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**UNIVERSITI PUTRA MALAYSIA**

RICULTURE • INNOVATION • LIFE

**1st International Electronic Conference on Chemical Sensors and  
Analytical Chemistry**

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

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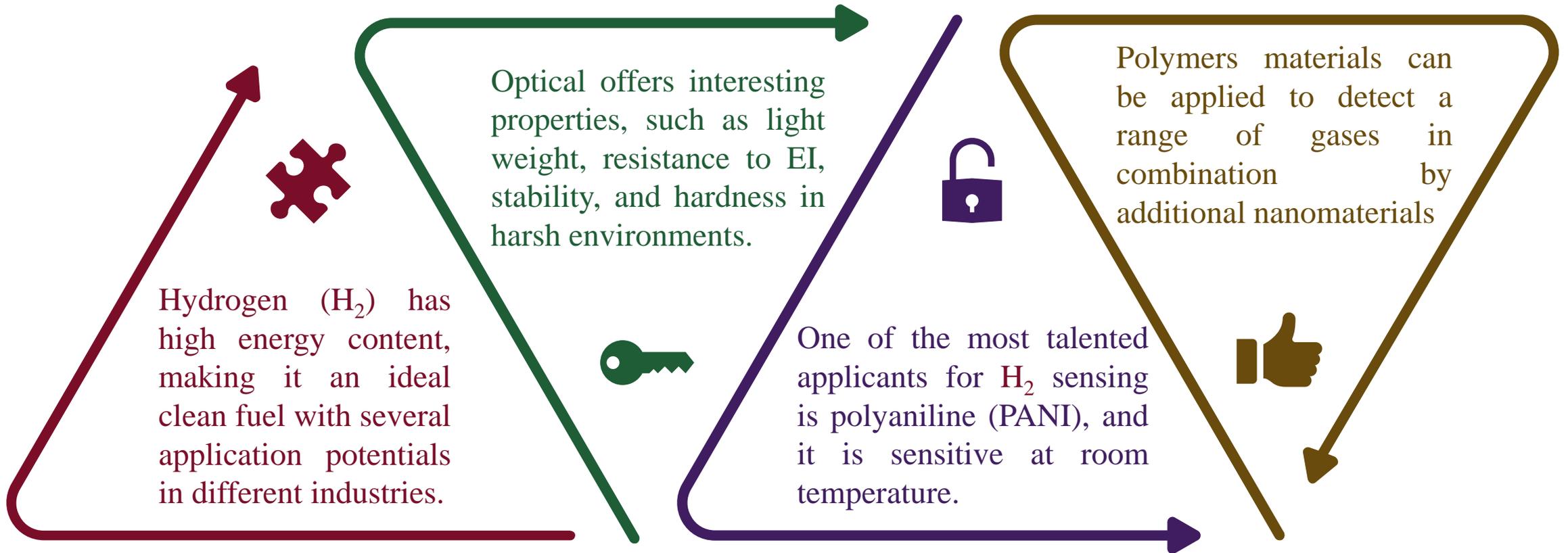
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# CONTENT



# INTRODUCTION



# PROBLEM STATEMENT

- Hydrogen is flammable at concentrations  $> 4$  vol% in the air and can explode at a wider range of 15–59 vol% at standard pressure.
- Currently, there are several types of H<sub>2</sub> sensors available.
  - Electrical sensors (i.e. chemiresistor or microelectronic) are susceptible to electromagnetic interference (EMI) which can affect their response to signals.



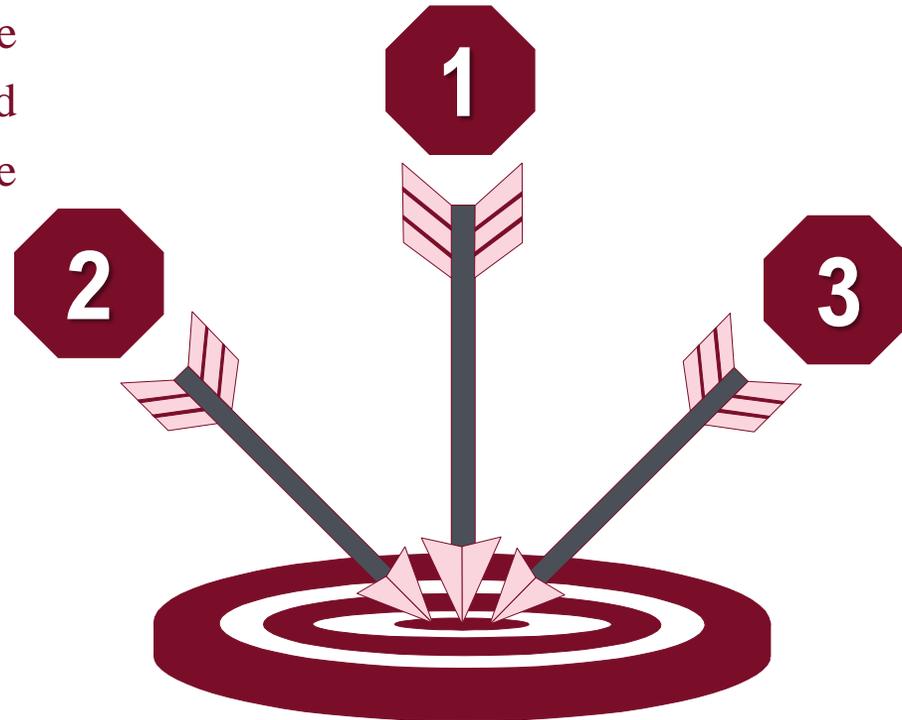
- On the other hand, optical fiber that offers other advantages, such as lightweight, small size, resistance to EMI, non-inductiveness, and ruggedness in harsh environments.
  - These properties make optical fiber an ideal candidate for H<sub>2</sub> detection in rugged environment.

# OBJECTIVES

To design and develop hydrogen gas sensors based on PANI coated on tapered optical fiber via drop-casting technique.

To evaluate the optical fiber sensor performance (sensitivity, response and recovery time, repeatability, and selectivity) based on absorbance measurement.

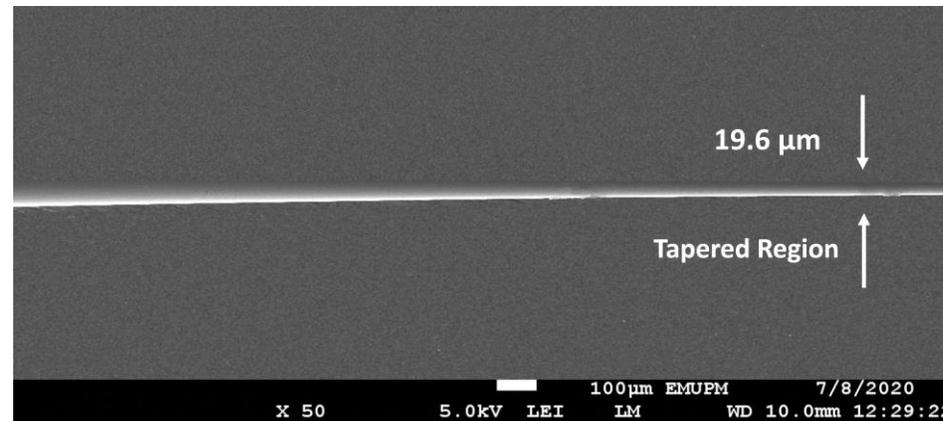
To discuss the sensing mechanism of gas molecules-sensing layer interaction of tapered optical fiber sensor.



# METHODOLOGY

## Fabrication of Tapered Optical Fiber

- Multimode Optical fiber (MMF) was fabricated with cladding and core diameters of 125  $\mu\text{m}$  and 62.5  $\mu\text{m}$  respectively, as a transducing platform.
- The MMF was tapered from cladding diameter of 125  $\mu\text{m}$  to waist diameter of 20  $\mu\text{m}$ , waist-length of 10 mm, and down taper and up of 5 mm.
- The tapering was done using the Vytran glass processing machine (Vytran GPX-3400).
- The machine works based on a heating and pulling process, using a graphite filament as a heater to achieve the desired geometry of the tapered profile.



(SEM) micrograph of the transition region of the prepared tapered multimode optical fiber (MMF)

# METHODOLOGY

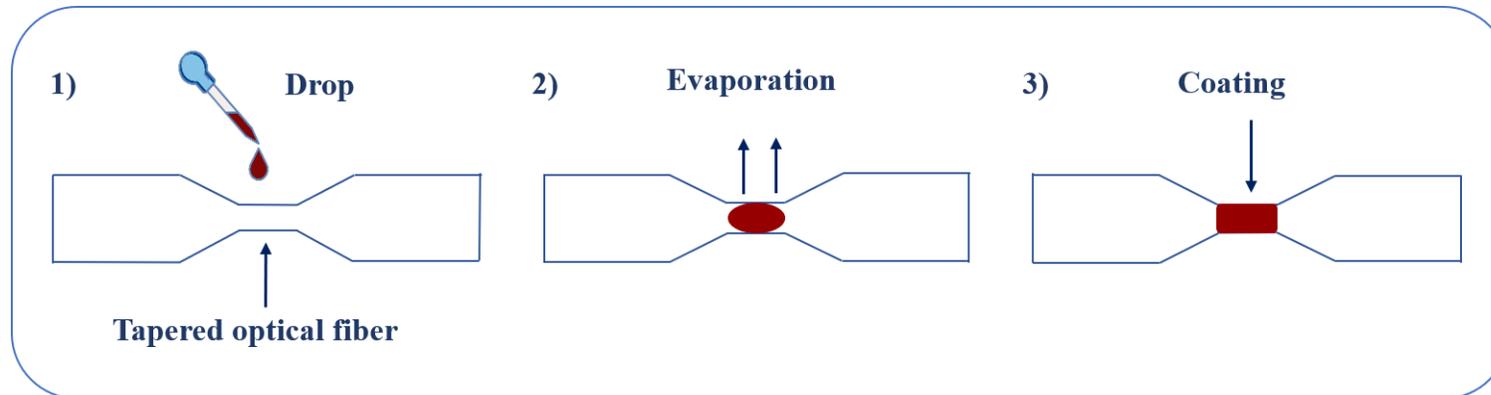
## Palladium Functionalization of the Tapered Optical Fiber

➤ The materials used to synthesis the PANI, as follow;

- aniline (0.16 M)
- 0.05 M perchloric acid (HClO<sub>4</sub>)
- 0.16 M of ammonium peroxydisulfate
- Ammonium peroxydisulfate solution

➤ The coating of the tapered optical fiber was done using the drop-casting technique.

- A drop of the mixture (approx. 10 μL) was dropped into the base of the tapered optical fiber.
- Heating the sample at 80 °C for 15 minutes in the oven to ensure complete evaporation of the aqueous medium.

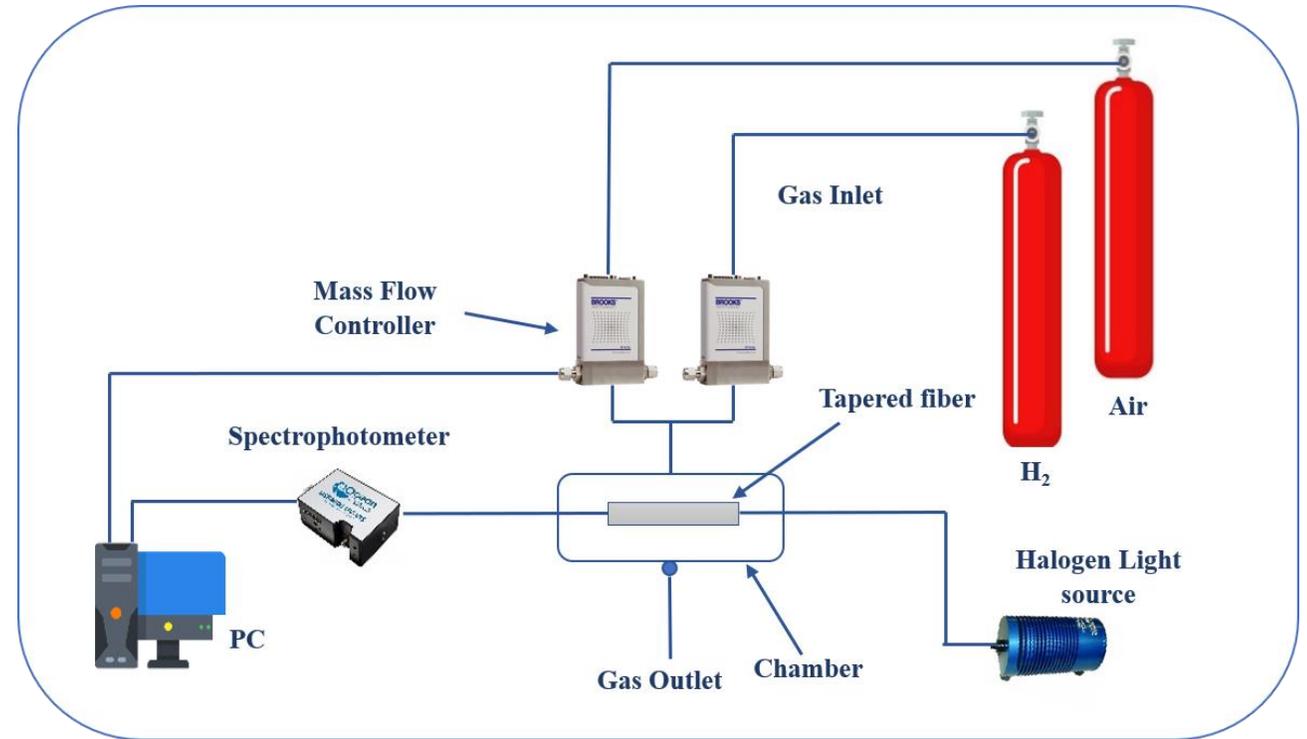


Steps of the drop-casting technique.

# METHODOLOGY

## The experimental setup

- The gas optical sensing system consists of a light source (Tungsten Halogen, HL-2000, Ocean Optics USA) with coverage wavelength of 360 to 2500 nm.
- A spectrophotometer (USB 4000, Ocean Optics USA) with a detection range of 200-1100 for monitoring the optical absorption spectrum.
- A dedicated gas chamber.
- The PANI coated sensor was placed in a closed gas unit and purged with the centrifuge from a computer-regulated mass flow controller at a gas flow rate of 200 sccm.

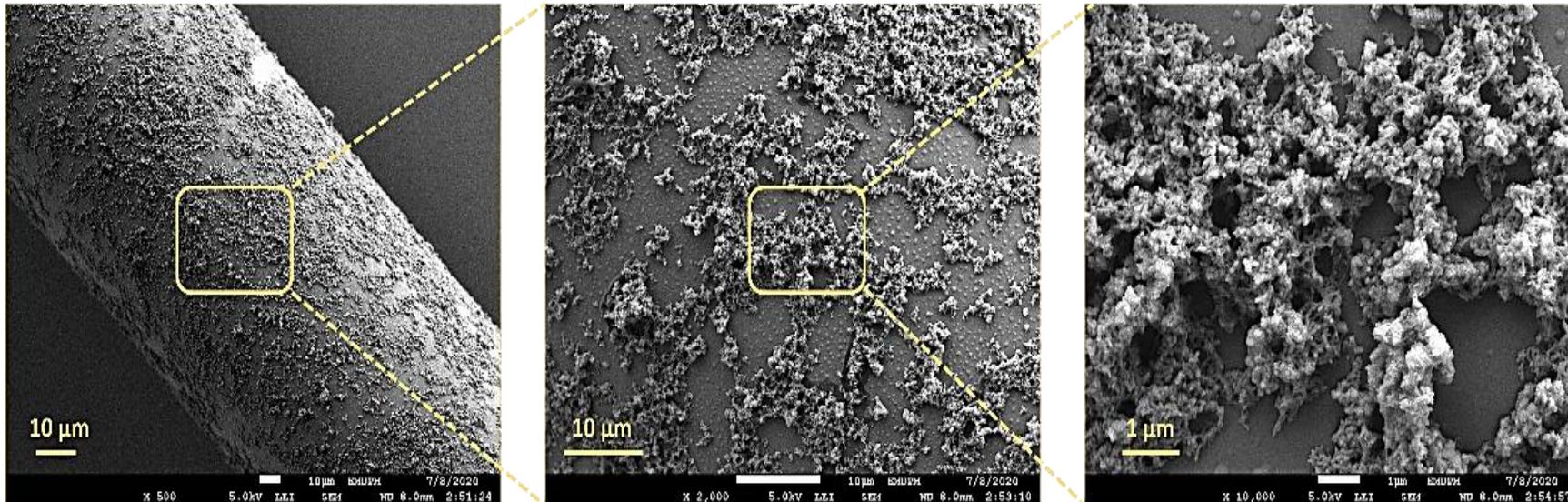


The experimental setup of the H<sub>2</sub> sensor.

# METHODOLOGY

## Material Characterization

- The films' morphology was observed using Field Emission Scanning Electron Microscope (FESEM) (JSM-7600F).
- The FESEM images of PANI show that PANI is mainly made of irregular grains and chips with sharp edges. Furthermore, the structure appears completely porous, forming extremely fine polyaniline particles

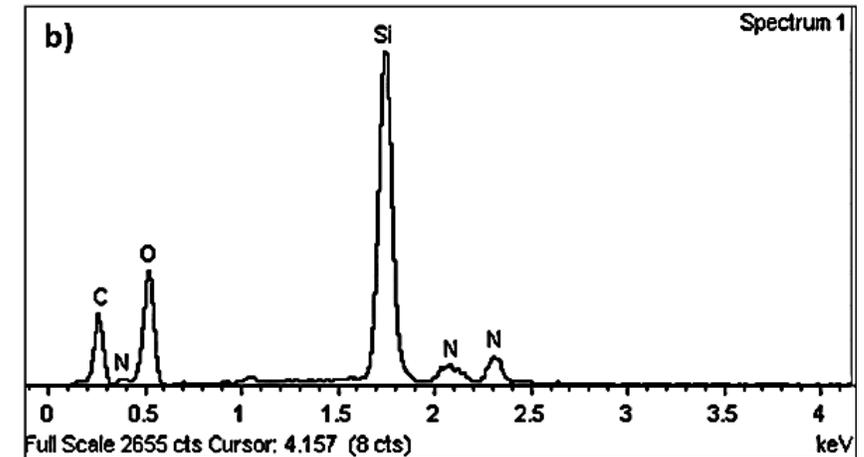
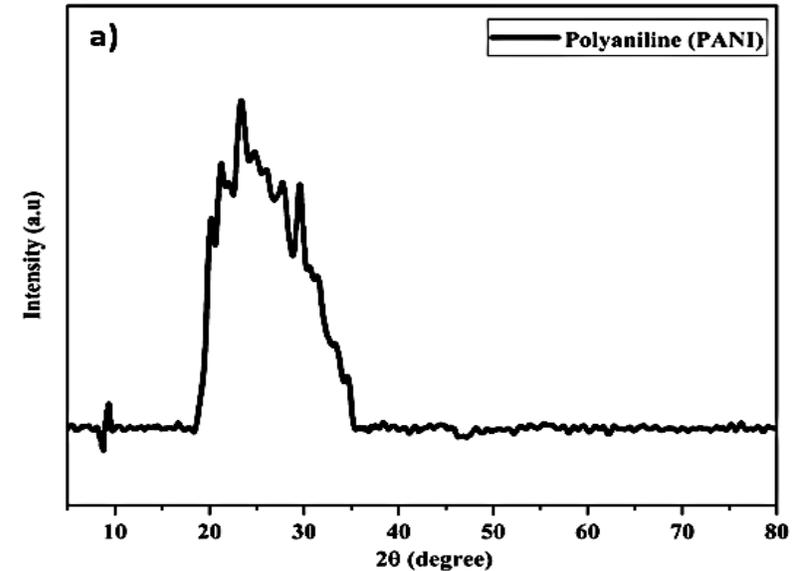


FESEM micrograph of polyaniline (PANI). .

# METHODOLOGY

## Material Characterization

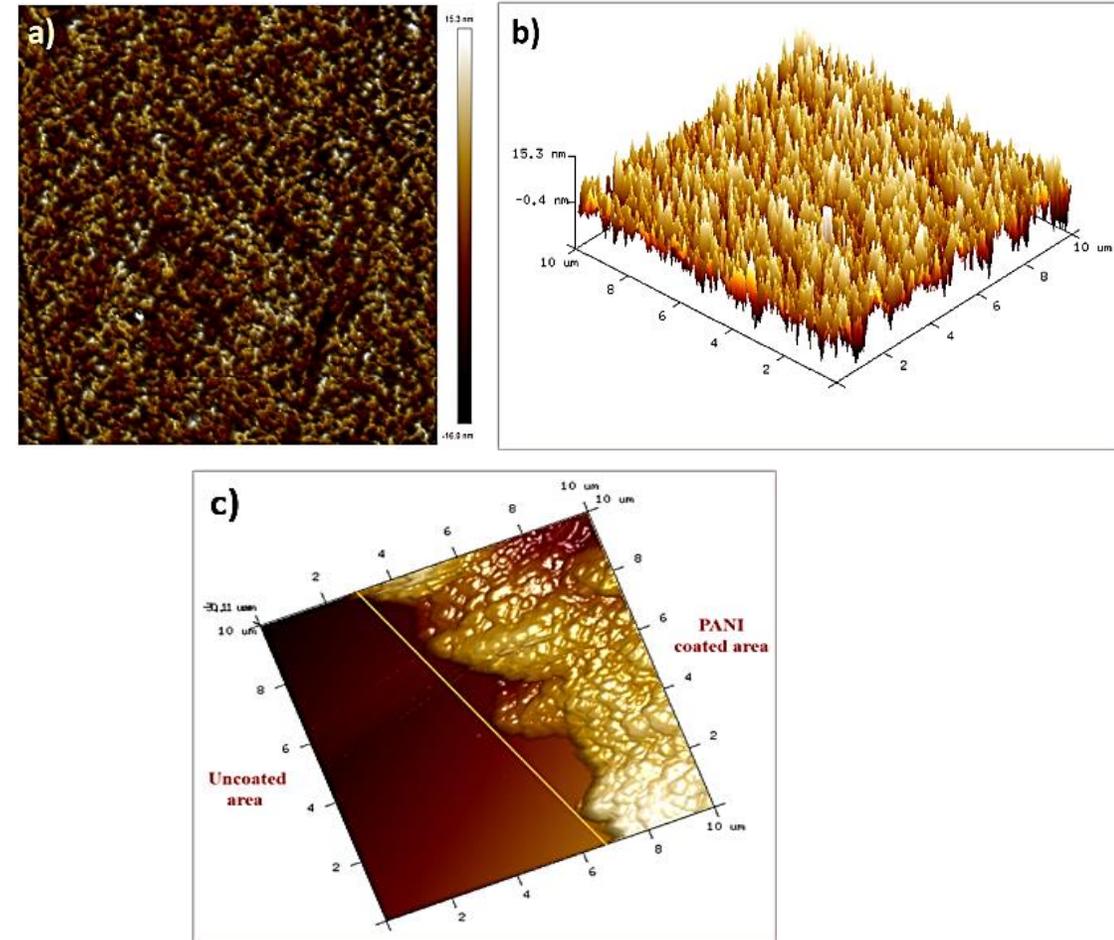
- Material identification and phase transition of PANI was observed by an X-Ray Diffraction (XRD) analysis (APD 2000) as shown in Figure (a).
  - XRD patterns of the PANI coated sensor recorded in range  $2\theta$ , from  $10^\circ$  to  $80^\circ$ .
  - An amorphous nature in a partially crystalline state with a diffraction peak at  $22.34^\circ$  (200).
- 
- The elemental composition was determined through an Energy Dispersive X-Ray (EDX) analysis as shown in Figure (b).
  - The EDX pattern of PANI showed that the important elements in PANI films are C, N, O and Si, as evidenced by their respective peaks.



# METHODOLOGY

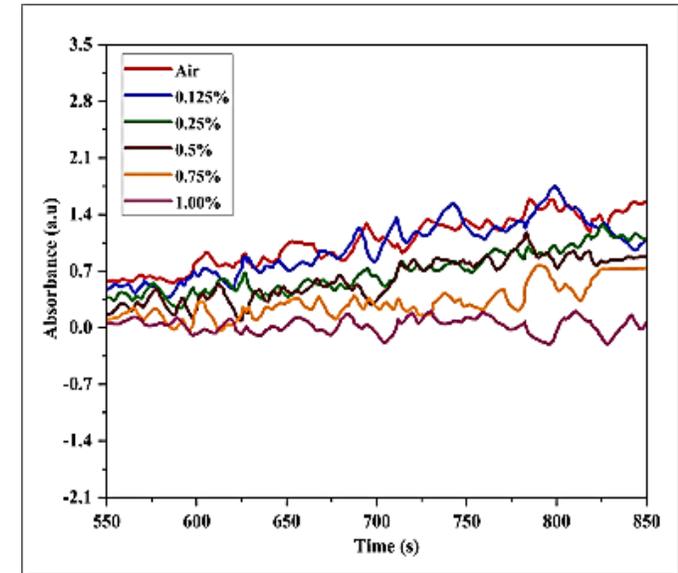
## Material Characterization

- The atomic force microscope (AFM) can verify the average surface roughness and thicknesses of PANI
  - A  $10 \times 10 \mu\text{m}$  section of the boundary area was scanned for the AFM analysis.
  - The average surface roughness values of the PANI were  $\approx 23.4 \text{ nm}$ , as shown in (a and b).
- As part of this study, the thicknesses of the PANI coatings were measured.
  - As part of this study, the thicknesses of the PANI coatings were measured. As shown in Figure 5c, The average thickness of the PANI coatings was  $690 \text{ nm}$ .

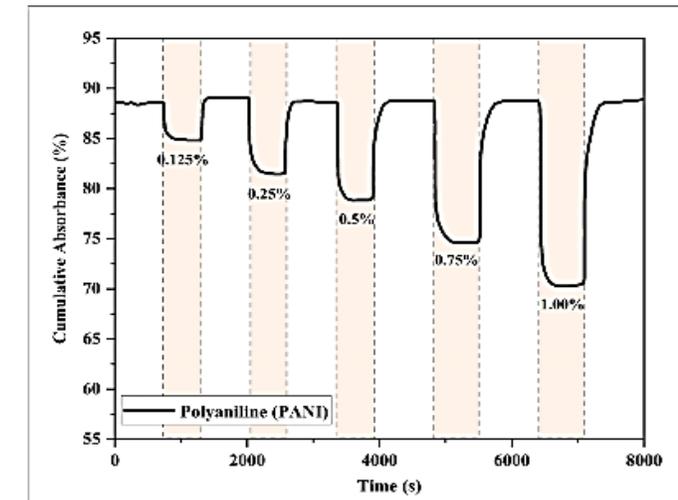


# RESULTS AND DISCUSSION

- The absorption spectra of the sensor coated with PANI to synthetic air at room temperature with different concentration 0.125% to 1.00% H<sub>2</sub>.
- The PANI sensor demonstrated notable changes in absorbance, especially in the wavelength range of 550-850 nm as shown in Figure (a).
- The response time and recovery time of the PANI coated sensor was 110 sec and 160 sec respectively. Changes in absorption at 0.125% H<sub>2</sub> are about 4% and 19% higher at 1.00% H<sub>2</sub> as shown in Figure (b).
- The PANI coated sensor showed stronger absorbance and recovery of H<sub>2</sub> at higher absorption changes.



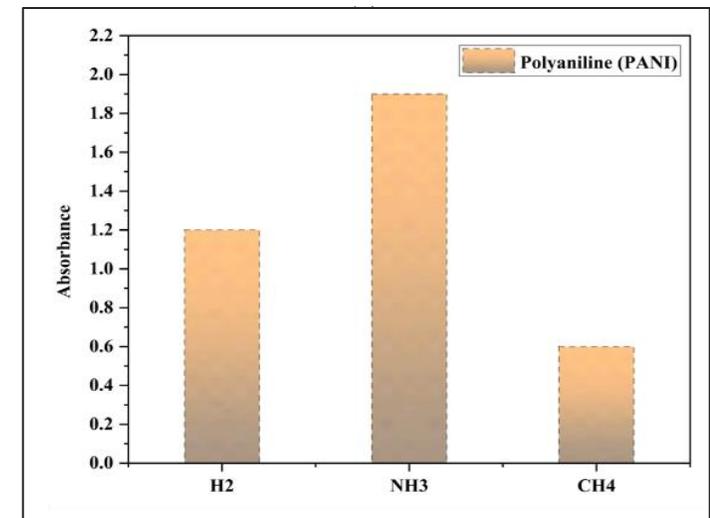
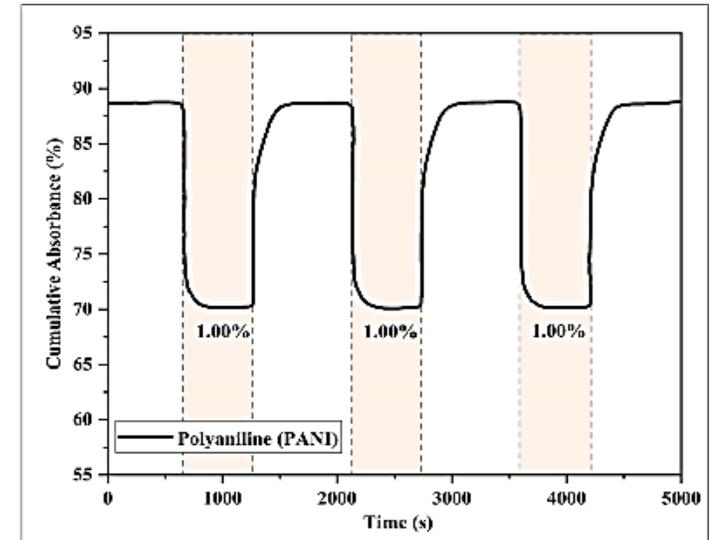
(a)



(b)

# RESULTS AND DISCUSSION

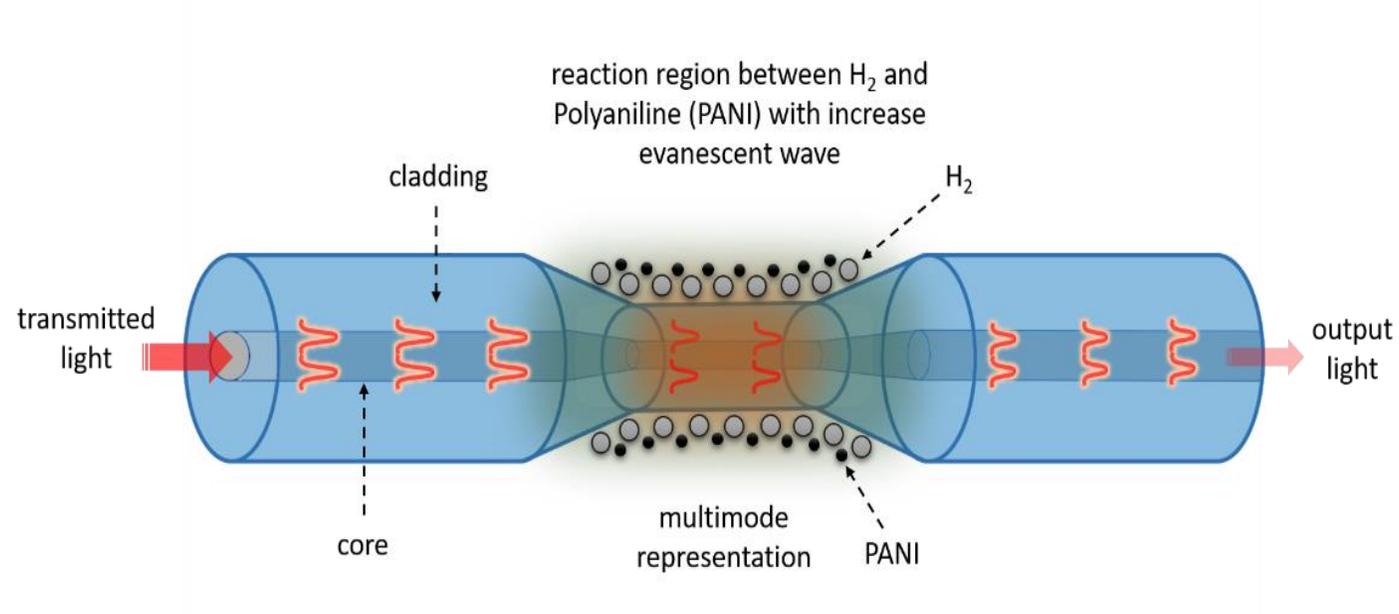
- Sensor repeatability was confirmed by exposure of the sensor to 3 cycles of 1.00% H<sub>2</sub>. Overall, the PANI coated sensor showed a high level of good repeatability of H<sub>2</sub>.
- A test for selectivity was done for PANI coated sensor toward NH<sub>3</sub> and CH<sub>4</sub> gas at 1.00%.
- The PANI-coated based sensor had a very high NH<sub>3</sub> absorption response but a substantially lower response for the other gases.



# RESULTS AND DISCUSSION

## The Sensing Mechanism for Tapered Pd NPs Coated Optical Fibers

- The H<sub>2</sub> sensor with PANI mechanism consists of two parts.
- The first is the physical absorption of gas molecules in the PANI.
- The charge transfer between the adsorbent and the PANI molecules is the second step.



Hydrogen-palladium sensing mechanism.

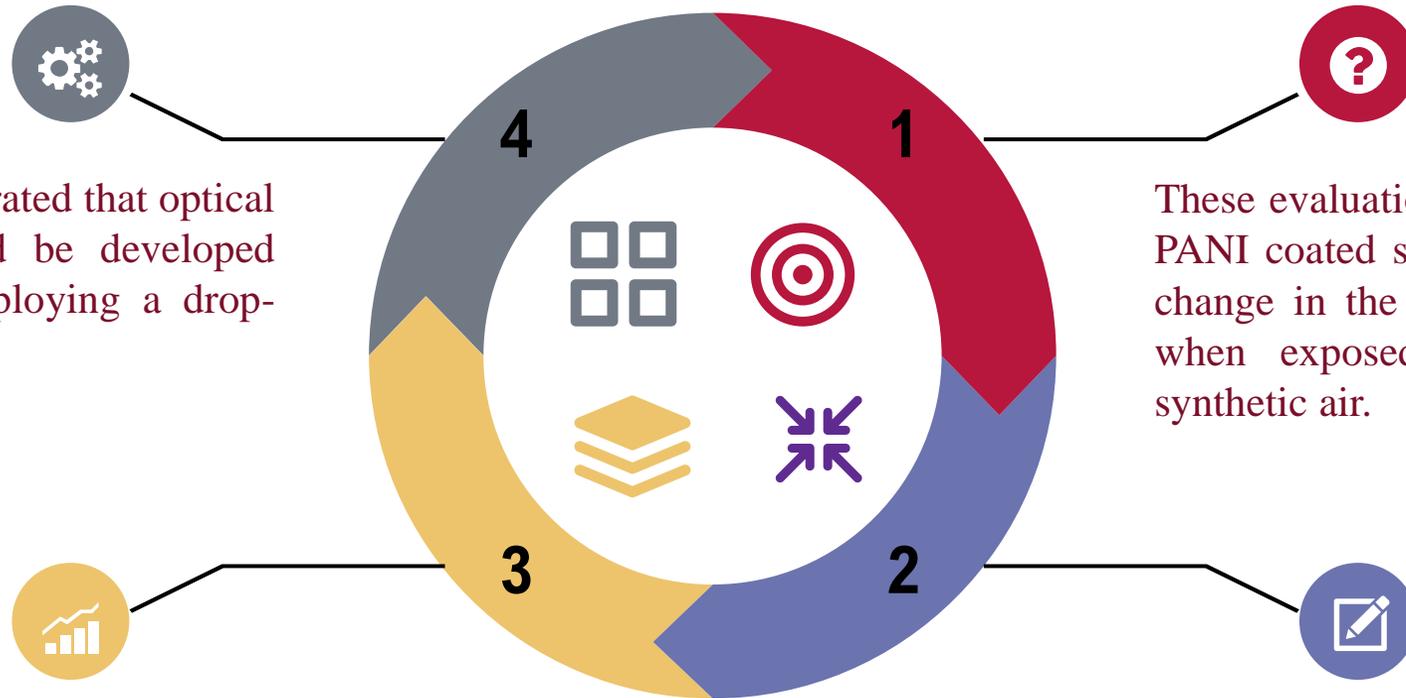
# CONCLUSION

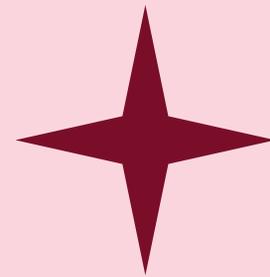
This study demonstrated that optical fiber sensors could be developed from PANI by employing a drop-casting technique.

The performance of the developed sensor was evaluated in terms of its response at room temperature using different concentrations of H<sub>2</sub> gas.

These evaluations indicated that the PANI coated sensor showed a 19% change in the absorbance response when exposed to 1.00% H<sub>2</sub> in synthetic air.

It is possible to develop an efficient and save H<sub>2</sub> sensor by using a simple and cost-effective approach under real conditions.





**THANK  
YOU**