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ZnO nanostructure-enhanced tapered polymer optical fiber sensors for pH detection

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This study presents an experimental investigation of tapered polymer optical fiber (POF) sensors enhanced with zinc oxide (ZnO) nanostructures for improved pH detection. Both ZnO-coated and uncoated POFs were fabricated and characterized to evaluate the effects of surface modification and probe length on sensing performance. The sensors were exposed to pH solutions ranging from 2 to 8, and their optical responses were assessed through variations in refractive index, output power, and voltage. Results indicate that ZnO-coated POFs exhibit enhanced sensitivity compared to uncoated fibers, with the 4 cm coated sensor achieving the highest sensitivity of 0.0464 V/pH and a linearity of 98.30%. The ZnO coating improved the evanescent field interaction by altering the refractive index profile, resulting in greater responsiveness to pH-induced optical changes. These findings highlight the potential of ZnO-coated tapered POFs for high-performance, low-cost pH sensing in biomedical and environmental applications.

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1. Introduction

Optical fiber sensing has been entirely revolutionized by fiber optic technology, and its benefits rapidly extended to a number of distinct industries, including environmental, chemical, and medical monitoring [1-6]. Since optical sensors offer great sensitivity, precision, and immunity to electromagnetic interference, they have emerged as a potential technique to quantify a variety of physical and chemical properties [7-9]. Current research has focused on developing polymer optical fiber (POF) sensors that can accurately and sensitively measure various parameters such as temperature, humidity, and pH [10-13].

By adding sensitive materials to the fiber's core, POF sensors' sensitivity can be enhanced further since the power of the evanescent wave inside the cladding will increase [14-16]. The effects of coating the POF with materials such as metal oxides, polymers, and nanoparticles have been the subject of numerous studies recently. Because of their distinct optical and electrical properties, zinc oxide (ZnO) coated POF sensors are a viable option. ZnO is a wide-bandgap semiconductor with a high refractive index and transparency in the visible region. ZnO coating improves sensor sensitivity by increasing the interaction between the evanescent wave and the detecting medium [17-19]. The POF sensor's performance can also be improved by tapering it to a specific length or diameter [20-23]. For example, the fiber's waist diameter must be decreased to allow a large portion of the evanescent field to pass through. As a result, research into the impact of POF structures on sensor performance is crucial for sensor development, and it is a focus of this study.

In order to ascertain the impact of coating the fiber core with zinc oxide on the sensitivity and linearity of the pH sensor, this work also presents a spectral investigation of zinc oxide-coated POF sensor. The study additionally examines how the fibers' optical properties and power output alter after being immersed in different pH concentration solutions. The findings of this research can contribute to the advancement of optical sensor technology and offer potential applications in various industries.

2. Method

2.1. Experimental setup

The schematic representation of the sensor system, which includes a red LED, POF sensor, receiver circuit, and multimeter is incorporated in Fig. 1. The receiver circuit's key components are photodetectors and optical amplifiers, while the red LED will serve as a light source with a wavelength of 650 nm. The photodetector, also known as an optical to electrical (O/E) converter, is a component in the front end of an optical receiver that transforms the sensor's incoming optical signal into an electrical signal, whereas the optical amplifier is configured as a trans-impedance amplifier. The LT1884 Op-Amp was used to retain a constant voltage across the IFD91B photodiode, preventing capacitance from being charged or discharged and thereby accelerating the response. During the experiment, the POF needed to be dipped into a solution that had been prepared in a petri dish for one minute before the data being recorded in Excel using the data streamer. The recorded data was the refractive index value of each individual concentration measured with a digital refractometer, and the output voltage was evaluated with a multimeter and recorded via microcontroller. The working principle of pH value based on plastic optical fibers utilizes changes in light intensity due to changes in power losses in optical fibers. Changes in pH levels result from the changes in the refractive index around the sensor.

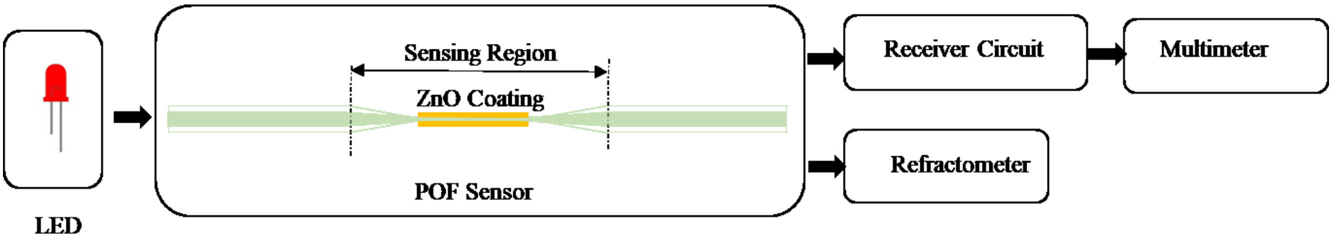


Fig. 1. Experimental setup of the sensor system (colour online)

2.2. pH solution and fiber probe preparation

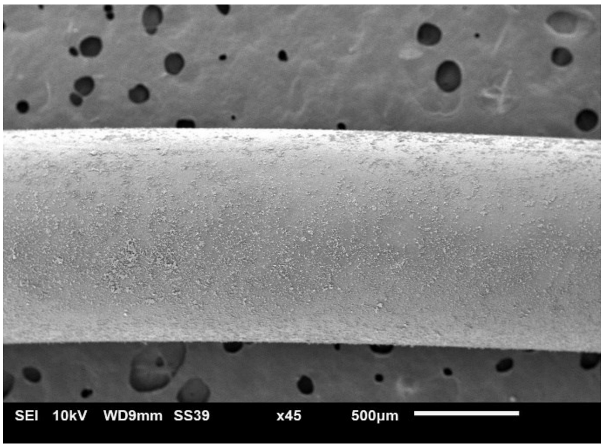
The pH solution was controlled by increasing and reducing the pH level from 2 to 8 by one increment each. The initial solution was mixed with ammonium to raise the pH of the solution, while hydrochloric acid was added to decrease it. The refractive index of each pH value was then determined using a refractometer. To prepare the POF probe as the sensing region, the POF jackets were stripped using a knife to expose the fiber. The exposed length of the fiber was fixed at 2 cm, 3 cm, and 4 cm. To increase the POF's sensitivity, each fiber will be tapered to 0.6 mm. The tapering step involves scrubbing the POF with sandpaper with a roughness of 1000 grit. This approach has the disadvantage of demanding careful handling because the brittleness of the fiber increases with decreasing fiber diameter.

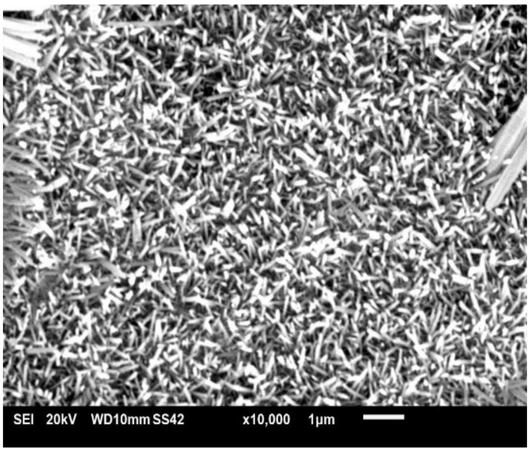
2.3. ZnO deposition by hydrothermal method

After the fiber has been prepared to the necessary exposed length and diameter, ZnO was deposited to cover the fiber's core using a hydrothermal seeding procedure. The seeding procedure includes preparing the seeding solution, treating the core of the POF, producing the nucleation centre on the POF, and annealing. First, for a good uniformity of ZnO growth, the tapered fiber was treated with 50 ml polysorbate 80 mixed with 500 ml deionized water. The POF was submerged for 10 minutes and dry for two hours. Simultaneously, during the core treatment, the seeding solution was prepped by combining two solutions made up of Zinc Acetate and Sodium Hydroxide. For the first solution, 0.088 g of Zinc Acetate Dihydrate was dissolved in 80 ml of ethanol for 30 minutes at 50 °C to produce 5 mM solution.

To cool the solution down, an additional 80 ml of ethanol was added. The second pH control solution was made by dissolving 0.016 g of sodium hydroxide in 80 ml of ethanol for 30 minutes at 50 °C. The mixture was stirred and yields a 5 mM solution. Finally, pH control solutions were gradually added into the zinc oxide nanoparticle seeding solutions with a 1ml pipette per minute.

The mixed solution was then placed in a water bath at 60 °C for 3 hours, and the hue of the solution changes from clear to clouded. The final step was to dip and dry the fiber to form a nucleation site on the POF. All POF samples were submerged in the seeding solution for 1 minute before being dried. This technique was repeated ten times, and the POF was annealed in an oven at 70 °C for three hours. The final phase was the growth process, which started with dissolving 2.97 g of Zinc Nitrate Hexahydrate and 1.40g of Hexamethylenetetramine in 1000ml of deionized water to generate a 10 mM solution. All of the seeded POF were vertically submerged in the synthesis solution and heated in the oven at 90 °C for 10 hours. However, after 5 hours, the solution was replaced with a new solution in order to retain the growth process conditions. After synthesis, the POF was repeatedly rinsed with DI water. Figs. 2(a) and 2(b) depict the top and magnified views of SEM images of vertically growth ZnO nanoparticles on POF, respectively.





(a) (b)

Fig. 2. SEM image of vertically oriented ZnO NPS deposited POF for (a) top view and (b) magnified SEM view of coated sensing region

3. Results and discussion

This section will discuss the performance of the proposed sensor in terms of pH analysis, output power, output voltage and sensitivity.

3.1. pH analysis

To evaluate the influence of pH fluctuations on refractive index (RI), a refractive index of pH solutions ranging from 2 to 8 was investigated. Reference measurements were realized with a conventional refractometer. The results show that as the pH of the solution increases, the RI gradually climbs from 1.3329 to 1.3355, as presented in Fig. 3. It is clear that pH has a significant influence on the refractive index, resulting in a wide range of refractive index values under varied pH conditions. The pH concentration-refractive index relationship will be adopted as a reference for sensor characterization, specifically output power and voltage.

This changes in the refractive index that occur cause changes in optical power which are detected by optical detectors, which will be amplified by the differential amplifier and converted from analog into a digital signal in output voltage form that is read on the computer. The measurement results of the pH sensor based on POF without ZnO coating for 2 cm is shown in Fig. 3.

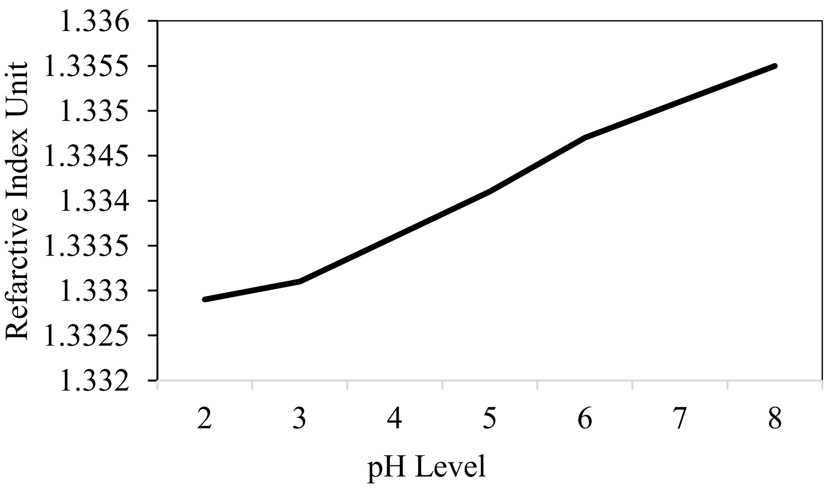


Fig. 3. Refractive index reading vs pH value

3.2. Output power

Prior to the pH refractive index test, the fabricated sensor was immersed in the pH solution to assess its performance for pH sensing. Measurements of power output were conducted on various lengths of polymer optical fiber (POF), encompassing both non-coated and zinc oxide-coated types, using an optical power meter. Fig. 4 depicts the power output of pH solutions ranging from 2 to 8 for three distinct probe lengths: 2 cm, 3 cm, and 4 cm. The solid line represents the uncoated POF sensor, whereas the dotted line indicates the ZnO POF sensor. The results revealed significant power reductions across all samples as the pH value increases. Specifically, the non-coated 2 cm POF experienced a decrease from a power level of -41.50 dBm to -44.79 dBm, while the ZnO-coated POF exhibited a reduction from -41.91 dBm to -45.64 dBm. Furthermore, the trend of decreasing power remained as the length of the fiber probe increased. A 3cm zinc oxide coated POF, for example, reduced from -47.55 dBm to -50.21 dBm, whereas a 4cm zinc oxide coated POF decreased from -48.17 dBm to -53.32 dBm. These results show an apparent correlation between pH values and recorded declines in output power, with longer fiber lengths experiencing greater losses. Moreover, a comparison of non-coated and zinc oxide coated POFs reveals the latter has a larger power loss, demonstrating that the coating material influences the sensor's performance.

As can be observed from Fig. 3, as pH levels increase, the refractive indices of the solutions differ. This will induce a mismatch between the refractive indices of the POF core and the cladding. This mismatch raises scattering and absorption losses, contributing to the observed power decrease. Furthermore, as the length of the probe increases, the cumulative effect of these losses becomes more evident, thereby leading the observed decrement in optical power transmission. According to theoretical principles, the addition of ZnO modifies can also modify the refractive index profile of the POF sensor, resulting in changes in measured optical power as the pH value varies [24].

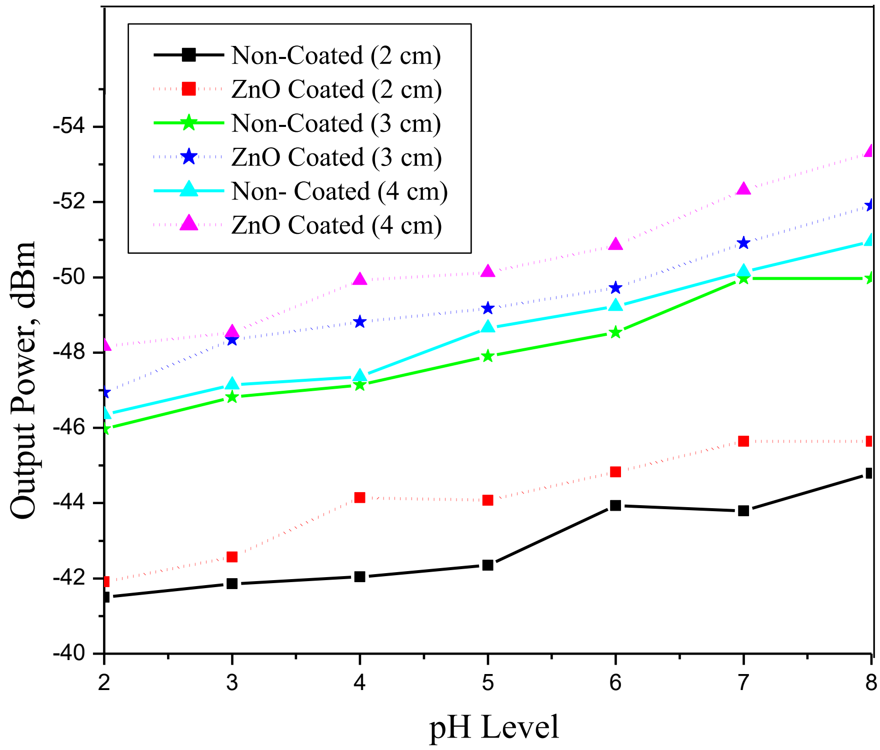


Fig. 4. Output power reading vs pH value (colour online)

3.3. Output voltage

The electrical characterization phase involved measuring the voltage output of the sensor receiver circuit. The graph in Fig. 5 shows the output voltage of the pH level sensor using tapered POF with and without ZnO coating. Non-coated sensors for tapered ranging from 2 cm to 4 cm are represented by solid line, while the dotted line represent the ZnO coated sensors. Each sensor exhibits distinct responses, indicating a decrease in output voltage as pH levels increase. It can be seen that increasing pH's level will cause changes in the refractive index and the intensity of light along POF. Increase in refractive index on optical fiber sensors results in greater power losses in optical fiber, and the output voltage becomes smaller. For instance, the output voltage of a 2 cm non-coated POF at pH 2 is 1.05 V, dropping to 0.81 V at pH 8, showing a significant decrease with increasing pH. This trend holds for various probe lengths and both non-coated and ZnO-coated sensors, with ZnO coating resulting in lower observed voltages due to increased refractive index changes.

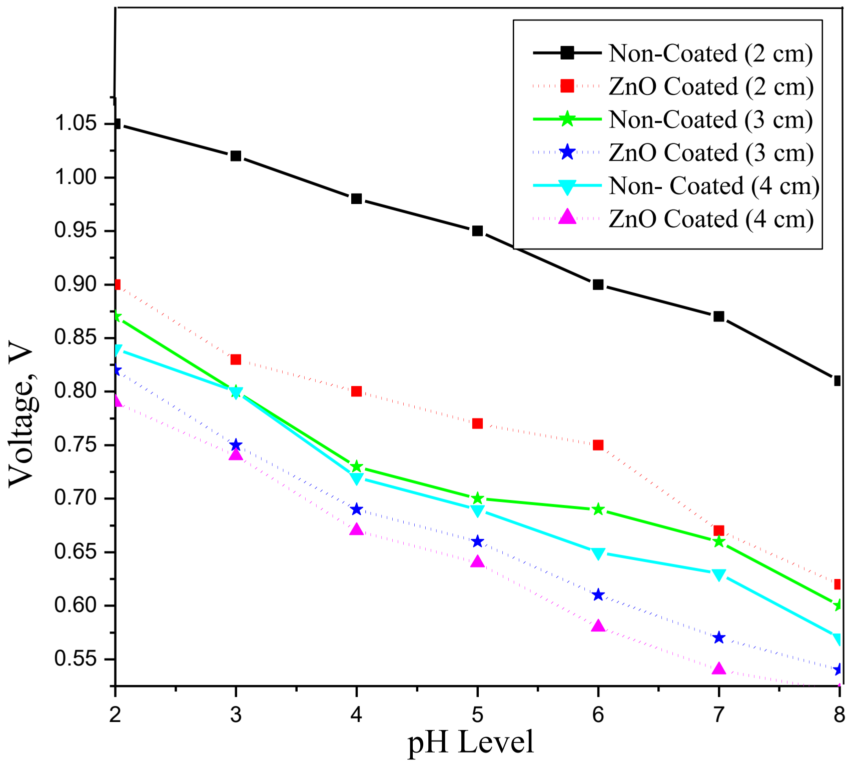


Fig. 5. Output voltage reading vs pH value (colour online)

3.4. Sensitivity output power

Sensitivity is a measure that states the relationship between the change in the output sensor and the change in the input of the sensor. In this project, the sensitivity of the sensor was obtained by calculating the slope of the curve [25]. Fig. 6 depicts the sensitivity graph illustrating the performance of the pH sensor. Notably, both the uncoated and ZnO-coated POF exhibit enhanced sensitivity with increased exposure length. The 4 cm ZnO-coated POF demonstrates the highest sensitivity at 0.0464 V/pH with a linearity of 98.30%. This phenomenon arises from the attenuation of light propagation subsequent to the application of POF coating, thereby facilitating enhanced interaction between the incident beam and the sensing medium. As a result, small changes in the surrounding medium induce corresponding fluctuations in transmitted optical power due to evanescent field interactions.

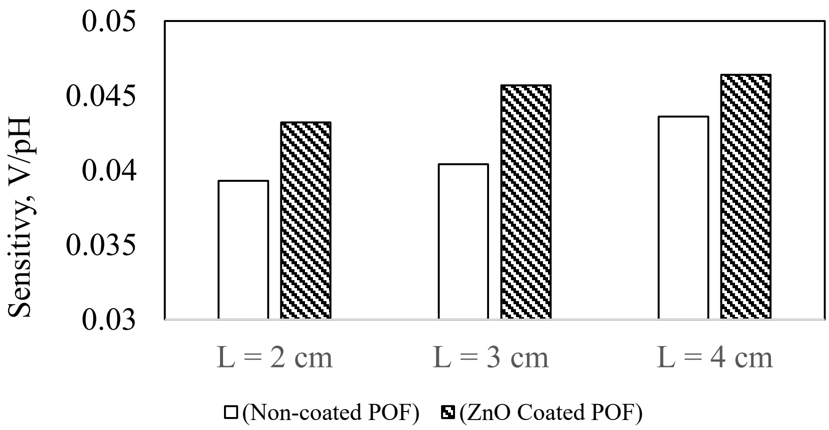


Fig. 6. Sensitivity of coated and non-coated POF sensor

4. Conclusion

In conclusion, the utilisation of zinc oxide-coated polymer optical fibres has resulted in significant advances in pH sensing technologies. Through a comparative analysis of coated and uncoated sensors, it was observed that the zinc oxide coating enhances the sensitivity of pH measurements. The study revealed that the coated sensors exhibit greater sensitivity to changes in pH values compared to their uncoated counterparts. Among all configurations, the 4 cm ZnO-coated POF achieved the highest sensitivity of 0.0464 V/pH with a linearity of 98.30%, outperforming uncoated sensors. The sensor sensitivity of the 4 cm ZnO-coated POF was found to increase by more than 10% compared to the uncoated POF. Additionally, the refractive index gradually increases with rising pH levels, showcasing the effectiveness of the sensor system in responding to pH variations within the tested range. These findings provide valuable insights into the performance of zinc oxide-coated fiber optics for pH sensing applications, offering promising prospects for advancements in optical sensor technology across diverse industries such as medical and chemical fields. The study sets a foundation for further research and development in the field of optical pH sensing, highlighting the potential for continued innovation and application of zinc oxide-coated fiber optics in real-world scenarios.

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