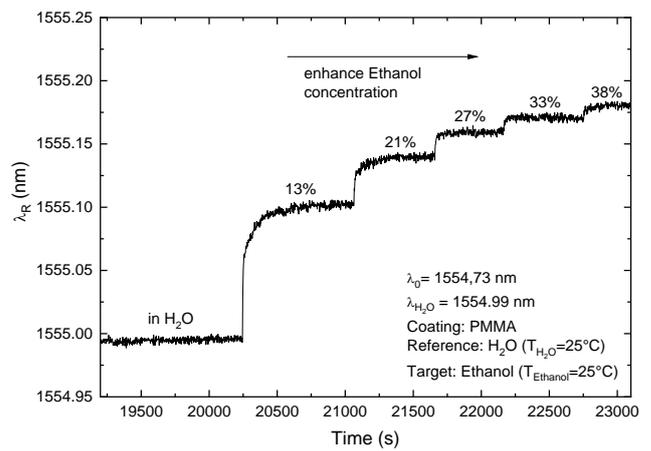


Figure 8. Response of the FBG Ethanol sensor at progressive Ethanol concentrations.

of Ethanol and again to distilled water at a constant temperature of 25 °C. The repeatability was estimated at an Ethanol concentration of 7.89 % v/v and for the distilled water (base line); here are reported a mean value of 0.075 nm with a standard deviation of 0.005 nm, and 0.071 nm and 0.003 nm, respectively. Therefore, peak values of λ_R and λ_{H_2O} show a good repeatability and reversibility.

Figure 10 a) compares the calibration curves of the FBG and the PMMA-coated FBG (i.e. the FBG Ethanol sensor) for Ethanol concentrations ranging from 0 to 38 % v/v at a constant temperature of 25 °C. It can be seen a linear trend for the FBG vs. Ethanol concentration. This is probably due to Ethanol absorption by the Acrylate coating of the FBG itself. On the other hand, the PMMA-coated FBG exhibits more complex behavior. In the latter case, although a linear branch can be observed for low concentration values, a polynomial curve is able to fit the data in the whole Ethanol range. A such nonlinear trend could be caused by the binding forces between the PMMA molecules, which could then limit the swelling of the coating layer (i.e. the Ethanol absorption) above a defined threshold. However, the sensitivity of the PMMA-coated sensor is much greater (more than one order of magnitude) than the FBG one. While Figure 10 b) reports the residual values of both the previous fitting curves.

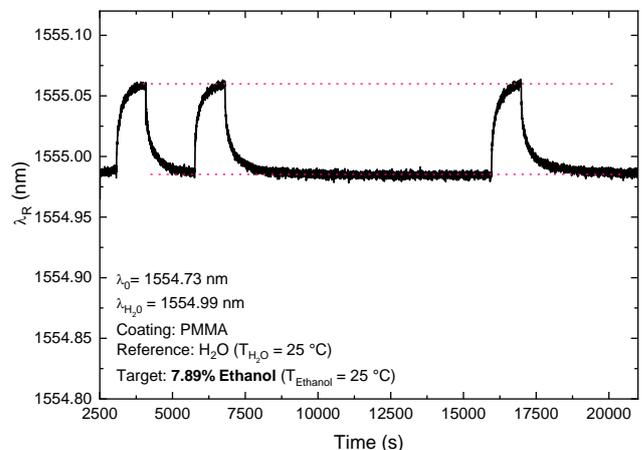
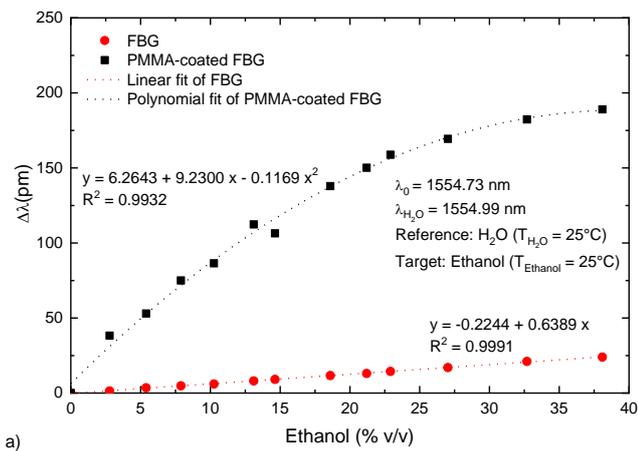
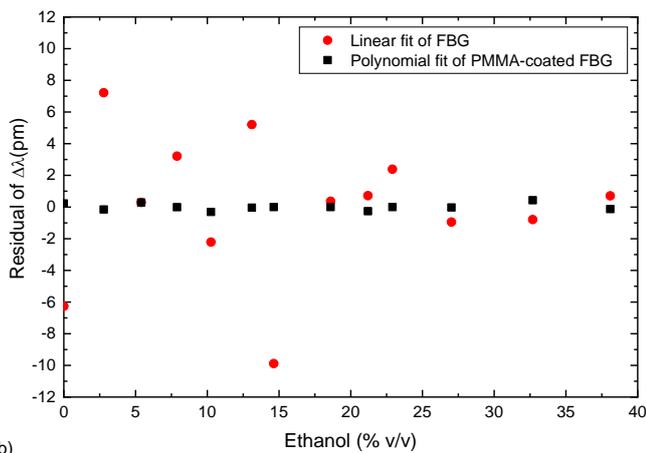


Figure 9. Repeatability of the FBG Ethanol sensor exposed at a fixed Ethanol concentration of 7.89 % v/v.



a)



b)

Figure 10. a) comparison between the calibration curves of the FBG and the PMMA-coated one and b) residual analysis of the fittings.

Figure 11 displays the first derivatives of the calibration curves towards time, for both the FBG and the PMMA-coated one. These curves give the sensitivity of the devices, highlighting that the PMMA coating leads to a significant increase of the FBG sensitivity to Ethanol. It is also noteworthy to observe that the sensitivity of the PMMA-coated FBG is not constant, but it strongly depends on the specific Ethanol concentration. On the other hand, the sensitivity of the FBG can be considered constant. Furthermore, the Acrylate coating of the FBG has a considerably lower absorption of Ethanol compared to the PMMA coating, as already highlighted by Figure 10 a).

3.3. Behavior of the FBG Ethanol sensor

The FBG Ethanol sensor exhibits a dynamic behavior as a function of the kinetics of the swelling phenomenon of the PMMA layer.

The typical values of the response and the recovery time are identified in Figure 12 for an Ethanol concentration of 7.89 % v/v at a temperature of 25 °C. These parameters are calculated as the time to reach the 90 % of the final value, when the sensor is exposed to Ethanol, and the time to reach the 90 % of the baseline wavelength, when the sensor is exposed to distilled water, respectively. The response time value is of 290 s, while the recovery one is of 660 s. By considering these results, it is possible to note how the sensing of Ethanol (i.e. the absorbing process) is faster than the desorbing one (the process to reach again the baseline wavelength in presence of only distilled water).

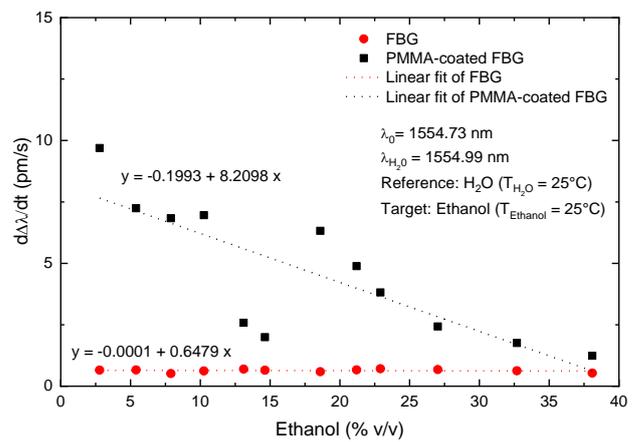


Figure 11. Sensitivity vs. Ethanol concentration for FBG and for PMMA-coated one.

4. CONCLUSIONS

This paper describes the manufacturing process and the metrological characterization of a FBG-based Ethanol sensor obtained by covering the Bragg grating with a layer of PMMA. The obtained device is able to evaluate the different Ethanol concentrations in distilled water at a constant temperature of 25 °C. In fact, the measurement data have shown a clear shift in the wavelength of the Bragg grating, strictly related to the increasing in Ethanol concentration. The FBG Ethanol sensor identifies specific and progressive concentrations of Ethanol, showing good repeatability. Its calibration curve highlights a polynomial trend that easily describes the whole range of considered concentration (from 0 % to 38 % in v/v of Ethanol in distilled water). Furthermore, compared to the calibration curve for FBG, better sensitivity has been achieved, especially for the low concentration of Ethanol. Finally, dynamic behavior for a fixed Ethanol concentration (7.89 %) has also been assessed. In this case, the response time (290 s) is more than half of the recovery one (660 s).

Future work will be focused on the cross-sensitivity investigation of the analyzed PMMA-coated FBG, in order to evaluate the selectivity of the device to other chemical compounds.

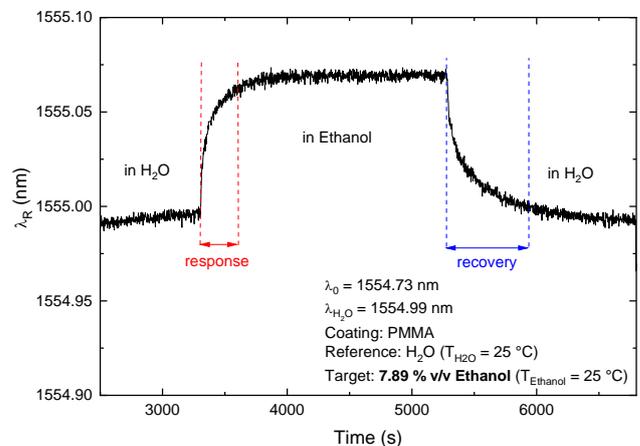


Figure 12. Response and the recovery time of the FBG Ethanol sensor exposed to a fixed Ethanol concentration (7.89 % v/v).

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