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Tapered Optical Fiber for Hydrogen Sensing Application Based on Molybdenum trioxide (MoO_3)

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INTRODUCTION

Hydrogen (H_2) has high energy content, making it an ideal clean fuel with several application potentials in different industries.

Optical offers interesting properties, such as light weight, resistance to EI, stability, and hardness in harsh environments.

One of the most talented applicants for **H_2** sensing is **MoO$_3$**, and it is sensitive at room temperature.

Metal oxide materials can be applied to detect a range of gases in combination by additional nanomaterials

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.

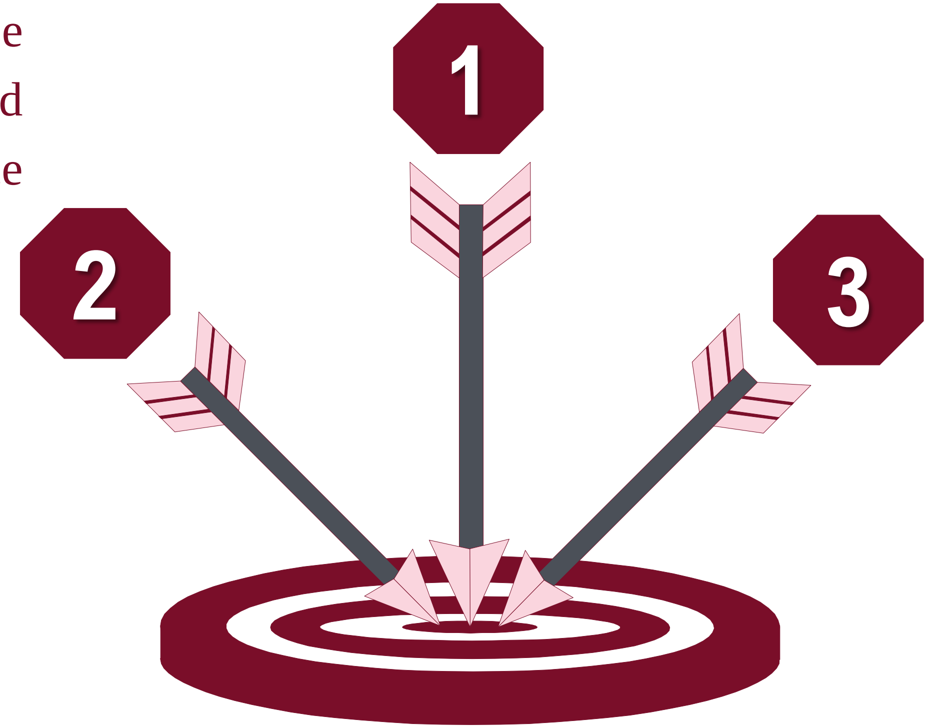
- Currently, there are several types of H_2 sensors available.
    - Electrical sensors (i.e. chemiresistor or microelectronic) are susceptible to electromagnetic interference (EMI) which can affect their response to signals.

- 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_2 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.

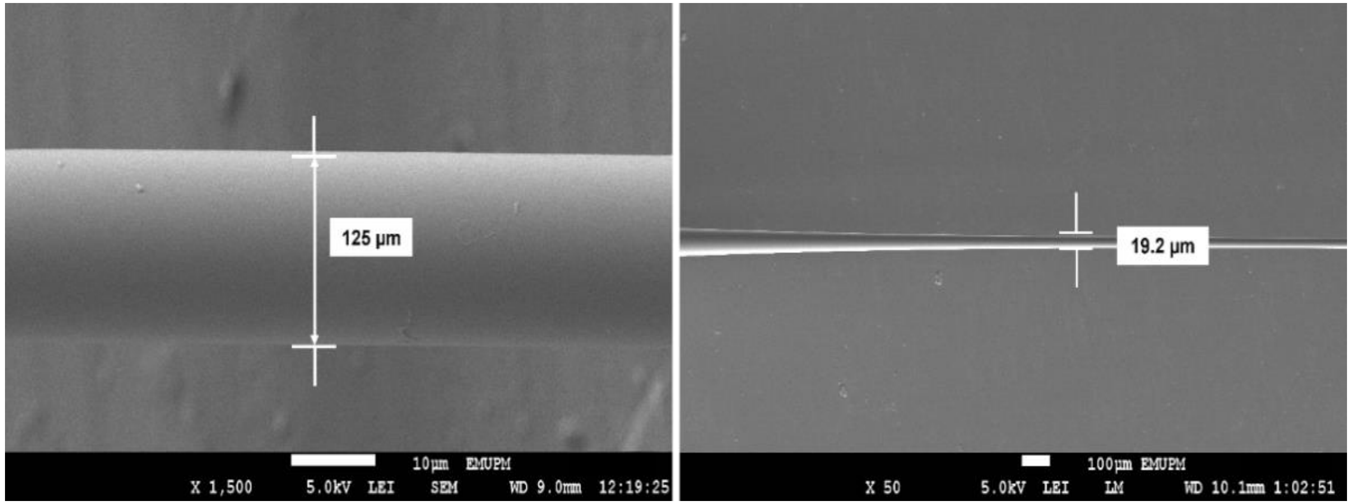


To discuss the sensing mechanism of gas molecules-sensing layer interaction of tapered optical fiber sensor.

METHODOLOGY

Fabrication of Tapered Optical Fiber

- Multimode Optical fiber (MMF) was fabricated with cladding and core diameters of 125 µm and 62.5 µm respectively, as a transducing platform.
- The MMF was tapered from cladding diameter of 125 µm to waist diameter of 20 µm, 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

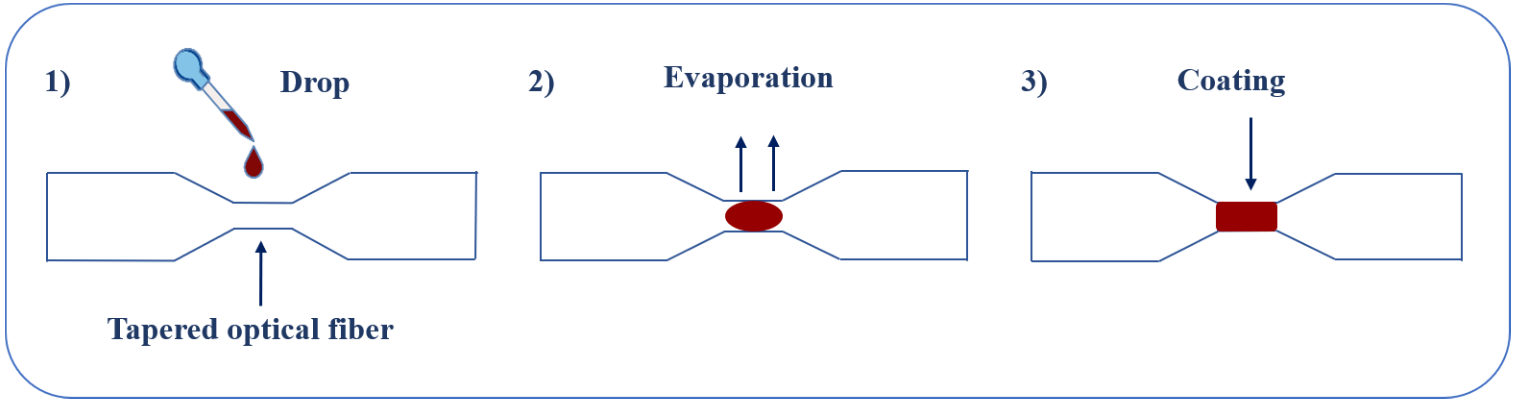
MoO_3 Functionalization of the Tapered Optical Fiber

- ➢ Molybdenum trioxide (MoO_3) powder was synthesized by simple solid decomposition method.

- 2.50 g of ammonium heptamolybdate tetrahydrate.
- Annealed in an alumina crucible at 500 °C for three hours in the air.
- A 10 mL of deionized water.
- A milk-white suspension after ultrasonic treatment for 30 minutes.

- ➢ 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 ^oC for 15 minutes in the oven to ensure complete evaporation of the aqueous medium.



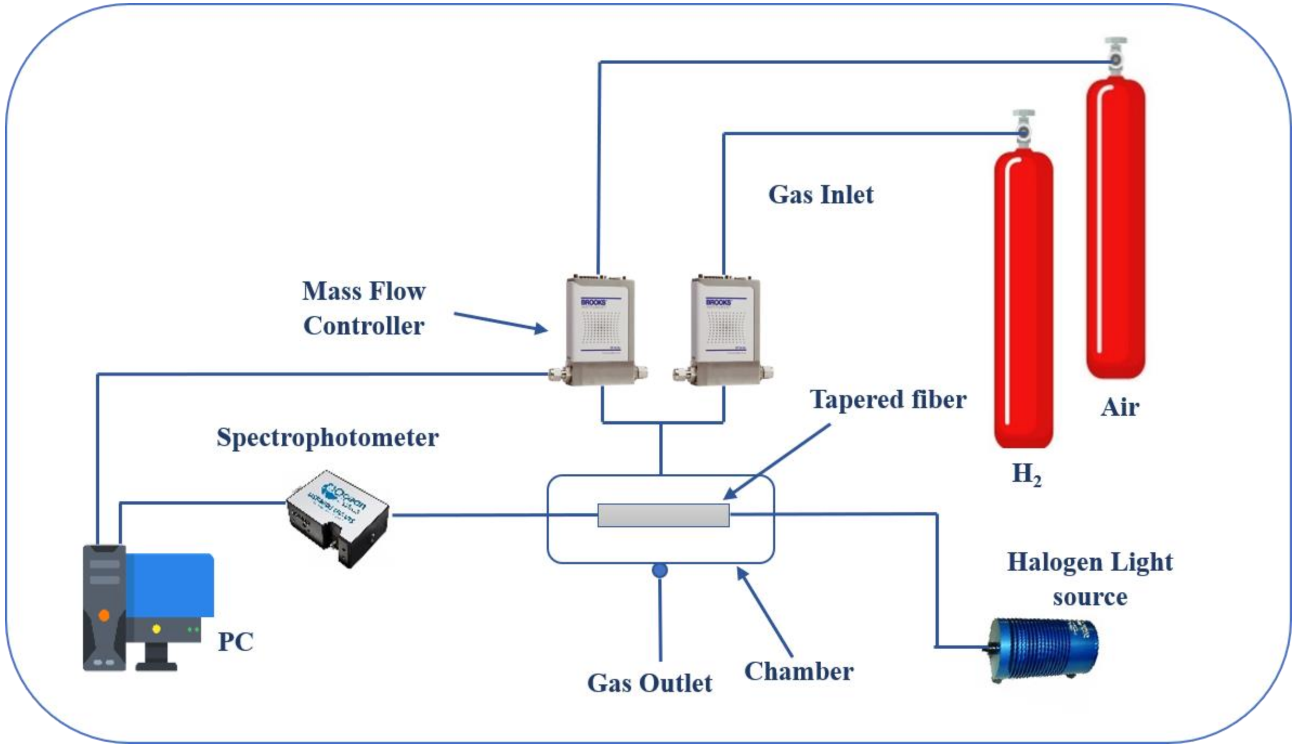
Steps of the drop-casting technique.

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 MoO_3 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.



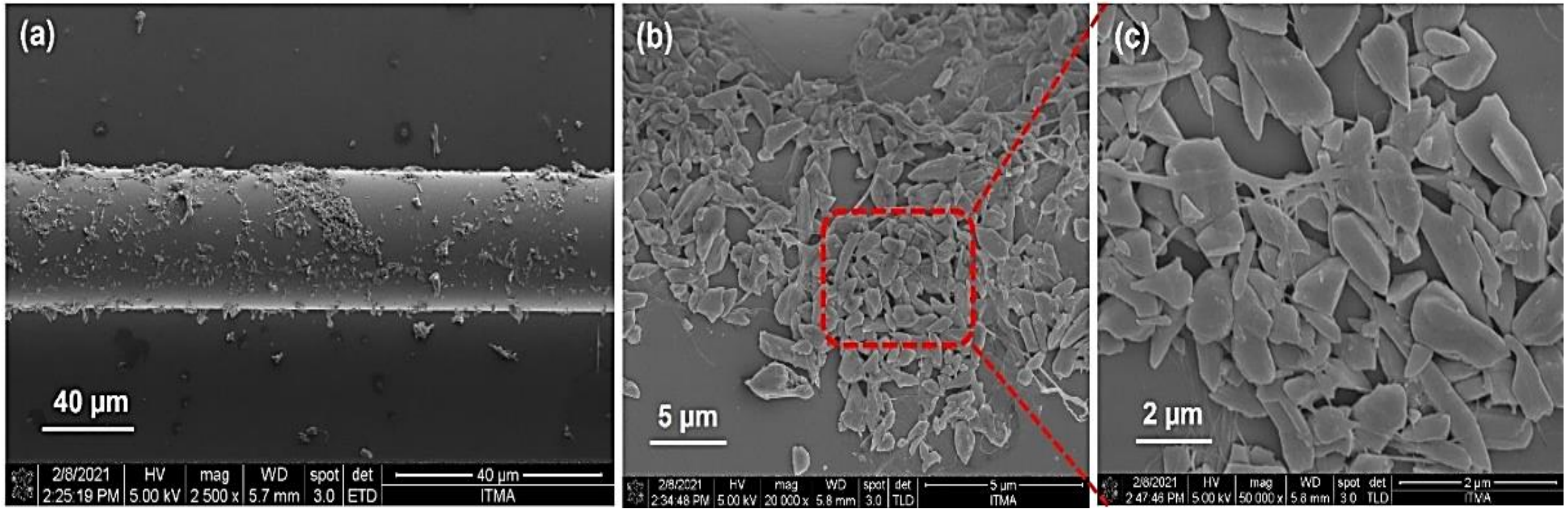
The experimental setup of the H_2 sensor.

METHODOLOGY

Material Characterization

- The films' morphology was observed using Field Emission Scanning Electron Microscope (FESEM) (JSM-7600F).

- The FESEM images of MoO_3 shows that it is The particles agglomerated in nature after attempting to form thin plate or flake-like formations. The gas analyte will interact more effectively with this plate-like structure.



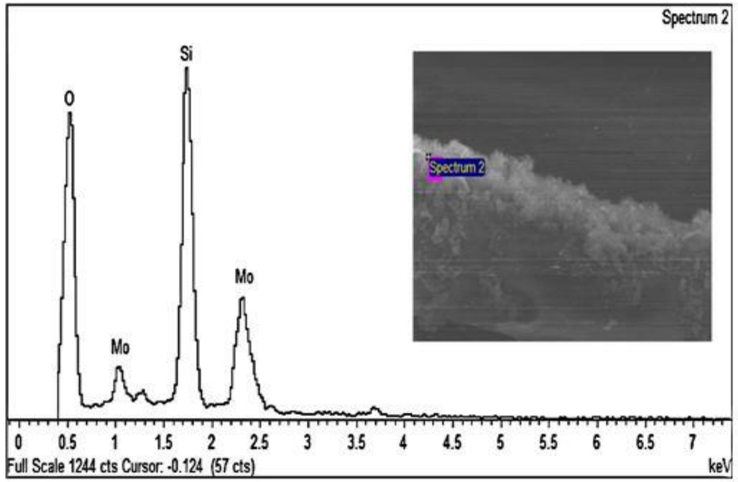
FESEM micrograph of MoO_3 .

METHODOLOGY

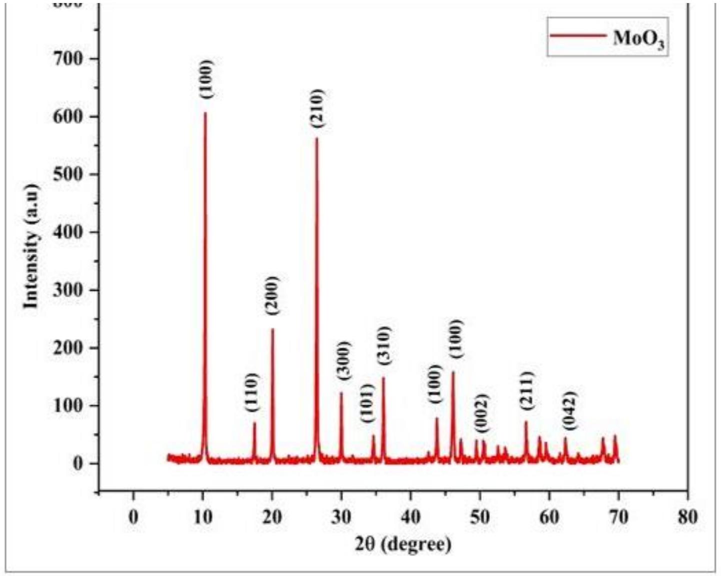
Material Characterization

- The elemental composition of MoO_3 was determined through an Energy Dispersive X-Ray (EDX) analysis as shown in Figure (b).
  - The EDX pattern of MoO_3 showed that the important elements in MoO_3 films are Mo, O and Si, as evidenced by their respective peaks.

- Material identification and phase transition of MoO_3 was observed by an X-Ray Diffraction (XRD) analysis (APD 2000) as shown in Figure (a).
  - XRD patterns of the coated sensor recorded in range 2θ, from 5° to 70°.
  - All the XRD peaks can be distinguished for the sample, and single-phase can be as-signed to crystal structures. The prominent diffraction peak corresponding to planes (100) and (210) placed at the highest density of MoO3.



(a)



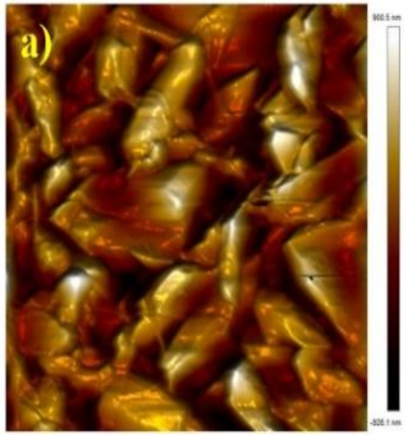
(b)

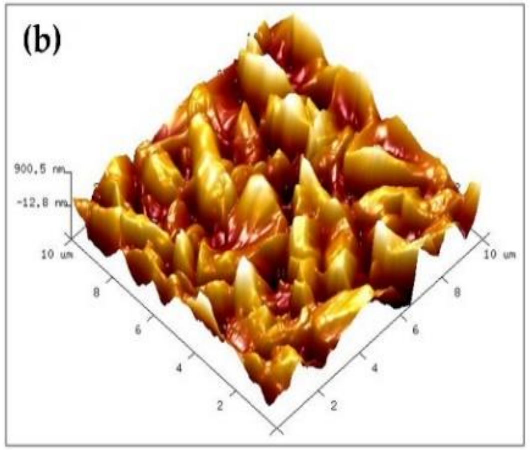
METHODOLOGY

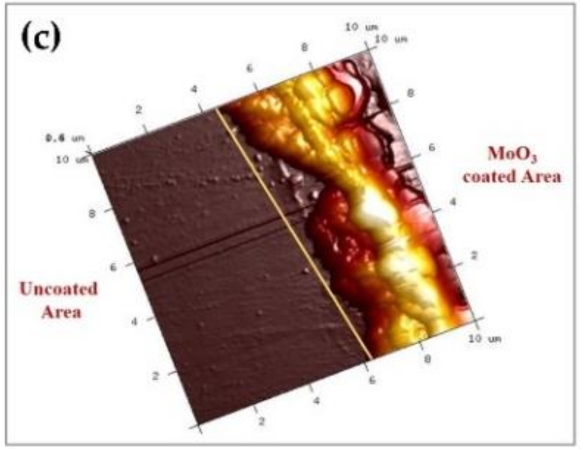
Material Characterization

- The atomic force microscope (AFM) can verify the average surface roughness and thicknesses of MoO_3
  - A 10×10 µm section of the boundary area was scanned for the AFM analysis.
  - The average surface roughness values of the MoO_3 were ≈ 44.98 nm nm, as shown in (a and b).

- As part of this study, the thicknesses of the MoO_3 coatings were measured.
  - As part of this study, the thicknesses of the MoO_3 coatings were measured. As shown in Figure 5c, The average thickness of the MoO_3 coatings was 181 nm.







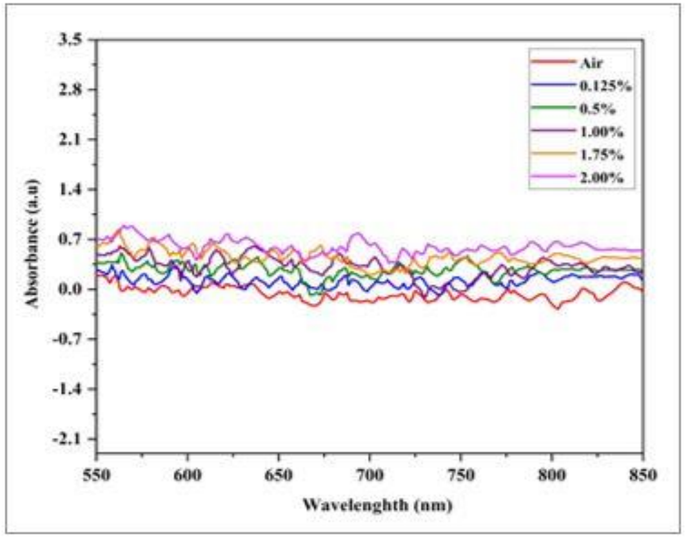
RESULTS AND DISCUSSION

- The absorption spectra of the sensor coated with MoO_3 to synthetic air at room temperature with different concentration 0.125% to 2.00% H_2 .

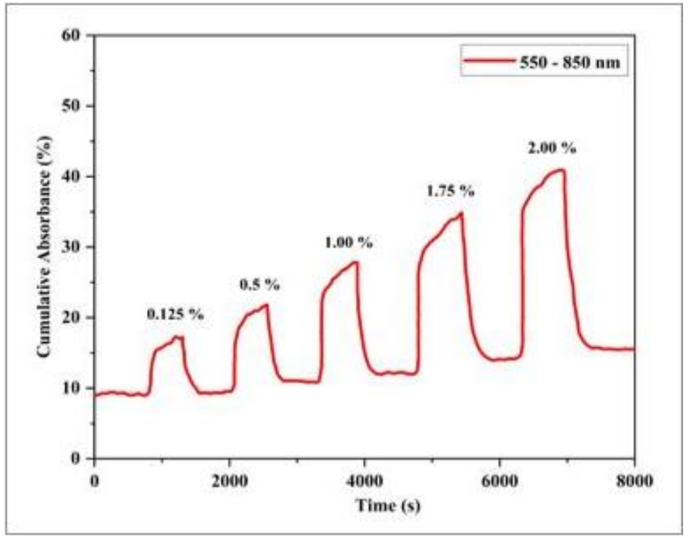
- The MoO_3 sensor demonstrated notable changes in absorbance, especially in the wavelength range of 550-850 nm as shown in Figure (a).

- The response time and recovery time of the MoO_3 coated sensor was 200 s and 220 s respectively. Changes in absorption at 0.125% H_2 are about 8% and 25% higher at 2.00% H_2 as shown in Figure (b).

- The MoO_3 coated sensor showed stronger absorbance and recovery of H_2 at higher absorption changes.



(a)



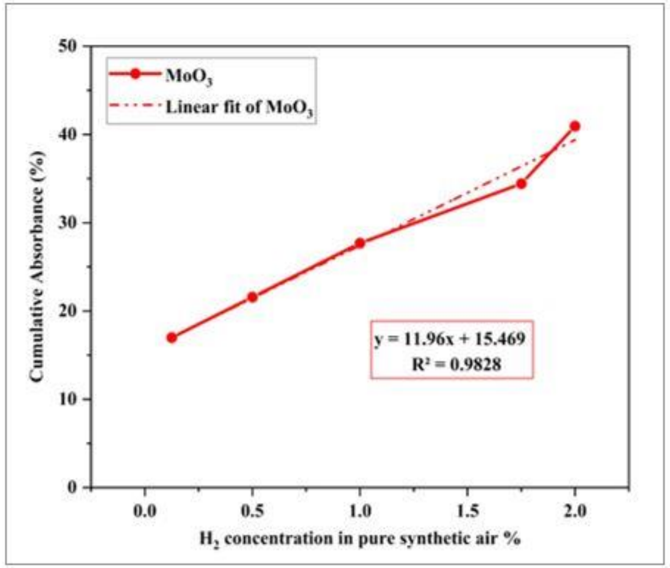
(b)

RESULTS AND DISCUSSION

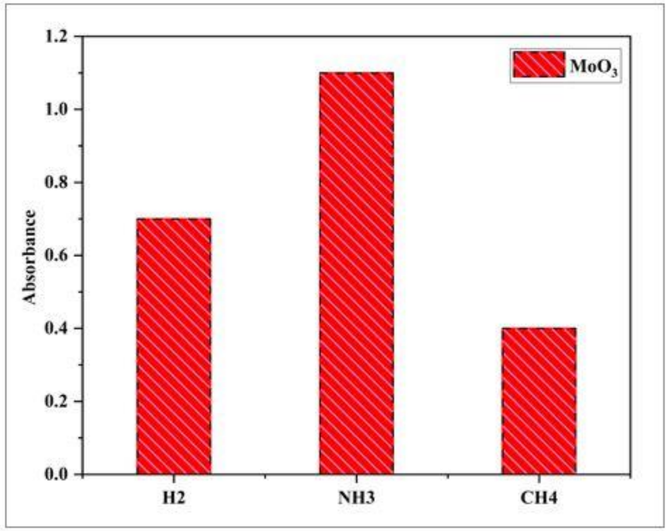
- Sensor repeatability was confirmed by exposure of the sensor to 3 cycles of 2.00% H_2. Overall, the MoO_3 coated sensor showed a high level of good repeatability of H_2.

- A test for selectivity was done for MoO_3 coated sensor toward NH_3 and CH_4 gas at 1.00%.

- The MoO_3 coated based sensor had a very high NH3 absorption response but a substantially lower response for the other gases.

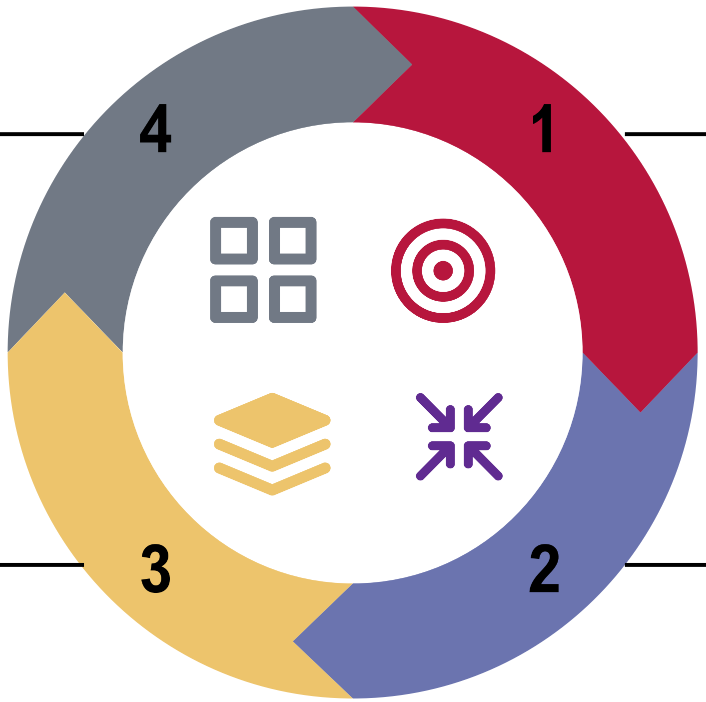


(a)



(b)

CONCLUSION



This study demonstrated that optical fiber sensors could be developed from MoO_3 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_2 gas.

These evaluations indicated that the MoO_3 coated sensor showed a 25% change in the absorbance response when exposed to 2.00% H_2 in synthetic air.

It is possible to develop an efficient and save H_2 sensor by using a simple and cost-effective approach under real conditions.

THANK YOU