

## Article

# TiO<sub>2</sub>-Coated Fiber-Optic Sensor for Monitoring Ambient pH Fabricated Using the Sol–Gel Method

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## Abstract

Environmental pollution, particularly water contamination, requires the continuous development of robust, effective monitoring technologies. While chemical optical sensors offer significant advantages for environmental monitoring, many traditional devices rely on complex combinations of chemical dyes with specific acid dissociation constants. To overcome these limitations, this study presents a dye-free approach centered on a fiber-optic pH sensor based on a TiO<sub>2</sub> thin film. The protective coating of multimode optical fibers was successfully removed using a controlled hydrofluoric acid (HF) treatment, enabling deposition of TiO<sub>2</sub> films via a low-cost sol–gel dip-coating method. Structural characterizations through X-ray diffraction (XRD) and Raman spectroscopy confirmed the preferential growth and high purity of the anatase phase. Furthermore, energy-dispersive X-ray spectroscopy (EDS) confirmed the presence and relatively uniform distribution of Ti on the fiber surface. The optical performance of the sensor was evaluated using a 940 nm light source over a broad pH range (4–14), where the device exhibited distinguishable stepped optical-power responses at four pH conditions spanning pH 4–14. A preliminary analysis yielded an apparent average slope of 0.40 μW/pH. These results support the feasibility of using sol–gel derived TiO<sub>2</sub> coatings in the preliminary development of dye-free optical devices for aqueous pH monitoring.

**Keywords:** optical fiber pH sensor; TiO<sub>2</sub> thin films; sol–gel



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## 1. Introduction

Human activities are causing ecological imbalance. Environmental pollution, particularly water pollution, threatens human life by making clean water scarce [1]. Addressing water contamination and developing effective solutions is essential to restoring ecological balance and safeguarding human health. Pesticides, microorganisms, organic matter, oils, greases, and heavy metals are major aquifer contaminants [2,3]. To address environmental degradation, innovative equipment is being developed to mitigate and compensate for impacts. Major technologies, including mobile phones, transistors, integrated circuits, and fiber optics, have enabled achievements that were once just concepts [4]. Among these, chemical optical sensors (COSs) stand out for detecting and monitoring environmental

contaminants driving water pollution. COS use electromagnetic radiation (light) and its absorbance, fluorescence, or luminescence changes to measure the concentration of specific analytes [5].

COSs are based on the modification and propagation of light within the fiber, such as the wavelength transmitted through the core of this conductor [6,7]. Studies have explored their use in monitoring deformations in civil and aeronautical projects, detecting leaks in industrial processes, and in the hydrocarbon sector [8–10]. In environmental monitoring, COSs are also used to measure specific water quality parameters, including temperature, pH, and the presence of contaminants. Optical sensors offer advantages over conventional systems: immunity to electromagnetic interference, distributed sensing over long distances, and safe operation in harsh environments [11]. Advances have been made in chemical measurements of variables like temperature, dissolved oxygen, and pH, enhancing environmental monitoring [12]. These sensors are now being researched for in situ and real-time analysis [13,14].

The rise of fiber optics has accelerated information exchange. This technology supports devices that improve data quality and security. Fiber-optic sensors excel at transmitting data via light. Advances in photocatalytic thin-film electrodes, such as titanium dioxide (TiO<sub>2</sub>) and zinc oxide (ZnO), have driven research. Their use facilitates the degradation of complex molecules in both gas and liquid phases [15].

Both ZnO and TiO<sub>2</sub> have been widely used in these applications due to their optical, electronic, chemical, and structural properties, optimizing sensor performance. Both semiconductors exhibit high transmittance and low absorption in the visible spectrum, thereby improving the performance of optical sensors. With high refractive indices ( $n > 2$ ), they enhance light-matter interaction and sensitivity. TiO<sub>2</sub> is chemically inert in harsh environments and stable under UV irradiation, with a lower electron-hole recombination rate, making it ideal for this field [16,17].

TiO<sub>2</sub> is a white solid and exists in four crystalline phases: rutile (tetragonal), anatase (octahedral), brookite (orthorhombic), and high-pressure TiO<sub>2</sub> ( $\alpha$ -TiO<sub>2</sub>). All forms occur naturally as minerals [18,19]. The anatase phase has more porous nanostructures with a larger surface area. This allows greater interaction with analytes. It also has a lower electron-hole recombination rate than other phases [20,21].

This semiconductor has been synthesized by various routes, including hydrothermal, coprecipitation, microwave, and sol-gel. The sol-gel method offers several advantages. It is a simple and low-temperature synthesis method. It is also low-cost and yields high-purity materials [16,22].

This work presents the design principles of a fiber-optic sensor based on TiO<sub>2</sub> thin films for measuring various environmental control parameters related to water quality, such as pH.

## 2. Materials and Methods

### 2.1. Materials

Multimode optical fiber (THORLABS FG050LGA, 50  $\mu$ m core diameter, 0.21 numerical aperture, Newton, NJ, USA), hydrofluoric acid (HF, Sigma-Aldrich, St. Louis, MO, USA). ACS reagent, 48%), hydrochloric acid (HCl, Sigma-Aldrich ACS reagent, 37%), titanium tetrabutoxide (Ti(OC<sub>4</sub>H<sub>9</sub>)<sub>4</sub>, Sigma-Aldrich, 97%), deionized water (H<sub>2</sub>O, deionized), ethanol (C<sub>2</sub>H<sub>5</sub>OH), and SiO<sub>2</sub> substrates (VELAB No. 1).

### 2.2. Optical Fiber Treatment

The original protective coating on the optical fiber was stripped to expose the glass fiber core and facilitate the deposition of a TiO<sub>2</sub> film as a new functional layer. Each optical

fiber was cut to 20 cm, with 5 cm of the polymer coating removed from the center. This region was then chemically treated with hydrofluoric acid for a controlled duration to fully expose the underlying fiber core.

After the coating was removed, the fibers were thoroughly cleaned to eliminate any residual impurities or surface contaminants. To enhance film adhesion, the fibers were treated with acid to activate surface hydroxyl groups ( $-OH$ ), a process known as passivation. The fibers were then repeatedly rinsed with deionized water and dried at room temperature.

### 2.3. Synthesis of the $TiO_2$ Sol

The  $TiO_2$  sol was prepared using a sol–gel process adapted from [23]. We modified the catalyst quantities to adjust the hydrolysis rate. For this, 13 mL of titanium tetrabutoxide, 2.5 mL of DI  $H_2O$ , 55 mL of  $C_2H_5OH$ , and 1 mL of  $HCl$  were mixed. The solution was stirred continuously for 30 min at room temperature. Next, a solution containing 1.35 mL of deionized water and 10 mL of ethanol was added dropwise to the initial mixture under continuous magnetic stirring, initiating hydrolysis. This controlled procedure lasted approximately 20 min and yielded a homogeneous solution.

### 2.4. Thin Film Deposition

$TiO_2$  films were deposited by dip-coating onto both  $SiO_2$  substrates and pre-treated optical fibers, as outlined above [24]. The immersion and extraction rates were both set to 3 mm/s, and substrates remained in the  $TiO_2$  sol for 3 s during each cycle. After dip-coating, the samples were dried at 100 °C for 3 min in a tubular oven to evaporate solvents and firm the coating. This cycle was repeated until each sample had three layers.

Samples were then subjected to a final heat treatment at 550 °C for 1 h in an oxygen atmosphere to enhance the films' crystallinity and structural stability. To prevent the thermal degradation of the optical fiber's protective jacket, only the 5 cm bare central sensing region was positioned within the uniform hot zone of the tubular oven. The remaining polymer-coated ends of the 20 cm fiber extended outside the heating zone, keeping the polymer at a safe temperature throughout the annealing process.

### 2.5. Sensor Characterization

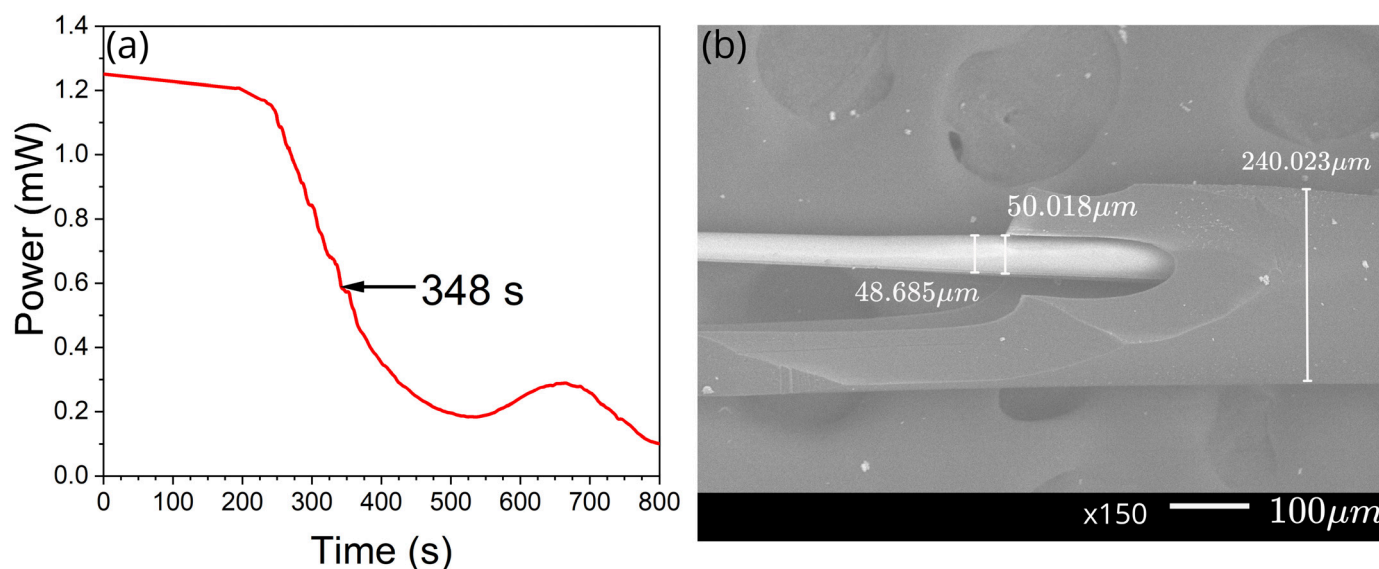
The sensor was evaluated using standardized aqueous buffer solutions at pH values of 4, 7, 10, and 14. To minimize macro-bending losses and maintain mechanical stability, the coated fiber was held in a straight configuration with fixed optical clamps. The sensing section was illuminated by coupling a THORLABS 940 nm LED light source (Newton, NJ, USA) into the optical fiber. Measurements were obtained by sequentially immersing the sensing region into individual containers, each containing a specific pH buffer. The sensor remained in each container for 3 min, during which the transmitted optical power was continuously monitored and recorded using a THORLABS PM320 optical power meter (Newton, NJ, USA). Between measurements, the sensor was transferred directly to the next buffer solution within approximately 10 s. No intermediate rinsing or drying step was performed. Separate containers were used for each pH solution; however, minor carryover between consecutive buffers cannot be completely excluded.

## 3. Results and Discussion

### 3.1. Fiber-Optic Treatment

Uniform coating removal is essential because residual coating can alter the phase shift of light propagating through the optical fiber. In this study, the hydrofluoric acid (HF) treatment time was determined to be 348 s. To establish this duration, sections of optical fiber were spliced, with one end connected to a light source (optical power: 6.5 mW;

wavelength: 940 nm) and the other to an optical measurement device and a computer for data acquisition and processing using Thorlabs PM320 software V1.0. Acid was applied to the uncoated portion of the optical fiber, and the decrease in optical power was monitored to determine the treatment time. Figure 1a presents the optical signal graph for the fiber immersed in HF. HF is highly aggressive toward silicate materials, unlike other piranha solutions or acids. During fiber-optic coating removal, fluoride ions ( $F^-$ ) displace hydroxide ions ( $OH^-$ ) from the silicate structure. HF induced corrosion depends on acid concentration, exposure time, temperature, and substrate thickness [25]. Figure 1b displays a micrograph of the fiber-optic cable after HF treatment, demonstrating effective coating removal and enabling estimation of the average core thickness.



**Figure 1.** (a) Optical signal of the fiber when immersed in HF. (b) Micrograph of the optical fiber with HF treatment.

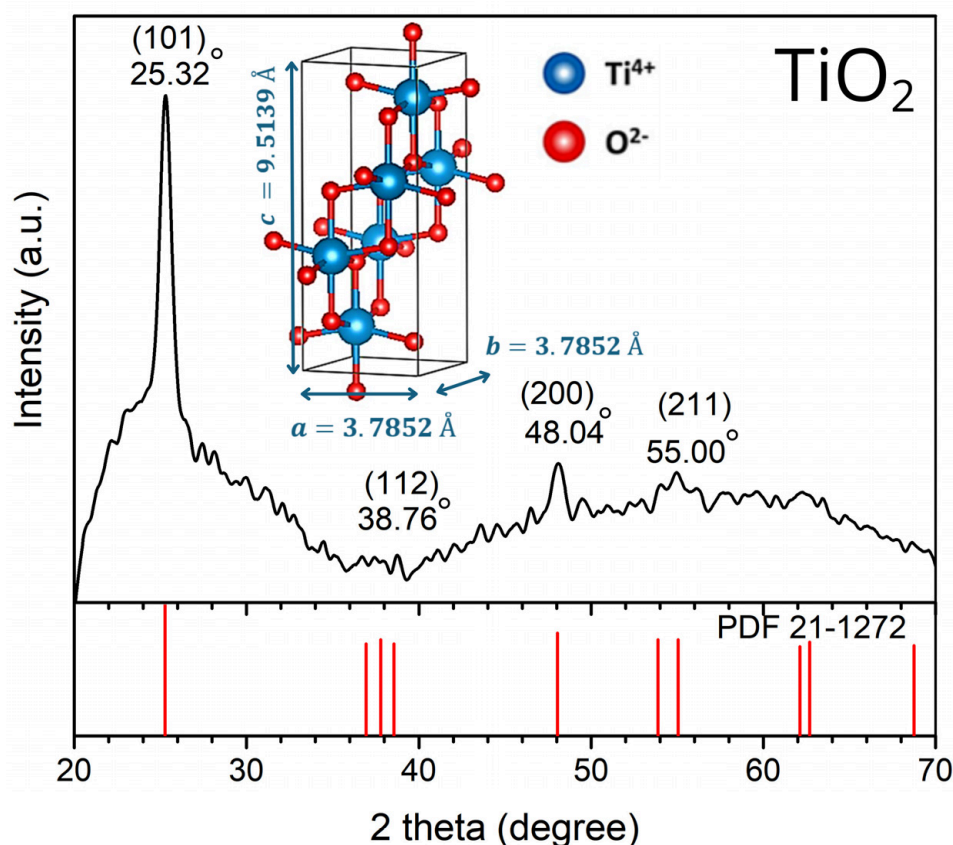
To facilitate more effective material characterization,  $TiO_2$  films were first deposited on Corning glass substrates before deposition onto optical fibers.

### 3.2. X-Ray Diffraction

To determine the structural properties, a Rigaku X-ray diffractometer (Tokyo Japan) ( $\lambda = 1.544 \text{ \AA}$ ) was used over a  $2\theta$  range of  $20^\circ$ – $70^\circ$ . The data were then compared with crystallographic card PDF 021-1272. As illustrated in Figure 2, the diffractogram of the film with three  $TiO_2$  layers shows peaks at  $2\theta$  values of  $25.32^\circ$ ,  $38.76^\circ$ ,  $48.04^\circ$ , and  $55.00^\circ$ , corresponding to the (101), (112), (200), and (211) planes, respectively, of the anatase phase [26]. Preferential crystal growth occurred in the (101) plane, which was used to estimate crystallite size using Scherrer's equation [27].

$$D = \frac{K\lambda}{\beta \cos \theta} \quad (1)$$

where  $D$  is the average crystallite size,  $K$  is the crystallite shape constant (0.9),  $\lambda$  is the wavelength of the incident X-ray radiation,  $\beta$  is the broadening of the diffraction peak at half its maximum height, and  $\theta$  is the Bragg angle.



**Figure 2.** XRD patterns of TiO<sub>2</sub> thin film.

The average crystallite size of the TiO<sub>2</sub> samples was 15.7 nm. Regarding the other characteristic planes of the anatase phase, (103), (004), (105), and (204), located around  $2\theta = 36.9^\circ$ ,  $37.8^\circ$ ,  $53.9^\circ$ , and  $62.3^\circ$ , respectively, they cannot be clearly identified due to the TiO<sub>2</sub> film thickness (279 nm), and these signals may overlap with those of the substrate used [28,29].

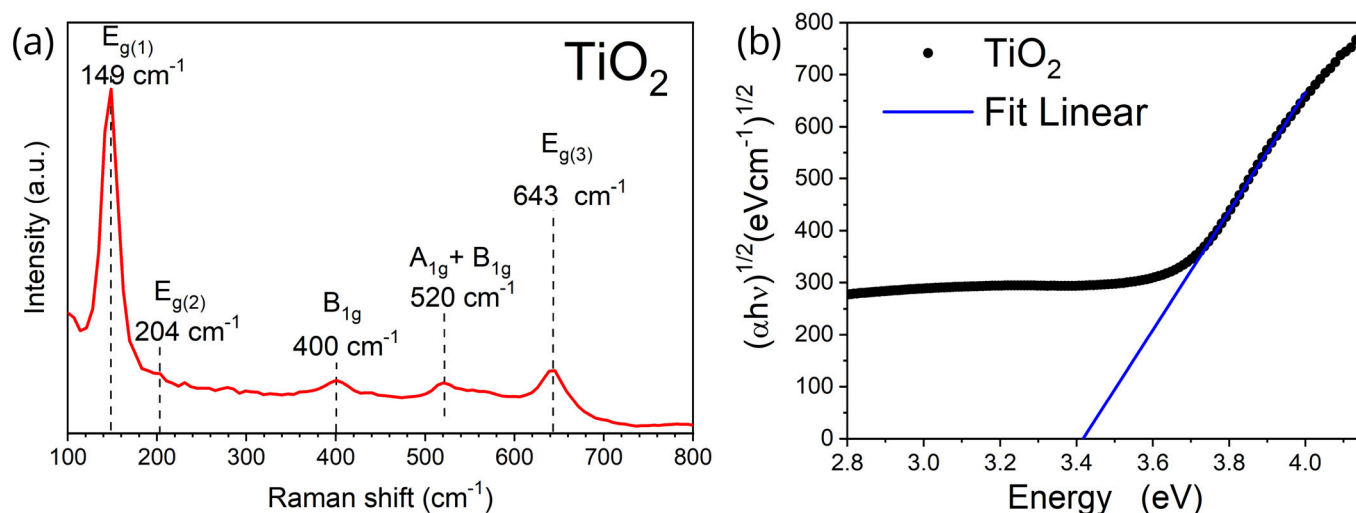
In the tetragonal crystal structure, the interplanar distance,  $d$ , the Miller indices ( $h$ ,  $k$ ,  $l$ ), and the lattice parameters,  $a$  and  $c$ , are related by the following equation [30]:

$$\frac{1}{d^2} = \frac{h^2 + k^2}{a^2} + \frac{l^2}{c^2} \quad (2)$$

The values found for  $a$  and  $c$  were 3.7852 Å and 9.51369 Å, respectively, which are in accordance with reports in the literature [31].

### 3.3. Raman Spectroscopy and UV-Vis Absorption

Raman spectroscopy confirmed the successful synthesis of the TiO<sub>2</sub> films. Measurements were conducted using an XploRA PUS HORIBA spectrophotometer (Kyoto, Japan) equipped with a 532 nm laser, scanning the 100 to 800 cm<sup>−1</sup> frequency region. The anatase phase of TiO<sub>2</sub> exhibits a tetragonal  $D_{4h}^{19}$  space group with six Raman-active modes:  $3E_g + 2B_{1g} + A_{1g}$  [32]. Figure 3 presents the Raman spectrum of the TiO<sub>2</sub> film. Vibrational bands at 149, 204, and 643 cm<sup>−1</sup> correspond to the  $E_g(1)$ ,  $E_g(2)$ , and  $E_g(3)$  modes, respectively. The 400 cm<sup>−1</sup> peak is attributed to the  $B_{1g}(1)$  mode, representing the symmetric bending vibration of the O-Ti-O bonds. The 515 cm<sup>−1</sup> peak is assigned to the  $A_{1g}+B_{1g}$  mode, where the  $A_{1g}$  mode corresponds to the antisymmetric bending vibration of the O-Ti-O bonds [33].



**Figure 3.** (a) Raman spectra and (b) Tauc plot of the TiO<sub>2</sub> thin.

These signals indicate a uniform composition within the TiO<sub>2</sub> thin film. The pronounced edge sharpness of Ti, compared to that of Si, demonstrates a well-defined interface between the TiO<sub>2</sub> thin film and the substrate [32].

The Tauc relationship was used to estimate the thin film's energy gap [34].

$$\alpha h\nu = A(h\nu - E_g)^n \quad (3)$$

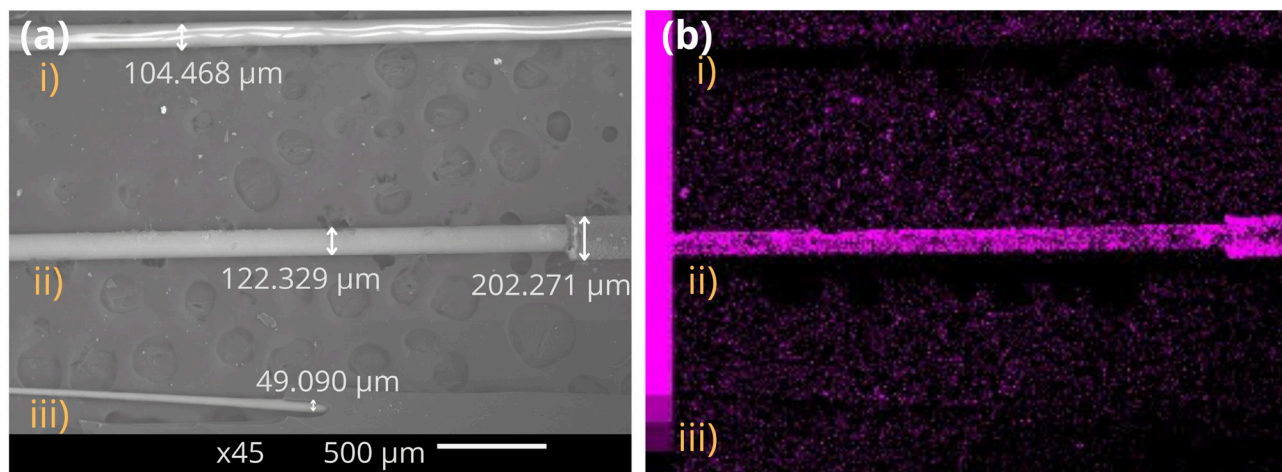
where  $\alpha$  denotes the absorption coefficient,  $A$  is a constant, and  $n$  equals 2 for indirect transition semiconductors [35]. The  $E_g$  value was determined by linear fitting ( $R^2 = 0.999$ ) of the  $(\alpha h\nu)^{1/2}$  versus  $(h\nu)$  plot (Figure 3b).

The estimated band-gap energy was 3.41 eV, slightly higher than the values commonly reported for anatase TiO<sub>2</sub> [36]. According to Igamov et al., this increase may be due to the formation of surface oxygen vacancies as new energy levels are introduced into the material [37].

### 3.4. Scanning Electron Microscopy and Energy-Dispersive X-Ray Spectroscopy (SEM/EDS)

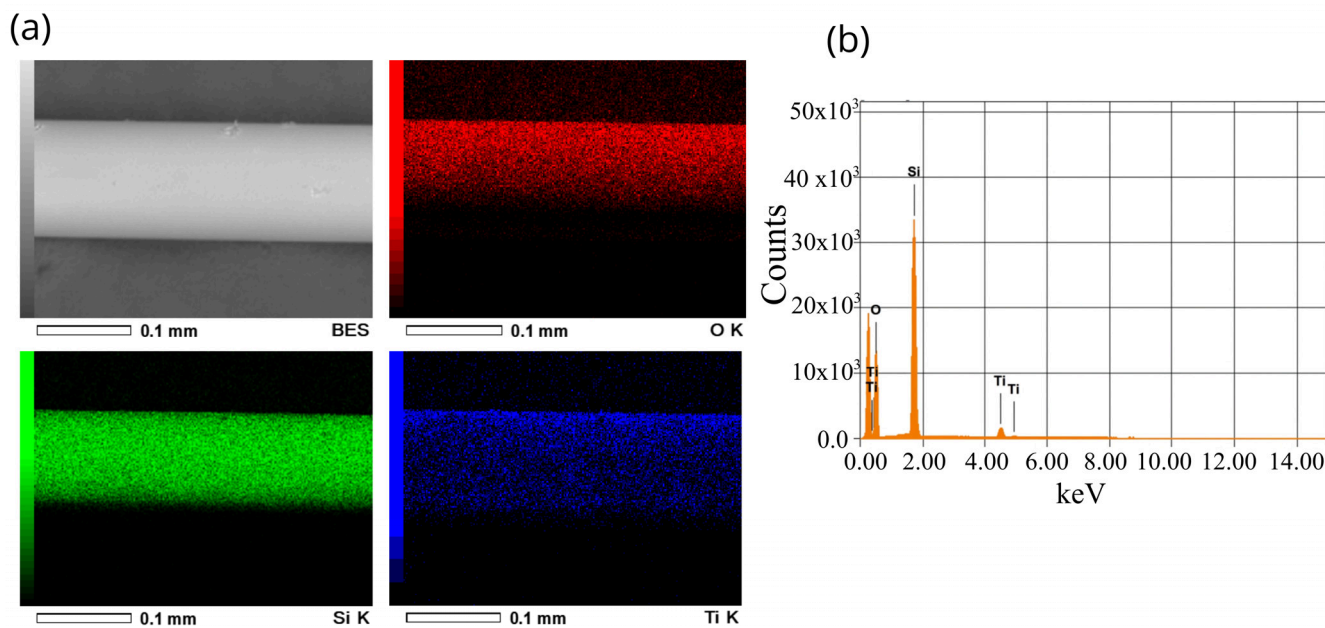
The micrographs corresponding to the pristine optical fiber, coated with TiO<sub>2</sub>, and treated with HF are shown in Figure 4a(i–iii), respectively. The pristine optical fiber (Thorlabs FG050LGA) has a nominal cladding diameter of 125 μm. The smaller apparent diameter observed in the micrograph (Figure 4a(i), 104.468 μm) is a recognized scanning electron microscopy (SEM) measurement artifact for highly cylindrical samples. In two-dimensional topographical projections, minor sample tilt and challenges in cursor placement at the equatorial plane frequently lead to measurement of a shorter geometric chord instead of the true maximum diameter [38]. After HF treatment, the diameter was reduced to 49.090 μm, a value consistent with the core diameter (50 μm) reported in its technical data sheet. This confirms the effectiveness of chemical etching in accessing the fiber core [39,40]. The un-etched cladding section of the fiber, as shown in Figure 4a(ii), maintained a diameter of approximately 122.3 μm. To confirm the presence and distribution of the semiconductor coating, Figure 4b presents the elemental titanium (Ti) mapping for each fiber state. The pristine fiber (Figure 4b(i)) and the strictly HF-etched fiber (Figure 4b(iii)) display no significant Ti signal. In contrast, Figure 4b(ii) shows a dense, continuous, and highly uniform Ti distribution exclusively along the coated section. This mapping demonstrates that the dip-coating process produced a morphologically uniform and homogeneous TiO<sub>2</sub> layer over the fiber [41]. The thickness measured on the simultaneously coated glass substrate was approximately 279 nm. Although the coating thickness on the curved fiber surface was

not measured directly, its thickness is expected to be in the nanometric range because the fiber and glass substrates were coated under identical deposition conditions.



**Figure 4.** (a) Micrographs of: (i) pristine optical fiber, (ii) optical fiber with a  $\text{TiO}_2$  film layer, and (iii) optical fiber with HF treatment. (b) Elemental Ti mapping of each of the analyzed optical fibers.

Figure 5a presents the micrograph and elemental mapping obtained by energy-dispersive X-ray spectroscopy (EDS) of the treated optical fiber. The elemental distribution maps confirm the presence of silicon (Si) and oxygen (O), which are characteristic of the fiber, as well as a uniform distribution of titanium (Ti) along the surface.



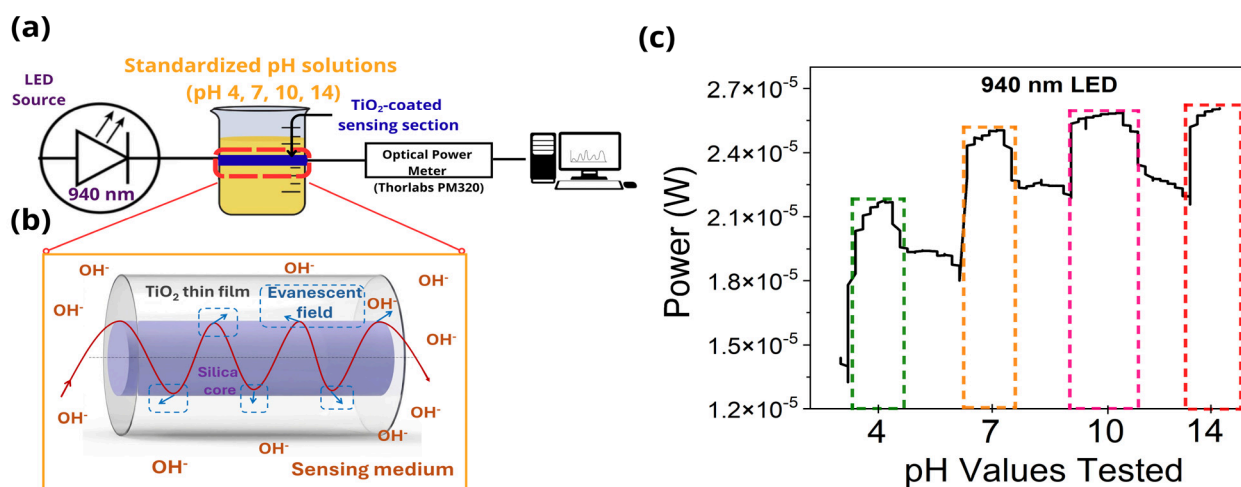
**Figure 5.** (a) Micrograph and elemental mapping, and (b) EDS spectrum of the optical fiber with a  $\text{TiO}_2$  film.

These results confirm the successful deposition of the  $\text{TiO}_2$  film obtained through sol-gel synthesis and subsequent heat treatment at 550  $^{\circ}\text{C}$  for one hour [32]. The EDS spectrum and its quantitative analysis (Figure 5b) further substantiate these findings, revealing mass compositions of 54.24% for O, 40.48% for Si, and 5.27% for Ti. In contrast to previous reports in which titanium or oxygen signals may be diminished due to passive films with thicknesses below the detection limit [42,43], the EDS spectrum in this study shows a

uniform distribution of titanium (Ti) across the fiber surface. The elemental concentrations in this sample strongly confirm the formation of the heterostructure.

### 3.5. Characterization of the Sensor for Measuring pH

Figure 6a presents the experimental setup used to characterize the optical fiber as a sensor. The device was evaluated according to the evanescent-field principle, in which light propagating through the fiber interacts with the  $\text{TiO}_2$ -modified segment. This configuration allows for the measurement of relative power drops associated with cladding losses (Figure 6b) [44,45]. The system operates as an intrinsic sensor, with its response determined by the optical power detected by the photodetector. Absorbance depends on the medium surrounding the fiber, and this effect is strongly influenced by the transmitted wavelength [6].



**Figure 6.** (a) Experimental setup for optical fiber sensor characterization; (b) a highly simplified schematic illustrating the light-matter interaction region, where lossy modes and evanescent fields interact with the  $\text{TiO}_2$  boundary; (c) characterization of a  $\text{TiO}_2$ -coated optical fiber as a pH sensor using 940 nm LEDs.

As illustrated in Figure 6c, characterizations were conducted using a 940 nm LED. The 940 nm wavelength (approximately 1.32 eV) is substantially lower than the 3.41 eV bandgap of the anatase  $\text{TiO}_2$  film. As a result, the sensor does not function through photocatalytic optical absorption. Instead, the near-infrared light acts solely as a non-destructive optical probe. This wavelength was chosen because longer wavelengths provide a greater evanescent penetration depth, thereby enhancing the sensitivity of the lossy modes to refractive-index variations at the interface [46]. Additionally, sub-bandgap illumination prevents the light source from inducing unintended photocatalytic reactions in the sensing medium, which enables stable and chemically unaltered pH monitoring.

Previous studies highlight the significance of semiconductor material thickness in determining coating properties. Izmirli and Cavdar demonstrate that reducing the  $\text{TiO}_2$  film thickness in the UV range to 670 nm enhances both the sensitivity and photovoltaic efficiency of photodiodes. In contrast, increasing the film thickness raises the series and sheet resistances and diminishes the material's antireflective properties [28].

Figure 6c shows the evolution of the optical power transmitted by the sensors when evaluated in standardized solutions. It is important to note that an overall attenuation in transmitted power was observed, stabilizing at approximately  $10^{-5}$  W. This decrease is mainly attributed to the physical coupling between the single-mode connectors and the multimode fiber sensing section. As reported by Jyoti et al., discrepancies in core size or refractive index generate significant light losses [14]. Similarly, Kumar et al., emphasizes

the need for optimal splicing to avoid attenuation [47], while Karthick notes that pores or microfractures in deposited films can also cause drops in optical power [48].

To minimize possible contributions from bulk refractive-index variations, standardized aqueous buffer solutions were used [49,50]. Nevertheless, because the refractive index of each specific buffer solution was not measured directly, possible contributions from differences in bulk refractive index or buffer composition cannot be completely excluded. A plausible interpretation of the observed response involves localized changes in the surface chemistry and effective optical properties of the TiO<sub>2</sub> coating.

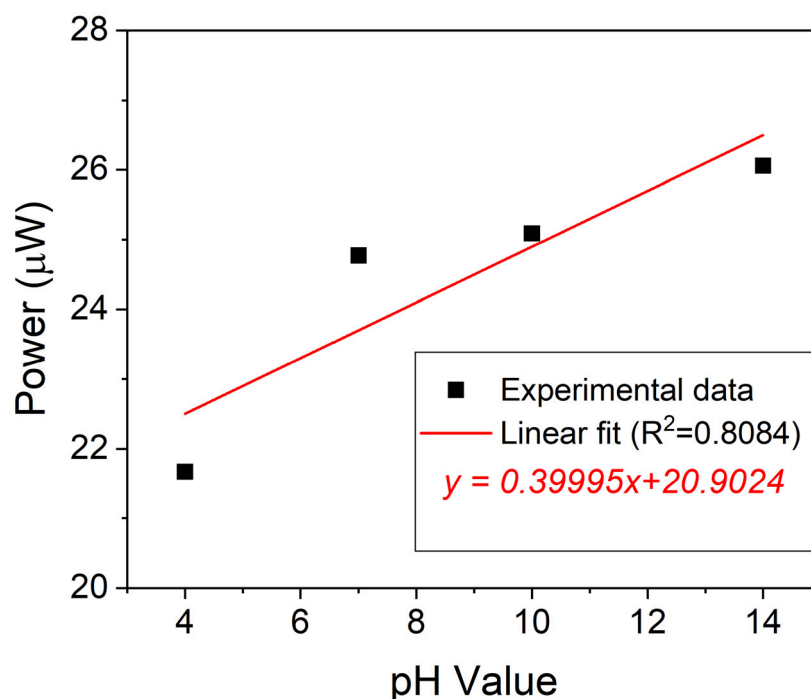
When evaluating the response to pH variations, a notable difference is observed at different pH levels; the sensor exhibits stepped behavior with clearly defined power jumps at pH transitions (4, 7, 10, and 14), showing greater sensitivity and a pronounced response. The physical mechanism responsible for these abrupt changes in transmitted optical power relies on the surface chemistry and the resulting refractive index modulation of the TiO<sub>2</sub> film. In aqueous environments, the surface charge of TiO<sub>2</sub> is controlled by the protonation and deprotonation of surface hydroxyl groups [51]. The ionization of these surface groups changes directly with the pH of the medium [52].

A bare silica core (SiO<sub>2</sub>) exhibits a point of zero charge near pH 2.0 to 3.0 [53]. Above its point of zero charge, the exposed silica surface is expected to be predominantly deprotonated. Nevertheless, because an HF-etched uncoated-fiber control was not evaluated, a contribution from the exposed silica surface cannot be excluded. In contrast, the isoelectric point of anatase TiO<sub>2</sub> is approximately pH 6.0 to 6.2 [51,52]. This property allows the TiO<sub>2</sub> surface to transition between different charge states.

Below its point of zero charge, the surface undergoes protonation in acidic media. It becomes positively charged, whereas in alkaline media, it undergoes deprotonation and becomes negatively charged [51,52]. These dynamic surface charge modifications alter the effective refractive index of the sensitive TiO<sub>2</sub> layer [54].

The deposition of a high-index anatase TiO<sub>2</sub> layer ( $n > 2.4$ ) onto a lower-index silica core ( $n \approx 1.45$ ) fundamentally alters the standard Total Internal Reflection (TIR) conditions. Under these circumstances, the optical behavior is consistent with a Lossy Mode Resonance (LMR) type or leaky-mode interaction [55]. The presence of a higher-index TiO<sub>2</sub> coating may promote coupling of optical power from the silica region into coating-associated lossy or leaky modes. Therefore, the observed behavior is consistent with an LMR-type or leaky-mode interaction, although confirmation would require wavelength-resolved measurements or electromagnetic modelling [56]. These guided coating modes are strongly confined near the semiconductor-liquid interface, making their attenuation highly sensitive to boundary conditions. As a result, modulation of the TiO<sub>2</sub> complex refractive index by pH directly influences the attenuation rate of these lossy modes, which may contribute to the observed optical-power variations and abrupt power transitions detected by the photodetector.

Throughout the 3-min continuous monitoring intervals at each pH level, the transmitted optical power exhibited a highly stable steady-state signal with minimal drift, indicating short-term signal stability during each individual exposure. To quantitatively assess performance, the steady-state optical power at each pH level was extracted from the dynamic response to construct a preliminary calibration plot (Figure 7). Based on the four evaluated pH conditions, the preliminary calibration plot exhibited an apparent average slope of 0.40  $\mu\text{W}/\text{pH}$ . The linear regression yielded an  $R^2$  value of 0.81, indicating moderate linearity. Because the analysis was based on four single measurements without replicate uncertainty, this value should be considered preliminary and does not establish sensor resolution or complete linearity over the entire pH range.



**Figure 7.** Preliminary relationship between steady-state transmitted optical power and the evaluated pH conditions (4, 7, 10, and 14). The linear fit yielded an apparent average slope of  $0.40 \mu\text{W}/\text{pH}$  and  $R^2 = 0.81$ .

Historically, among the large number of optical pH sensors, very few manage to cover a wide range (pH units) without depending on complex combinations of chemicals and dyes with different acid dissociation constants ( $pK_a$ ) [57,58]. In current technology, research focuses on the design of photodetectors that achieve superior performance and detectability metrics. In this regard, optical sensors incorporating inorganic materials such as  $\text{TiO}_2$  stand out for their large surface area, high porosity, and highly adaptable, stable structure [14].

Table 1 compares the present proof-of-concept configuration with selected recently reported miniaturized multimode-fiber pH probes. Several of these devices employ fluorescent dyes or hydrogel-based matrices and have been evaluated mainly within physiological or moderately acidic-to-alkaline pH ranges [59–61]. In contrast, the present device uses a dye-free, sol–gel-derived  $\text{TiO}_2$  coating and was evaluated at four representative conditions spanning pH 4–14. Based on these four single measurements, an apparent average slope of  $0.40 \mu\text{W}/\text{pH}$  was obtained. However, this value should be regarded as preliminary because replicate measurements, uncertainty estimates, and additional calibration points were not available. Therefore, further experiments are required to establish repeatability, stability, response time, reversibility, and performance in buffers with different compositions.

Finally, the optimization of sol–gel process parameters and the surface treatments applied to the optical fiber were key factors in achieving film deposition and successful sensitivity to different standardized pH concentrations.

The present work should be regarded as an initial proof-of-concept study. The optical response was evaluated at four representative pH values using a single increasing sequence. Therefore, the current dataset does not allow a complete assessment of repeatability, sensor-to-sensor reproducibility, reversibility, hysteresis, memory effects, resolution, long-term stability, or possible cross-sensitivity to buffer composition and bulk refractive index. Further validation should include an etched uncoated-fiber control, direct refractive-index measurements, different buffer compositions at the same pH, additional calibration points, replicate sensors, and repeated forward and reverse pH cycles.

**Table 1.** Quantitative comparison of performance parameters between the proposed TiO<sub>2</sub> coated optical fiber sensor and recently reported miniaturized multimode-fiber pH probes.

| Reference            | Sensing Mechanism and Wavelength                      | Sensor Geometry and Coating                          | pH Range | Sensitivity                             | Response Time                                       | Advantages/Remarks                                                             | Repeatability/Stability                                                 |
|----------------------|-------------------------------------------------------|------------------------------------------------------|----------|-----------------------------------------|-----------------------------------------------------|--------------------------------------------------------------------------------|-------------------------------------------------------------------------|
| This Work            | Evanescent Field Attenuation (940 nm)                 | HF-etched MMF/Sol-gel Anatase TiO <sub>2</sub>       | 4.0–14.0 | Apparent average slope: 0.40 $\mu$ W/pH | Not formally determined; signal monitored for 3 min | Dye free TiO <sub>2</sub> coating; four tested pH conditions spanning pH 4–14. | Not evaluated                                                           |
| Chauhan & Singh [54] | Evanescent Wave Absorption (633 nm)                   | Clad-modified MMF/PANI-ZnO nanocomposite             | 2.0–10.0 | 2.46 $\mu$ W/pH                         | 9 s                                                 | Good stability, requires complex polymeric nanocomposite synthesis.            | R: repeated pH 5/9 cycles<br>S: 30 min, pH 5–7–9                        |
| Zhang et al. [59]    | Fluorescence Emission (Ex: 488 nm, Em: 555.5 nm)      | Tapered~ MMF with ball lens/Epoxy + BCECF dye        | 6.0–8.0  | 1001.57 counts/pH                       | 0.5 s                                               | Assembly-free lens, high signal-to-noise ratio, narrow physiological range.    | R: 5 cycles (pH 6–8)<br>S: <3% signal fluctuation (1800 s)              |
| Zhou et al. [60]     | Ratiometric Fluorescence (Ex: 405/450 nm, Em: 515 nm) | MMF linked to hydrogel/PEGDA + HPTS microspheres     | 3.0–9.0  | 1.64 Ratio/pH                           | 1.97 min (acid-base)                                |                                                                                | R: repeated pH 7–8 cycling<br>S: <1% drift (1 h); stable after 2 weeks. |
| Min et al. [61]      | Optical Transmission Loss (Broadband visible)         | PDMS optical fiber/CMCS-PA@Fe hydrogel + Rhodamine B | 4.0–8.0  | High linearity ( $R^2 = 0.983$ )        | <30 s                                               | Dual function (monitoring and photothermal therapy), in-vivo wound healing.    | R: 5 cycles<br>S: good (values not reported)                            |

R: Reuse; S: Sensitivity.

## 4. Conclusions

A TiO<sub>2</sub>-coated, HF-etched optical fiber was fabricated and evaluated as an initial proof-of-concept pH-responsive device. XRD and Raman spectroscopy confirmed the formation of anatase TiO<sub>2</sub>, while SEM/EDS analysis verified the presence and relatively uniform distribution of Ti on the fiber surface. The thickness measured on the simultaneously coated glass substrate was approximately 279 nm; however, the coating thickness on the fiber was not measured directly.

The device exhibited distinguishable steady-state optical-power levels at pH 4, 7, 10, and 14 under 940 nm illumination. A preliminary analysis based on these four conditions produced an apparent average slope of 0.40  $\mu$ W/pH and moderate linearity. Further experiments involving control fibers, denser calibration points, repeated measurements, reverse pH cycles, direct refractive-index measurements, and independent sensor specimens are required before repeatability, reproducibility, reversibility, resolution, and long-term stability can be established.

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