

# Tapered Single-Mode Optical Fiber pH Sensor Based on Lossy Mode Resonances Generated by a Polymeric Thin-Film

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**Abstract**— Lossy mode resonances can be generated with certain polymeric nanostructures, such as those obtained with a multilayered assembly of poly(allylamine hydrochloride) and poly(acrylic acid). This coating is adsorbed by the electrostatic self assembly technique onto a tapered single-mode optical fiber, in order to evaluate its performance when detecting pH. According to the results reported in this work, the high sensitivity given by a tapering process in a single-mode optical fiber is increased by the effect of this kind of electromagnetic resonances. Particularly, in a pH range from 4.0 to 6.0 the overall wavelength shift of this sensor reaches 200 nm and the transmission at the resonance wavelengths can fall down to -50 dB. These data provide results which can be taken into account to detect pH with high accuracy.

**Index Terms**— Polymeric thin-films, lossy mode resonances, optical fiber sensors, tapered single mode fibers.

## I. INTRODUCTION

OPTICAL fiber sensors have been analyzed and reported extensively during the last decades [1-5]. It is well known that there are two main types of fibers: multimode fiber (MMF) and single-mode fiber (SMF). Moreover, several structures can be fabricated with these devices, such as long period gratings (LPGs), fiber bragg gratings (FBGs), hollow cores (HCFs) or tapered optical fibers (TOFs). In addition, optical fibers present good properties such as electromagnetic immunity, light weight, low transmission losses in the communication windows, wide bandwidth and easiness to handle [6]. This leads to a great variety of possibilities as far as sensing applications is concerned.

Among the different structures mentioned before, cladding removed optical fibers and tapered single-mode optical fibers (T-SMFs) are especially interesting because they permit to access the evanescent field of the modes propagating along the fiber. However, this access is better in the case of T-SMFs

since parameters such as waist length and waist diameter permit a better control of the evanescent field [7,8]. This is the sense for using T-SMFs in this work.

Regarding the fabrication procedure, there are several theories and methods when tapering optical fibers, such as electric arc discharge or heating with a traveling burning flame while fibers are tapered. This last method is used in this work [9-11]. The structures obtained with these methods can be classified into two main groups: adiabatic and non-adiabatic tapers [12-14]. Adiabatic T-SMFs present no power losses through the spectrum since there is a coupling between the fundamental mode ( $HE_{11}$ ) and  $HE_{12}$  mode [15]. On the other hand, in non-adiabatic tapers there is a coupling to higher order modes, which generates modal interferometry and, therefore, oscillations. In this contribution adiabatic tapers are used, which permits to avoid the effects of higher order modes.

Apart from that, the adsorption of a thin-film onto an optical waveguide can introduce losses to the propagation of light in the optical waveguide, leading to electromagnetic resonance phenomena. Depending on the properties of the thin-film, several cases can be distinguished [16].

The most well-known is surface plasmon resonance (SPR). A SPR occurs when the real part of the thin-film permittivity is negative and higher in magnitude than both its own imaginary part and the permittivity of the material surrounding the thin-film (i.e. the optical waveguide and the surrounding medium in contact with the thin-film). In this case coupling occurs between light propagating through the waveguide and a surface plasmon [17,18].

A less explored group is guided mode resonances or lossy mode resonances (LMRs) [19-22]. They occur when the real part of the thin-film permittivity is positive and higher in magnitude than both its own imaginary part and the material surrounding the thin-film.

The properties of LMRs can be obtained with either TE polarized or TM polarized light. In this sense, multiple resonances can be generated without modifying the optical fiber geometry, and they can be obtained with a wide variety of materials, e.g. polymers and semiconductors, with different properties than those typical of SPR: metals.

It has been proved that the polymeric LbL combination of poly(allylamine hydrochloride) and poly(acrylic acid) [PAH /

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PAA]<sub>x</sub> permits to construct a thin-film that, deposited on a cladding removed multimode optical fiber, leads to the generation of LMRs with an optical set-up [23]. The aim of this work is to study the combination of both the T-SMF and the polymeric-induced LMR for the study and development of an optical fiber pH sensor based on this technology. The high sensitivity obtained generally by detecting with a T-SMF is expected to be increased by the effect of a [PAH / PAA]<sub>x</sub> nanostructure, which induces a LMR that provides properties such as good wavelength resolution and therefore, reliability.

The remainder of this paper is organized as follows. First of all, the set-up and the materials used to fabricate the nanostructure are presented in section II. The construction and the results obtained with this sensor are approached in section III. Finally, the corresponding conclusions are extracted in section IV.

## II. SENSOR DEVELOPMENT

### A. Materials

Poly(allylamine hydrochloride) (PAH) (Mw ~ 65,000) and poly(acrylic acid) (PAA) (Mw ~ 15,000) were purchased from Sigma-Aldrich Inc. Based on them, two polyelectrolytic solutions were prepared. The polycationic one was a 10 mM PAH aqueous solution, whereas the polyanionic one was a 10 mM solution of PAA in water. The solvent used for the solutions was ultrapure deionized water (18.1 MΩ / cm) and they were adjusted at pH 4.5. The pH of the solutions was verified by using an electronic pH-meter (Crison Inc.) and adjusted with hydrochloric acid (HCl 1 M) or sodium hydroxide (NaOH 1 M), from Panreac Inc., until the optimal value was reached.

To detect pH, several PBS buffers with values 4.0, 5.0 and 6.0 were purchased from Panreac Inc. Stock pH solutions were prepared by mixing the tampon solutions with ultrapure water in a ratio 1:5 and adjusting the exact value by dropping acid or base.

Regarding the optical material, a SMF-28 standard single-mode optical fiber was provided by Telnet-RI. A super-luminescent emitting diode (SLED) HP-83437A white light source between 1150 and 1680 nm launched the optical power into the fiber and an optical spectrum analyzer (OSA), HP-86142A, detected the optical output response.

### B. Fabrication of the T-SMF and experimental set-up

Before the thin-film deposition process, the substrate was pre-treated. One meter piece of SMF fiber was stripped and cleaned with ethanol in order to remove the covers and to clean the surface. The tapering process was based on a travelling burner with a small flame in which the pulling process was controlled, as reported in [11]. The system, designed by [23], permitted to heat the fiber while it was tapered, which permitted to adjust the required parameters to obtain a specific taper geometry. In this case, the dimensions of the sensitive region were adjusted to 30 μm of diameter ( $\varnothing_w$ )

and 10 mm of length ( $L_w$ ) in the uniform waist region, and 3 mm length ( $L_t$ ) in the linear transition region, as it is shown in Fig. 1.

Once the structure was fabricated, it was fixed to a glassy framework to keep it straight, and then spliced to SMF pigtailed connected to the light source and detector. The initial losses of the T-SMF were less than 1 dB and the spectrum did not present oscillations, which indicated that the taper was adiabatic.

### C. Deposition of the nanocoating and LMR generation

As mentioned in the abstract, the layer-by-layer electrostatic self assembly (LbL - ESA) technique [24-26], was used to deposit the polymeric thin-film onto the tapered fiber (Fig. 1). Prior to start the process, the substrate was fixed to a 3-axis robotic arm designed by [23] and immersed in piranha solution (70% H<sub>2</sub>SO<sub>4</sub>, 30% H<sub>2</sub>O<sub>2</sub>) during 10 minutes in order to acquire negative charge. Then, it was rinsed with ultrapure water to remove undesired impurities.

Once the T-SMF was pre-treated, the first step was to immerse it in the PAH solution during two minutes, to assemble positive charges. Here, some of the polymeric chains were adsorbed onto the substrate, whereas some others did not, so they needed to be removed. That is why the next step was to clean up the fiber with ultrapure water. Then, this substrate was immersed in the PAA solution during two minutes, which provided negative charge to the T-SMF. Finally, a new rinse of ultrapure water was applied so that those negative chains that may not adhere to the system were removed. At this point, the first polymeric bilayer was formed. This process was repeated 30 times, which were sufficient to center the LMR in the middle of the OSA detection window, between 1150 and 1680 nm. In this way, it was possible to monitorize the wavelength displacements experimented by the LMR when detecting the pH variations, wherever the resonance shifted. Additionally, to characterize the evolution of the LMR during the construction of the thin-film, the climatic conditions were stabilized at 23 °C of temperature and at a relative humidity of 35 %. The transmission spectrum in every bilayer was monitored during the process and it was referenced to the initial spectrum

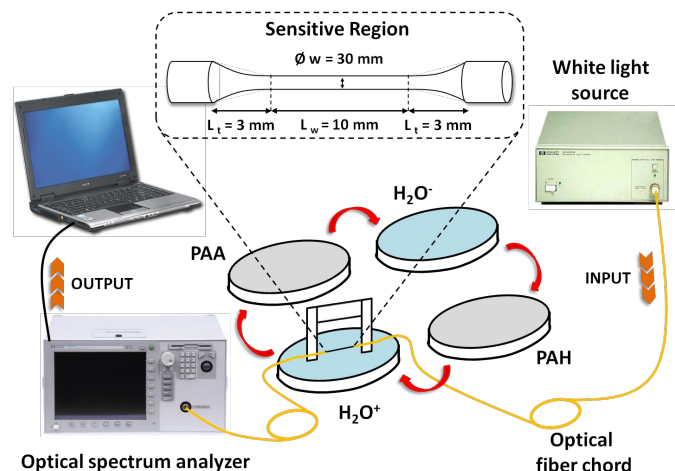


Fig. 1. Set-up of the experiment used to fabricate the pH sensor.

obtained when the sensor was immersed for the first time in the PAH solution.

The thickness of the [PAH / PAA]<sub>30</sub> thin-film is addressed based on the reports of [28,29]. According to [28], by depositing these polymer chains at pH 4.5, it is estimated that the average thickness for a monolayer of PAH is 20.5 Å, whereas the thickness for a PAA monolayer is 46 Å. This means that a [PAH / PAA] bilayer is 66.5 Å thick. Hence, at the end of the process, a thickness of 200 nm is reached by the deposited matrix. Moreover, by applying the algorithm proposed in [29] to our experimental results, a total thickness of 255 nm is estimated. The reason why this value is not the same as that of reference [28] is the difference in the experimental conditions of the deposition. Depending on critical parameters such as the drying time between monolayers, LbL technique can vary from one thickness to another. However, in spite of this, our estimation is close to [28], and the conclusion is that the nanocoating grows up quickly, which is interesting in terms of time efficiency in the achievement of the LMR.

This idea is confirmed in Fig. 2a, which shows the evolution of the transmission of the LMR since it appears at bilayer 22 until the end of the construction process, at bilayer 30. The LMR presents a wavelength shift to the red and the depth of the resonance transmission increases, reaching almost -34 dB at 1410 nm when the experiment ends. This wavelength shift is explained in [22,31] with modal analysis. The resonance wavelength of LMR coincides with the cutoff wavelength of lossy-modes guided in the thin-film. As the thickness of the thin-film increases, there is a progressive red shift of the cutoff wavelength. Consequently, LMRs experiment a red shift. The increase in the resonance depth is explained by the dependence of the thin-film refractive index on the wavelength. Apart from that, the resonance bandwidth, which is dependent on the wavelength [31], is also increased.

This discussion is corroborated with the spectra of Fig. 2b, where the arrows indicate that the LMR experiments an increase in wavelength displacement and depth. Moreover, there is one more aspect to observe, which is the double peak presented by the resonance during its generation. This phenomenon corresponds to the separation of the two different polarizations that form a typical LMR: TE and TM. In fact, the resonance obtained is the contribution of both modes as it is described in [31].

### III. DETECTION AND RESULTS

As it has been mentioned before, the construction was stopped at an optimal spectral position, which is indicated in Fig. 2. By locating the wavelength of the resonance point with a higher attenuation it could be possible to detect pH by registering the wavelength as a function of pH.

In this sense, five PBS solutions were adjusted to pH 4.0, 4.5, 5.0, 5.5 and 6.0 as explained in section II.A. The experimental set-up was the same as that shown in Fig. 1. The

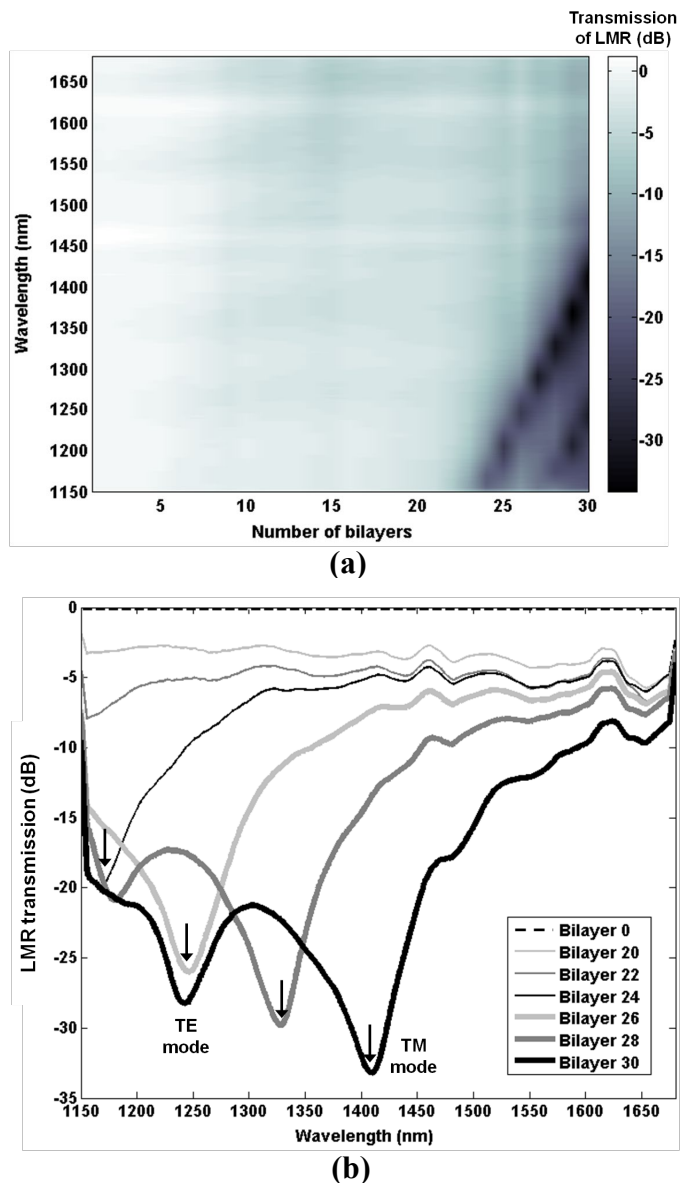


Fig. 2. Evolution of the LMR while the construction takes place in plan sight (a) and profile sight (b). To make these pictures, every spectrum was obtained after the immersion in the polyelectrolytic solutions, taking into account the first one as the reference of the measurement.

proof consisted in subjecting the sensor to several cycles between pH 6.0 and 4.0 in order to study its repeatability and response time.

Regarding the characterization of the LMR, the results obtained are graphically described in Fig. 3. Here, the different wavelengths at which the sensor stops after immersing in every pH solution are presented. At the beginning of the experiment, the sensor is immersed in the pH 6.0 PBS buffer solution, which causes a wavelength shift from 1410 to 1375 nm without any considerable movement in the transmission of the LMR, fixed at -34 dB. The explanation of this is that when the [PAH / PAA]<sub>30</sub> assembled structure reaches this pH value, both polymers are oppositely charged. However, while PAH chains are fully ionized, PAA ones are close to fully ionized, which provokes a strong adhesion between molecules. As a result, there is a compression in the polymeric structure due to

a deswelling process [28,30]. This induces a change in the thickness and the refractive index of the thin-film and, therefore, the resonance shifts to lower wavelengths.

In addition to this, a single peak is observed when detecting pH, instead of the dual peak observed at the end of the fabrication process. This is not a problem for the purpose of this study, since the surrounding medium outside the detector is different to air or water (PBS) and, due to the changes in the refractive index, the re-organization of the coupling modes makes TE and TM modes propagate together.

From this point on, the resonance experiments a progressive wavelength shift to the red as a function of pH. The explanation is that below pH 6.0, the polymeric matrix experiments a great swelling effect due to the quick loss of strong bonds between PAH and PAA [28,30]. As a result, the thin-film expands, reaching its highest values. This induces a change in the refractive index and, consequently, both a red-shift and an increase in the resonance depth. Concretely, when pH equals 5.5, the LMR locates around 1435 nm with almost -46 dB of transmittivity. A similar magnitude is obtained at pH 4.5, but the resonance is located at 1515 nm. When the LMR reaches 1560 nm with -50 dB of transmittivity, this is an indication of pH 4.0. Finally, for pH 5.0 a dual peak centered at 1470 nm can be observed in Fig. 3. This is caused by a lack of sensitivity of the OSA at this wavelength range. In this situation, the transmission of the LMR decays below the sensitivity limit of the OSA, so it is difficult for an electronic device to detect where the real peak of the resonance is. That is why, in the following lines, an algorithm to estimate accurately the resonance wavelength as a function of pH is proposed.

The spectra shown in Fig. 3 have been extracted from Fig. 4a, which is analyzed as follows. It represents the evolution of the different spectra as a function of time for every pH solution

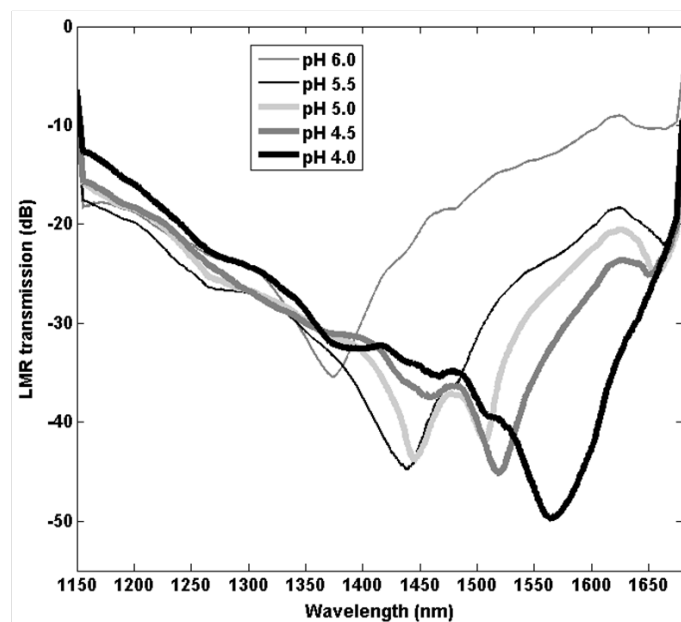


Fig. 3. pH detection for the sensor proposed. The resonance shifts to higher wavelengths when pH decreases from 6.0 to 4.0.

during the double detection cycle carried out with the PBS solutions. The repeatability of this sensor can be appreciated as the spectra draw the same figure for the same pH value. It is noticeable that when pH is close to 5.0 the spectra owns the dual peak commented before, so it is necessary to apply the algorithm to detect where the minimum of the resonance is. In this sense, Fig. 4b shows the result of applying the algorithm to every spectrum in Fig. 4a. Basically, its objective is to calculate the resonance wavelength of every spectrum. To this purpose, the vector of wavelength values with transmitted power below a threshold value is obtained. This is done for different threshold values. The central point of each vector is obtained and finally a weighted average is performed. Here, it is corroborated that the range of detection of the LMR varies between 1375 and 1560 nm; the wavelengths that correspond to the sensor responses at pHs 6.0 and 4.0 respectively in Fig. 3. Apart from that, there is an important wavelength shift for

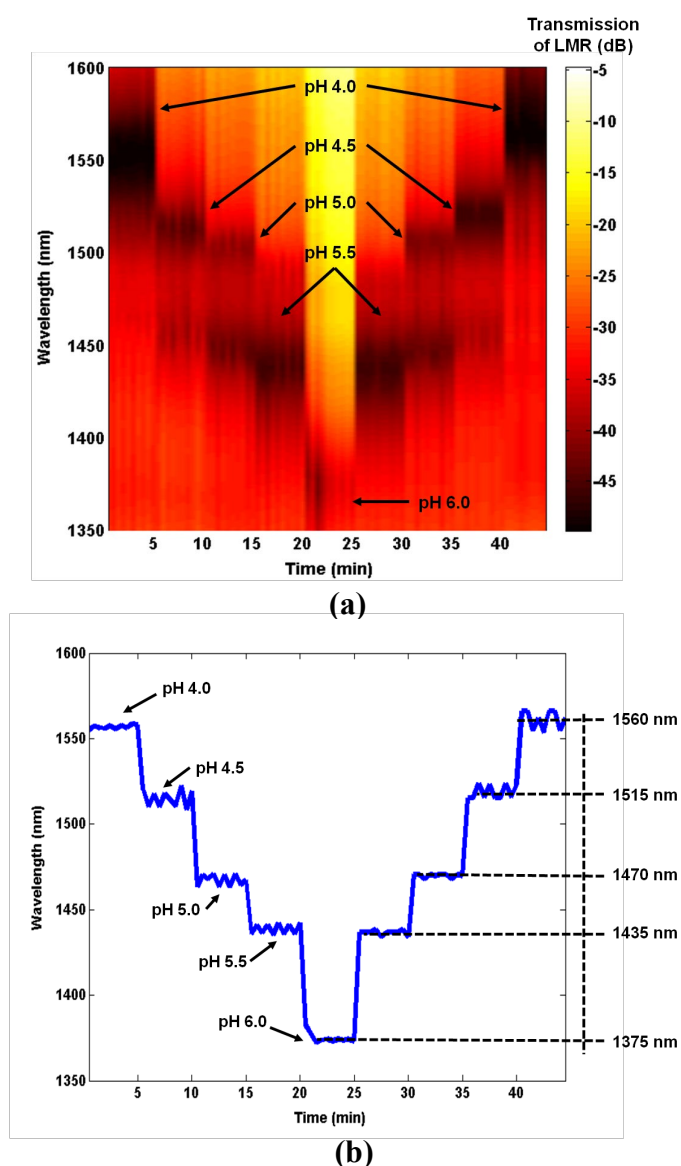


Fig. 4. Sensor repeatability for a 4-6-4 pH cycle. (a) Plan sight showing the shifts of the different spectra monitored during the experiment. (b) Tracking of the minimum of every spectrum after processing (a) with the algorithm proposed. There is a clear displacement in wavelength between pH solutions.

every pH value in this experiment. This leads to distinguish among the different pH values with enhanced resolution. To estimate this resolution value, the maximum noise provided when a monitoring of the resonance wavelength is done at a fixed pH value is obtained: 5 nm. By translating this noise to pH units, a resolution of 0.05 pH units is obtained.

Finally, the response time of the sensor when detecting pH is studied. Focusing on the second cycle of the detection shown in Fig. 4, the response time for every change of pH can be estimated as shown in Fig. 5. As an example, in Fig. 5 the response time to a variation from pH 6.0 to pH 5.5 is represented. Here, the horizontal axis is expressed in seconds to see the performance in a better way. As it can be observed, the response time is around 60 seconds. In this case, the [PAH / PAA]<sub>x</sub> polymeric matrix experiments a great swelling effect, which provokes a refractive index change that makes the resonance wavelength rise from 1375 to 1435 nm, as has been mentioned [28,30].

At this point it is interesting to notice that the results obtained in this section have been achieved by using a T-SMF, which dimensions correspond with those indicated in Fig. 1 and at the climatic conditions presented in section II.C. In the experiments performed in this work, the influence of the temperature has not been analyzed, though it is known that a [PAH / PAA]<sub>x</sub> polymeric matrix is sensitive to temperature variations, as studied in [32]. Apart from that, it is also known that the pH of the polymeric solutions and the materials used can be modified in order to change the optical properties of the thin-film [22,33]. Moreover, from a practical point of view, by reducing the T-SMF waist diameter once a short length has been fixed, the sensitivity can be improved [20-21]. This could be used for tuning the behaviour of the LMR towards the design of optimized pH T-SMF sensors.

#### IV. CONCLUSION

This contribution presents a novel pH sensor based on the high sensitivity given by the combination of a tapered SMF fiber and a lossy mode resonance phenomenon induced by a polymeric thin-film deposited onto it. The optimal range for

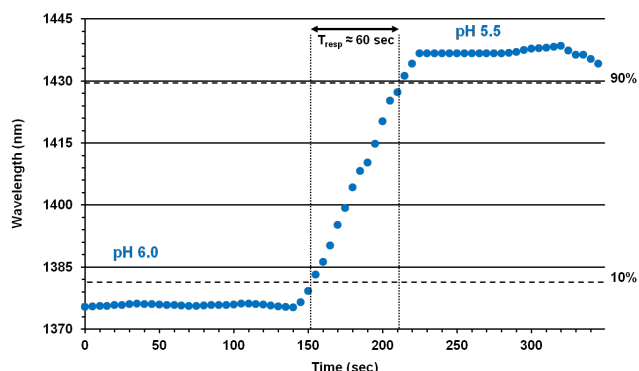


Fig. 5. Response time of the sensor. The time separation between samples is 5 seconds, which is the time needed by the OSA to acquire each piece of information.

this detector is located between pH 4.0 and 6.0 and the performance presents an estimated resolution of 0.05 units of pH. The response time reaches around 60 seconds in this pH range. Moreover, the stability of the measurements is adequate enough to consider it for the design of pH sensors based on this technology. What is more, these results can be tuned or enhanced by modifying sensor parameters both in the substrate and in the thin-film, so that the sensor is capable to detect higher pHs, (e.g. to detect blood pH in human beings), in order to diagnose acidosis or alkalosis states.

Finally, a wide variety of magnitudes may be detected by using this technique based on changes in the thickness of a polymeric matrix, such as immunosensors [34].

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