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Room Temperature Hydrogen Sensing Based on Tapered Optical Fiber Coated with Polyaniline (PANI) †

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Abstract: This work demonstrates a hydrogen (H_2) sensor at room temperature made of tapered optical fibers coated with a polyaniline (PANI) nanofiber. A transducing platform was constructed using a multimode optical fiber (MMF) with a 125 μm cladding and a 62.5 μm core diameter. To enhance the light evanescent field surrounding the fiber, the fibers were tapered from 125 μm in diameter to 20 μm in diameter with 10 mm waist and coated PANI using the drop casting technique. Various characterization techniques, such as field emission scanning electron microscopy (FESEM), energy dispersive X-ray (EDX), differential X-ray (XRD), and atomic force microscopy, have been used to establish the PANI's properties. When H_2 is subtracted, the optical properties of the PANI layer change, resulting in a change in light absorption. The fabricated sensor was tested by exposing it to H_2 at different concentration from 0.125% to 1%. In this case, the sensitivity , response and recovery times were 15.928/vol%, 110 s and 160 s, respectively. The improved hydrogen sensor holds great promise for environmental and industrial applications due to its ability to operate at room temperature.

Keywords: Hydrogen (H_2), Tapered optical fiber, Polyaniline (PANI), Drop casting technique.

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1. Introduction

Hydrogen (H_2), due to its high fuel efficiency, abundance, uncontaminated character and sustainability, is one of the possible solutions to the impending energy crisis [1]. It is also a reliable gas for misdiagnosis in power transformers [2,3]. H_2 has also been used in other sectors, such as aerospace engineering, mineral refineries, oil exploration, chemical processing, cryogenic refrigeration, and many more [4]. However, the high diffusion coefficient (0.16 cm2 / s in air), low ignition energy (0.018 mJ), wide explosion concentration range (4% -75%), and high heat of combustion (285.8 kJ / mol) convert them to gases. explosive and potentially dangerous for use, transport and storage [5].

On the other hand, optical sensors, rely on optical fibers, which have unique characteristics, such as lightness, small size, electromagnetic interference resistance, instability, and stiffness in harsh environments [6]. Due to their peculiar properties, optical fibers are perfect candidates for detection in harsh environments [7].

Recently, the scientific community has made significant progress on the synthesis and application of conducting polymers [8–10]. One of the promising materials for gas sensing is polyaniline (PANI), which among polymers has the peak environmental stability. It is currently known as the only conducting polymer that is stable in air [11]. As a sensor, PANI is particularly helpful because it is sensitive at room temperature [12,13] and it can be applied to detect a range of gases in combination by additional nanomaterials [14].

Several optical hydrogen sensors using PANI as an energy transformer have been reported in recent years. Most of them rely on fiber gratings (FBGs) [15] and plastic optical fiber [16]. To make fiber optic sensors sensitive to their surroundings, most of them must be functionalization [17]. In this research, PANI coated with dissolved, tapered optical fibers is used to detect hydrogen gas.

2. Experiments

2.1. Tapered Optical Fiber Fabrication

The H_2 gas sensor was developed using a multimodal tapered fiber optic (MMF) with cladding and core dimensions of 125 m and 62.5 m, respectively, as a transducing platform. For reduction, the Vytran glass processing system (Vytran GPX-3400) was employed. The machine operates on a heating and pulling principle, with a graphite filament acting as a heating source to create the desired tapered profile geometry. The MMF had a 125 mm cladding diameter that tapered to a 20 mm waist diameter, a 10 mm waist length, and a 5 mm tapering top. The picture of the tapered optical fiber created with the tapered area is shown in Figure 1.

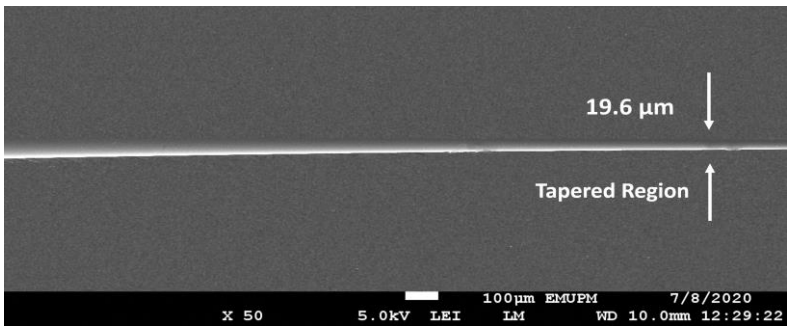

Figure 1. Scanning electron microscopy (SEM) micrograph of a tapered multimode optical fiber's transition area (MMF).

2.2. PANI Functionalization on Tapered Optical Fiber

Aniline was purified by distillation before polymerization. The purified aniline (0.16 M) was instantly dissolved in pre-prepared 0.05 M perchloric acid (HClO4) [18,19] (Merck, 70-72%) to avoid atmospheric oxidation. In another volumetric flask, 0.16 M of ammonium peroxodisulfate was also dissolved in HClO4, and both mixtures were rested overnight. Ammonium peroxodisulfate solution was carefully added to the aniline solution with continuous stirring at room condition. The oxidative polymerization reaction of the mixture was left stirring for 24 hours. The obtained PANI (dark green precipitate) was filtered and washed with ethanol (EtOH) until colourless supernatant liquid can be observed in order to minimise the amount of unreacted monomers and oligomers [20]. It is then dried at 60°C until a constant weight is obtained. A drop casting technique was used to coat the tapered optical fibers. To guarantee full evaporation of the aqueous medium, a drop of mixture (about 20 l) was put into the base of the tapered optical fiber using a micropipette, and the sample was heated in the oven at 80 oC for 40 minutes.

A light source (tungsten halogen, HL-2000, Ocean Optics, Dunedin, FL USA) with a coverage wavelength of 360 nm to 2500 nm, and a spectrophotometer (USB 4000, Ocean Optics USA) make up the experimental phase of the gas optical sensing system. It can detect anything between 200 and 1100 times per second. The optical gas detection system's experimental setup includes a customized gas chamber for measuring the optical absorption spectrum. The PANI coated sensor was placed in a close gas unit and purged with a centrifuge at a gas flow rate of 200 sccm from a computer managed mass flow controller. Figure 2 depicts the H_2 sensor's experimental setup.

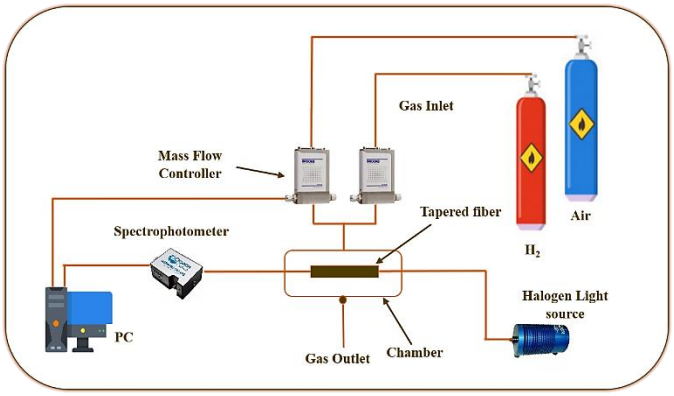

Figure 2. H_2 sensor experimental setup.

2.3. Materials Characterization

FESEM (JSM-7600F) was used to examine the films' morphology, while EDX analysis was used to assess their original composition. XRD examination indicated material identification, crystallization, and the PANI phase transition (APD 2000). FESEM images of PANI nanoparticles are shown in Figure 3. The PANI is mostly made up of uneven grains and chips with sharp edges, as can be observed. Furthermore, the structure seems to be totally porous, creating very small polyaniline particles that can expand the liquid-solid interfacial [21,22].

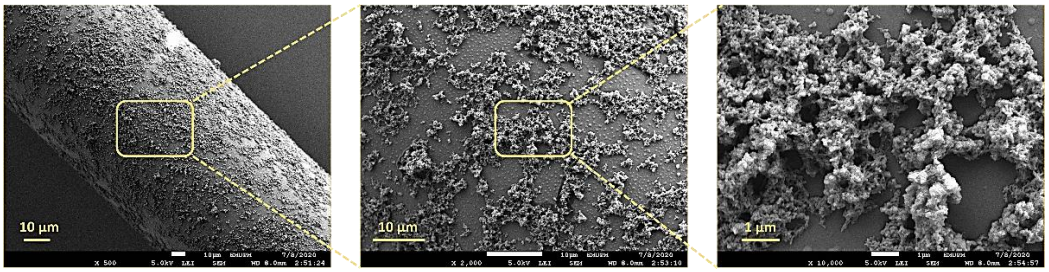

Figure 3. FESEM micrograph of Polyaniline (PANI).

Figure 4 (a) shows the XRD pattern of the PANI, which shows an amorphous nature in a partly crystalline condition with a diffraction peak at 22.34°. (200). Due to the repeating of benzenoid and quinoid rings in the PANI chains, this pattern displays poor conductive polymer crystallinity. Figure 4b shows the EDX pattern of PANI, which revealed that the key elements in PANI film are C, N, O, and Si, as seen by their peaks. The silica fiber employed as the substrate shared the silicon (Si) peak.

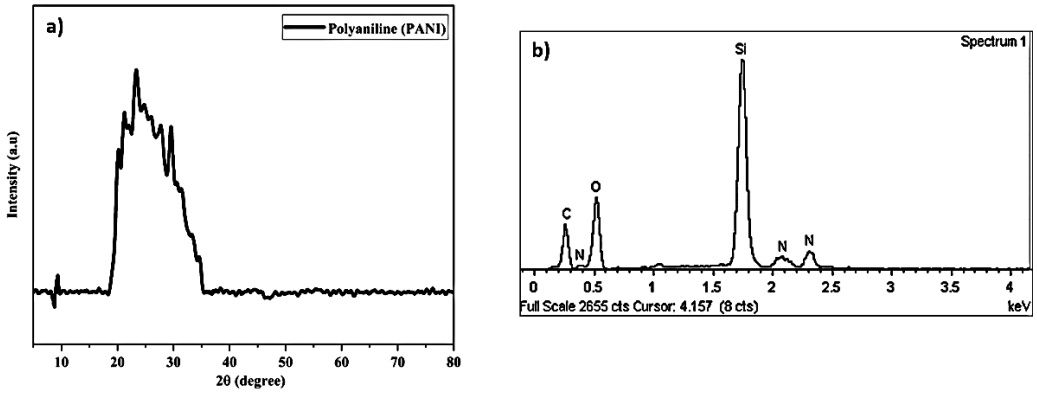

Figure 4. (a) XRD pattern of PANI, and (b) EDX measurement of PANI.

The atomic force microscope (AFM) can verify the average surface roughness and thicknesses of PANI. A 10×10 μm section of the boundary area was scanned for the AFM analysis. The average surface roughness values of the PANI were ≈ 23.4 nm, as shown in Figure 5 (a and b). As part of this study, the thicknesses of the PANI coatings were measured. As shown in Figure 5c, measurements were taken by surrounding parts of fiber with aluminum tape and then assessing the thickness differences between coated and uncoated fiber. The average thickness of the PANI coatings was 690 nm.

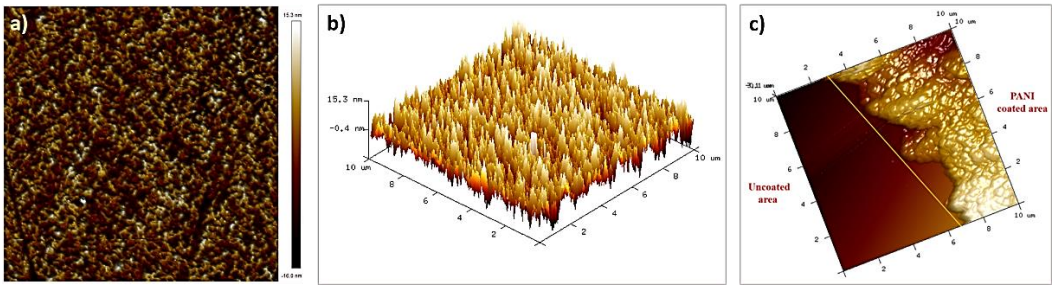

Figure 5. Figure 5. 2D topography AFM images (a) PANI, (b) 3D topography AFM images of PANI and (c) 3D topography AFM images of the boundary area between the uncoated and coated fiber for PANI sensing layer.

3. Results and Discussion

The absorption spectra of the developed sensor coated with PANI to synthetic air and 1.00% H_2 at room temperature. As shown in Figure 6a, the PANI coated sensor shows notable changes in absorption, particularly in the wavelength range of 550 to 850 nm. The sensor performance of the PANI coated sensor was monitored in term of cumulative absorbance, which is the product of a combination of response curve over a particular wavelength range. Figure 6b shows the dynamic response of PANI coated based sensors to H_2 concentrations in air ranging from 0.125% to 1.00% at room temperature. The response and recovery times of the developed PANI coated based sensor was 110 seconds and 160 seconds, respectively. The absorbance change was approximately 4% at 0.125% H_2 and 19% higher with 1.00% H_2. Compared with the results of previous studies [23,24]. the PANI coated based sensor showed greater H_2 absorbance and recovery, as well as advanced compromise differences.

The repeatability of the PANI coated based sensor was tested by exposing it to three cycles of 1.00% H_2. Overall, the PANI coated based sensor showed a strong and stable absorbance response as well as high repeatability towards H_2.

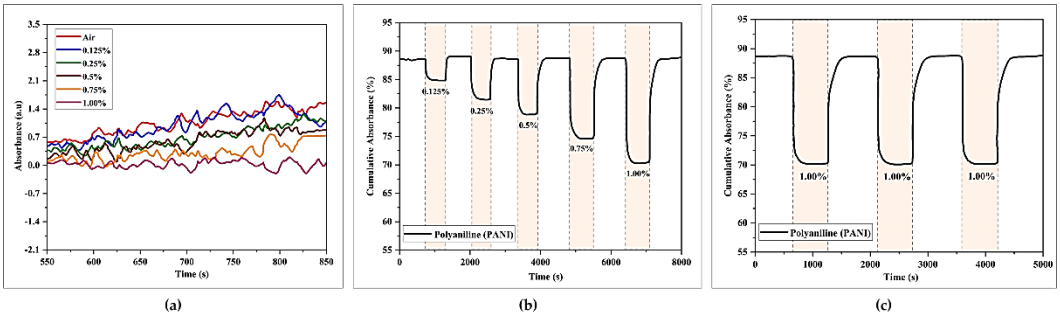

Figure 6. (a) Absorbance versus optical wavelength, (b) dynamic absorbance curves, and (c) repeatability of PANI coated based sensor towards H_2.

Figure 7a shows the absorption versus H_2 concentration for PANI coated based sensors. The PANI coated based sensors had a sensitivity of 15.928/vol% and a linearity slope of 98%. When measuring gas sensing properties, selectivity is an important key to consider. As shown in Figure 7b, the sensor's absorption properties toward ammonia (NH_3) and methane gas (CH_4) at a concentration of 1.00% were investigated. The PANI coated based sensor had a very high NH_3 absorption response but a substantially lower response for the other gases. Furthermore, the adsorption of PANI based materials was highly selective for polar molecules such as NH_3, whereas sensitivity was low for non-polar molecules such as H_2 and CH_4 [25].

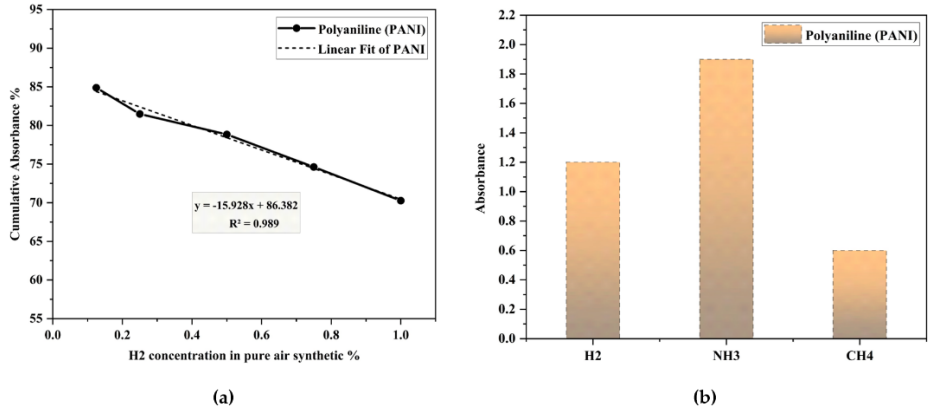

Figure 7. (a) Absorbance changes at different H_2 concentration for PANI coated based sensor and (b) the selectivity of PANI coated based sensor.

4. H_2 Mechanism of PANI Coated on Tarped Optical Fiber

The H_2 sensor with PANI mechanism consists of two parts, as illustrated in Figure 8. The first is the physical absorption of gas molecules in the PANI, which causes changes in the refractive index of the optical fiber's surface, which in turn will lead to changes in the amount of light transmitted in the fiber. A higher RI would allow more-light to escape, lowering the intensity of light detected by the spectrophotometer. The charge transfer between the adsorbent and the PANI molecules is the second step. The charge transfer from the electron donating H_2 gas to PANI changes the surface chemistry of the sensor layer as it is absorbed into the walls and sides of PANI. This involves changes in the sensor layer's optical properties, as the light that traveled through the fiber is absorbed by environmental changes, causing spectrum shifts.

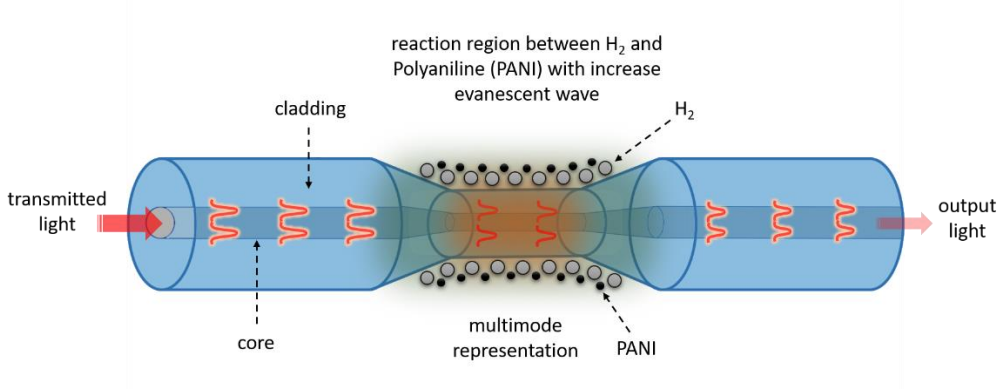

Figure 8. Hydrogen-PANI sensing mechanism.

5. Conclusions

Using a drop casting technique, this work proved that optical fiber sensors could be fabricated from Polyaniline (PANI). The response of the established sensor to various concentrations of H_2 gas at room temperature was used to measure its efficiency. When exposed to 1.00 % H_2 in synthetic air, the PANI coated based sensor enhanced its absorption response by 19%, according to these findings. The selectivity invistigation indicate that the PANI based optical sensor response strongly towards ammonia, methane and hydtogen chemicals.The findings suggest that an affordable and easy methodology may be utilized to enhance an effective, accurate, and repeatable H_2 sensor in real-world atmospheric conditions.

Author Contributions: Conceptualization, M.M.A., and M.H.Y.; methodology, M.M.A., S.H.G., A.H.I and M.H.Y.; writing—original draft preparation, M.M.A.; review and editing, M.H.Y., M.A.M., S.P., and N.A. All authors have read and agreed to the published version of the manuscript.

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Conflicts of Interest: The authors declare no conflict of interest.

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