

# Chemical Bath Deposition of MoS<sub>2</sub> Thin Films on Plastic Optical Fibre for Room-Temperature Hydrogen Sensing

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Hydrogen gas (H<sub>2</sub>) is an increasingly vital energy carrier, but its highly flammable nature demands reliable and selective sensing systems. In this study, an optical H<sub>2</sub> sensor was developed by coating two-dimensional molybdenum disulfide (MoS<sub>2</sub>) thin films onto a side-polished plastic optical fibre (POF). The MoS<sub>2</sub> sensing layer was synthesized using the chemical bath deposition (CBD) method, producing nanostructured films with porous morphology (10–40 nm nanogranules) and good crystallinity as confirmed by XRD. The deposited MoS<sub>2</sub> layer adhered uniformly to the fibre surface, enabling strong interaction with the guided light through the exposed fibre core. Optical absorbance measurements revealed a clear, consistent response to H<sub>2</sub> concentrations (0.25–1.00 %), with a linear sensitivity of 0.3084 a.u./% H<sub>2</sub> (R<sup>2</sup> = 0.9476). The sensor exhibited rapid response times decreasing from 5 min at 0.25 % H<sub>2</sub> to 3 min at 1.00 % H<sub>2</sub>, and recovery times between 5–7 min depending on concentration. These results confirm that nanostructured, crystalline MoS<sub>2</sub> coatings provide abundant active sites for H<sub>2</sub> adsorption, leading to fast and sensitive optical detection. This integration of CBD-grown MoS<sub>2</sub> with optical fibres demonstrates a promising pathway for low-power, electromagnetic interference (EMI) immune, and compact H<sub>2</sub> sensing applications.

**Keywords:** MoS<sub>2</sub>, hydrogen sensor, optical fibre, optical sensing, chemical bath deposition, room-temperature

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H<sub>2</sub> is increasingly regarded as a clean and sustainable energy vector due to its high energy density, non-toxic nature, and zero carbon emissions when employed in fuel cells [1, 2]. It plays a pivotal role in diverse industrial processes such as petroleum refining, ammonia synthesis, and methanol production [3, 4]. With growing momentum toward global decarbonization, H<sub>2</sub> is expected to support future low-carbon energy systems through grid stabilization, long-duration storage, and fuel for transportation infrastructure [5, 6].

Nevertheless, the physical properties of H<sub>2</sub> present significant safety concerns. As the lightest

and smallest molecule, it can easily permeate through tiny gaps and rapidly diffuse into air [7]. Additionally, H<sub>2</sub> is colourless, odourless, and tasteless, making it difficult to detect without specialized sensors [5]. Its flammability range is broad (4–75 % by volume in air), and due to its low ignition energy, even small leaks can lead to catastrophic explosions if exposed to a spark or flame [8]. This makes the accurate detection of H<sub>2</sub> at low concentrations vital for safety in H<sub>2</sub> production, storage, and delivery [9].

Traditional H<sub>2</sub> sensors based on metal oxide semiconductors (MOS) exhibit high sensitivity and long-term stability; however, they typically require a

high operating temperature (200-400 °C), which increases energy consumption and limits safe deployment near flammable gases [10, 11]. Tang et al. reported a graphene-based H<sub>2</sub> sensor operating at room temperature, where UV-assisted carrier modulation and polymer molecular sieves enabled fast response (around 3 s at 5 ppm) and improved humidity resistance; however, the sensing mechanism relied on electrical readouts and complex surface modification [12]. Other materials like conducting polymers offer room-temperature operation but suffer from poor repeatability and are sensitive to humidity, limiting their reliability in practical applications [9, 13].

To overcome these limitations, two-dimensional (2D) materials such as molybdenum disulfide (MoS<sub>2</sub>) have gained attention for gas sensing applications due to their large surface-to-volume ratio, chemically active sites, and unique electronic structure [12, 14]. MoS<sub>2</sub>, especially in its semiconducting 2H phase, allows gas adsorption that alters its optical and electronic properties, making it highly suitable for optical detection [15, 16]. However, achieving uniform and low-temperature deposition of MoS<sub>2</sub> on curved or flexible surfaces remains a fabrication challenge.

Optical fibre-based sensing platforms offer several advantages for H<sub>2</sub> detection, particularly in environments where electromagnetic interference (EMI), flammability, or remote access are concerns. These sensors are passive, intrinsically safe, and immune to EMI, making them ideal for harsh or hazardous environments. Luong et al. reported an optical H<sub>2</sub> sensor based on Pd and Pd-alloy nano-hydride, where H<sub>2</sub> absorption induced measurable changes in optical transmission, achieving sub-second response times and ppm-level detection limits at room temperature, while maintaining intrinsic electrical safety [17].

In another optical approach, Pd-based thin films and nanostructures integrated with optical transducers were tested for H<sub>2</sub> sensing, relying on H<sub>2</sub>-induced modulation of optical intensity or wavelength shift,

offering remote and EMI-immune detection but often involving complex material fabrication or noble-metal coatings [18]. Side-polished POF enables evanescent field interaction with the surrounding medium, making them suitable for integration with functional layers like MoS<sub>2</sub> for optical sensing [13, 19].

In this study, MoS<sub>2</sub> thin films were deposited onto side-polished POF using chemical bath deposition (CBD), a simple and cost-effective technique well suited for optical transducer fabrication [20, 21, 22, 23]. The fabricated MoS<sub>2</sub> coated POF was tested for its optical absorbance response to varying H<sub>2</sub> concentrations at room temperature, to confirm its functionality as a H<sub>2</sub> sensor. This work investigated the feasibility of employing MoS<sub>2</sub> thin films as an active sensing layer on optical fibres to obtain insights into the underlying sensing mechanism that enables low-power, EMI-immune, and highly sensitive H<sub>2</sub> detection.

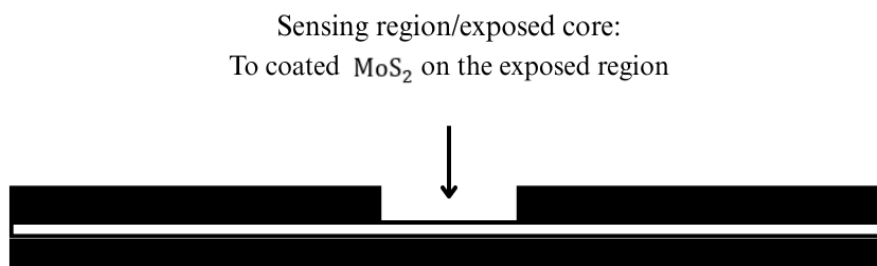
## EXPERIMENTAL

### Materials

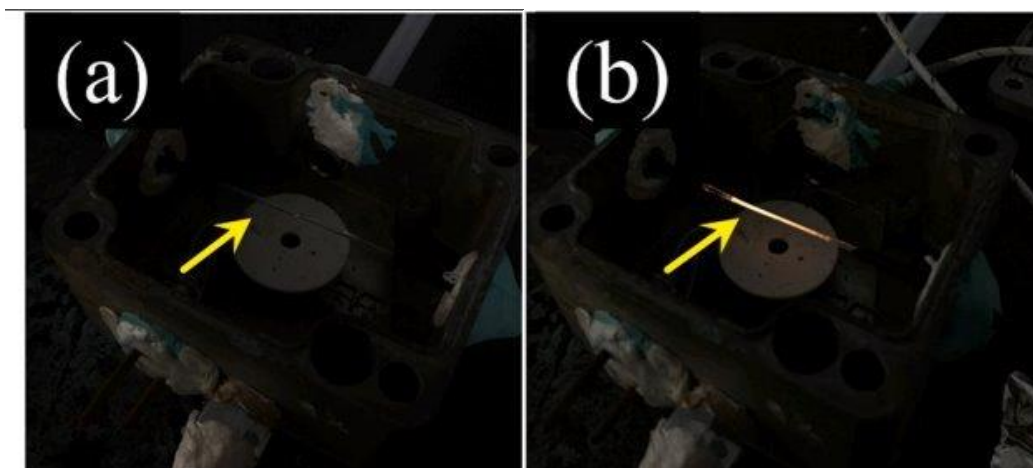
The materials used in this study included sodium sulphide (Na<sub>2</sub>S, LabServ, 96.33-97.31 % purity), sulfuric acid (H<sub>2</sub>SO<sub>4</sub>, Merck, 95-97 % purity), and ammonium heptamolybdate ((NH<sub>4</sub>)<sub>6</sub>Mo<sub>7</sub>O<sub>24</sub>, Sigma-Aldrich, Assay 81.0-83.0 % purity).

### Stage 1: Preparation of POF

POF with a core diameter of 1.0 mm and polymethyl methacrylate (PMMA) cladding were used as the optical substrate. A side-polishing process was conducted to expose the fibre core, enabling evanescent wave interaction with the sensing material, as shown in Figure 1. The cladding was partially removed over a 1.5 cm length using fine-grade sandpaper under controlled pressure to avoid damaging the core. The exposed section was cleaned using ethanol and dried at room temperature prior to coating.



**Figure 1.** Illustration of side polished POF with exposed core for the evanescent wave interaction.



**Figure 2.** Photographic comparison of light propagation in POF: (a) cladded POF and (b) uncladded POF after side-polishing, both positioned inside the gas chamber.

To validate the effectiveness of the side-polishing process, Figure 2 presents photographic evidence of light propagation within the POF positioned inside the gas chamber. Figure 2(a) shows normal POF without any modification, where light confinement within the core is maintained due to total internal reflection. In contrast, Figure 2(b) shows the POF after side-polishing, wherein partial removal of the cladding exposed the core, allowing evanescent field leakage and enabling interaction with the external medium.

This comparison shows the difference in light propagation within the core and the leaking in cladding.

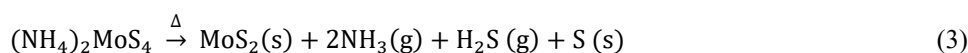
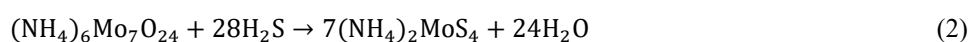
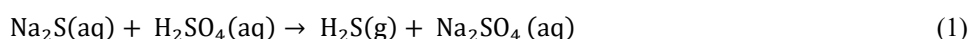
### Stage 2: Synthesis of MoS<sub>2</sub>

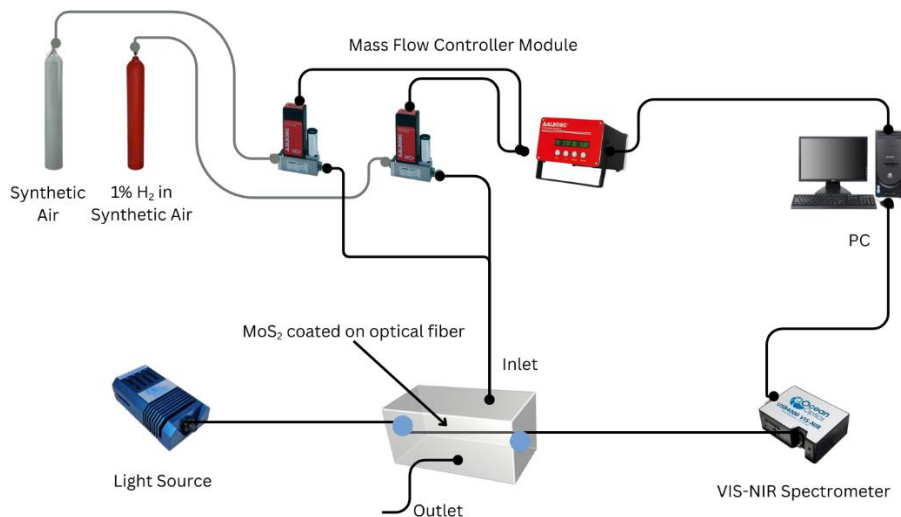
Molybdenum disulfide (MoS<sub>2</sub>) thin films were deposited using a chemical bath approach, which leverages the in-situ generation of sulphide species and their subsequent complexation with molybdenum precursors. Initially, 10 mL of sodium sulphide (Na<sub>2</sub>S, 0.2 M) was acidified with 1 mL of sulfuric acid (H<sub>2</sub>SO<sub>4</sub>, 1 M) in a fume hood, resulting in a turbid suspension attributed to the evolution of H<sub>2</sub>S gas. The introduction of 10 mL ammonium heptamolybdate ((NH<sub>4</sub>)<sub>6</sub>Mo<sub>7</sub>O<sub>24</sub>, 1 mM) facilitated the formation of

soluble thiomolybdate intermediates (e.g., MoS<sub>4</sub><sup>2-</sup>), as evidenced by the dark orange/brown solution. The reaction bath was maintained at 60 °C to enhance precursor reactivity and drive subsequent reduction-sulfurization pathways toward MoS<sub>2</sub> nucleation.

The POF substrates were side-polished and subsequently pre-cleaned with ethanol prior to MoS<sub>2</sub> deposition. Then, the substrates were vertically immersed in the bath, where heterogeneous nucleation and oriented growth of MoS<sub>2</sub> nanosheets occurred over 30 min. Upon completion, the substrates were withdrawn, rinsed with deionized water to eliminate unreacted ionic species, and dried overnight under ambient conditions, yielding adherent MoS<sub>2</sub> coatings suitable for optical sensing applications.

The synthesis began with the acidification of sodium sulfide (Na<sub>2</sub>S) using sulfuric acid (H<sub>2</sub>SO<sub>4</sub>), which generated hydrogen sulfide gas (H<sub>2</sub>S) while leaving sodium sulfate (Na<sub>2</sub>SO<sub>4</sub>) in the aqueous phase (Equation 1). The freshly formed H<sub>2</sub>S subsequently reacted under acidic conditions with molybdate species to promote sulfidation, producing the orange/brown tetrathiomolybdate (Mo – S) complex (Equation 2). This intermediate was essential, as its thermal decomposition in the final step yielded the targeted molybdenum sulfide product (Equation 3).





**Figure 3.** Schematic diagram of the optical H<sub>2</sub> sensing setup.

### Stage 3: Optical Characterization Towards H<sub>2</sub>

The POF coated with MoS<sub>2</sub> was placed inside a custom-built sealed gas chamber for sensing evaluation. A halogen light source was connected to one end of the fibre, and a spectrometer was attached to the other end to record transmitted light intensity. Optical absorbance was monitored in real time while the POF was exposed to H<sub>2</sub> gas at concentrations of 0.25 %, 0.5 %, 0.75 %, and 1.00 % in synthetic air. Each exposure cycle was followed by a recovery phase in synthetic air to evaluate sensor performance.

The sensing setup utilized the MoS<sub>2</sub>-coated POF as the sensing element, as shown in Figure 3. The system included a halogen light source, sealed gas chamber, synthetic air and H<sub>2</sub> inputs via mass flow controllers, as well as a VIS–NIR spectrometer for real-time absorbance monitoring.

## RESULTS AND DISCUSSION

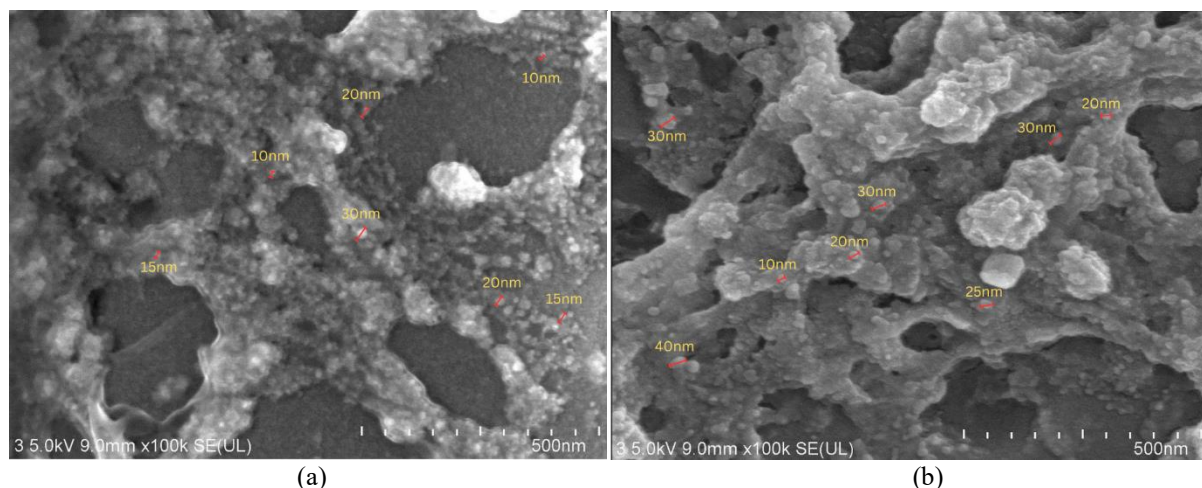
### Material Characterization

#### FESEM

Figure 4 shows FESEM micrographs of two different surface regions in the same MoS<sub>2</sub> coated sample at 100k× magnification. Both regions exhibited a porous and non-uniform network with interconnected voids, but with local variations in surface roughness and

particle distribution. In Figure 4(a), the surface was dominated by smaller clusters of nanogranules (10 - 20 nm) decorating the pore walls. Meanwhile, figure 4(b) highlights the presence of larger agglomerates and a wider range of nanogranules, from 10 to 40 nm, forming over the porous framework. Such morphological heterogeneity is typical in solution-processed or deposited MoS<sub>2</sub> films and directly affects the density of active adsorption sites available for H<sub>2</sub> interactions. Similar porous and heterogeneous MoS<sub>2</sub> morphologies with nanoscale granules and interconnected voids have been widely reported, where variations in particle size, edge density, and surface roughness were found to strongly influence the availability of active sites for H<sub>2</sub>-related surface reactions [24, 25].

Other observations have been reported in recent studies. Wu et al. observed that pristine MoS<sub>2</sub> displayed a flake-like porous structure, while Pd decoration introduced nanoparticles along the sheet edges and surfaces, modifying porosity and surface area [8]. Likewise, Ma et al. noted that Pd doping roughened the MoS<sub>2</sub> surface and introduced distinct grain boundaries compared to pristine films, which facilitated the creation of new adsorption sites for H<sub>2</sub> [26]. Kim et al. reported that noble metal nanoparticle decoration maintained the overall flake-like MoS<sub>2</sub> morphology while introducing uniformly distributed nanoclusters, thereby enhancing sensing activity through geometric and electronic effects [27].



**Figure 4.** FESEM micrographs of the MoS<sub>2</sub> coated sample at 100k times magnification showing different surface regions: (a) a porous surface with fine particle decoration along the pore walls; (b) a surface region with larger agglomerates distributed over the porous matrix.

To conclude, both images highlight the heterogeneous morphology of the same sample, which provided abundant active sites for H<sub>2</sub> adsorption.

#### EDS

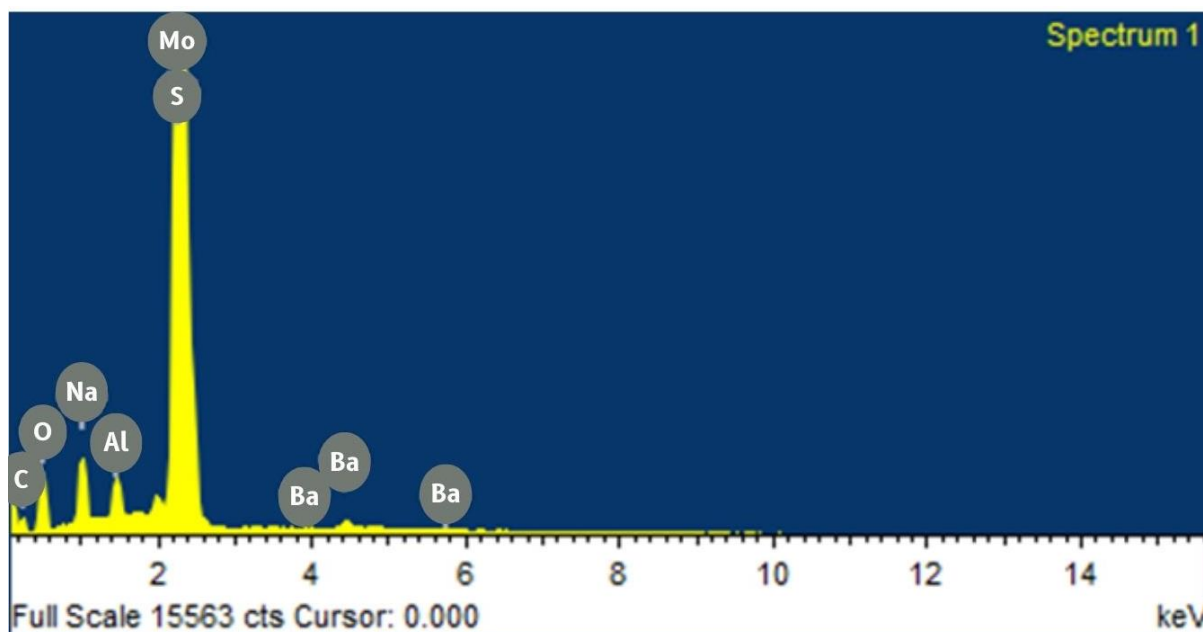
EDS analysis was conducted to evaluate the elemental composition of the MoS<sub>2</sub> sensing layer. The spectra obtained from two different surface points of the sample revealed the dominant presence of molybdenum (Mo) and sulfur (S), confirming the successful formation of MoS<sub>2</sub> on the substrate. The atomic ratio between S and Mo at both points was consistent with the ideal stoichiometry of MoS<sub>2</sub>, suggesting that the synthesis conditions were effective in promoting the

targeted compound formation, in agreement with previous studies [8, 26].

In addition to Mo and S, carbon (C) and oxygen (O) were also detected. These elements are commonly attributed to adventitious carbon from environmental exposure or to residual organic species from precursor solutions used during the deposition process [27]. The detection of minor elements such as sodium (Na) and aluminum (Al) is likely due to contributions from the glass substrate, which may contain aluminosilicate or soda-lime components. Such occurrences are frequently observed in thin film studies involving glass-supported deposition [26].

Element	Weight %	Atomic %
C K	12.68	30.28
O K	12.18	21.84
Na K	2.64	3.30
Al K	0.94	1.00
S K	37.43	33.49
Mo L	32.81	9.81
Ba L	1.32	0.28
Total	100.00	

**Figure 5.** EDS elemental composition (weight% and atomic%) of the MoS<sub>2</sub>-coated POF.



**Figure 6.** Energy-dispersive X-ray spectroscopy (EDS) spectrum of the synthesized MoS<sub>2</sub> film.

Furthermore, a low-intensity barium (Ba) signal was identified at both measurement points. This is presumed to originate from the underlying glass substrate, particularly if it contained barium-based constituents. Similar substrate-related Ba detection has been reported in EDS analyses where high accelerating voltages permit deeper X-ray interaction with the substrate material [8]. The minimal level of Ba observed was not expected to interfere with the chemical or sensing characteristics of the MoS<sub>2</sub> layer.

Overall, the EDS results confirmed the elemental composition and purity of the MoS<sub>2</sub> film, with no evidence of foreign contamination that would compromise the performance of the hydrogen sensor.

#### XRD

The XRD pattern of the synthesized MoS<sub>2</sub> nanostructures (Figure 7) showed distinct diffraction peaks at  $2\theta$  values of approximately 32.03°, 33.87°, 38.68°, 43.04°, and 48.76°. These peaks were indexed to the (100), (101), (103), (006), and (105) crystallographic planes of the hexagonal 2H-MoS<sub>2</sub> phase, in agreement with the JCPDS standard data. The presence of these well-defined peaks confirmed the successful formation of crystalline MoS<sub>2</sub> [28].

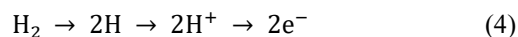
The relatively broad nature of the diffraction peaks indicates the nanostructured character of the material, which is often associated with a small

crystallite size and lattice strain. The slight deviations in peak positions compared to standard reference values may arise from internal stress, size-induced broadening, or interfacial interactions with the underlying substrate during the deposition process [28].

The XRD analysis verified that the synthesized sample was predominantly crystalline MoS<sub>2</sub> with a hexagonal structure, and the observed broadening suggests that the material possessed nanoscale features favourable for gas-sensing applications due to the increased surface-to-volume ratio.

#### Sensor Performance

The n-type semiconductor nature of MoS<sub>2</sub> originates from the availability of sulfur vacancies, which not only release free electrons but also act as active adsorption sites for gas molecules. This trait makes MoS<sub>2</sub> highly responsive to changes in the chemical environment [29]. So, when MoS<sub>2</sub> is exposed to H<sub>2</sub> at room temperature, physical adsorption and electronic doping occur simultaneously. The dissociative adsorption of H<sub>2</sub> forms S–H bonds on the MoS<sub>2</sub> surface, where H<sub>2</sub> atoms serve as electron donors, as in Equation 4. This increases the carrier concentration, which raises the Fermi level and enhances n-type conductivity [16].



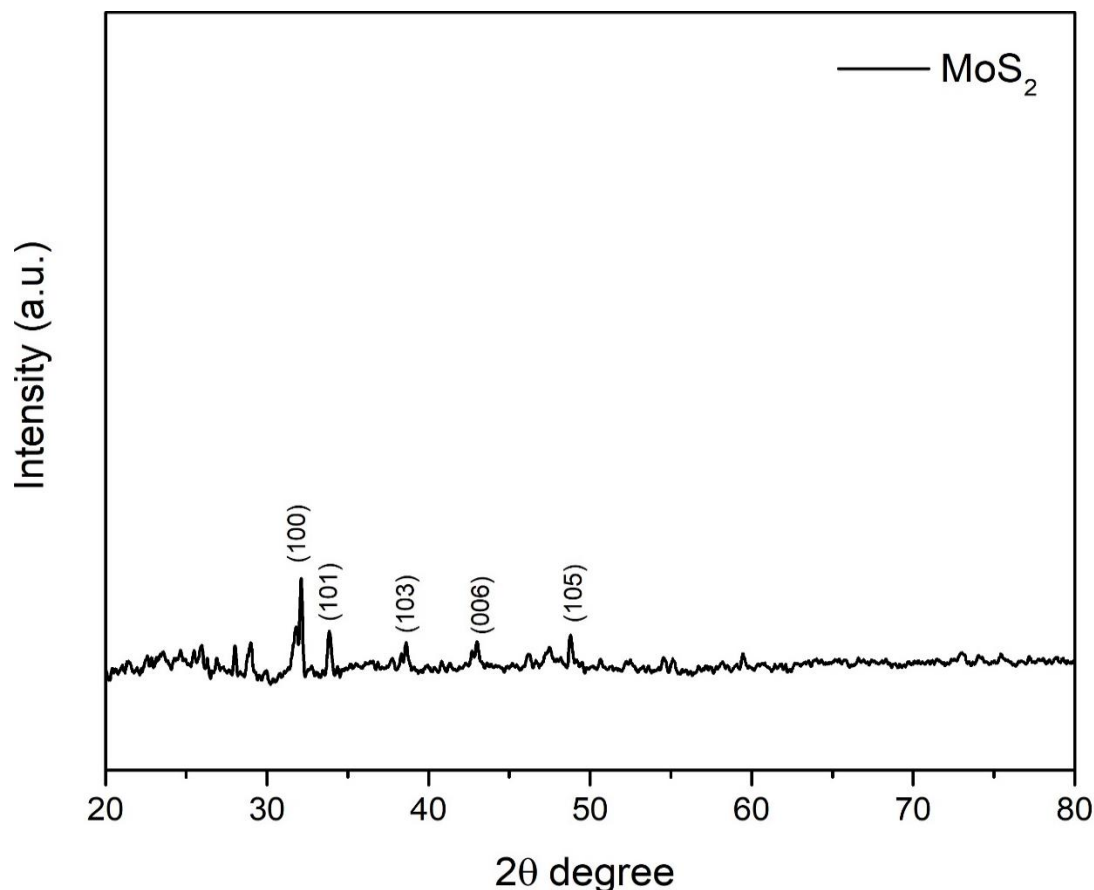


Figure 7. X-Ray diffractogram of the synthesized MoS<sub>2</sub>.

These electrons enter the conduction band of MoS<sub>2</sub>, modifying its dielectric function [30]. However, no formal redox reaction takes place at this stage. Molybdenum remains in the Mo<sup>+4</sup> oxidation state as the electron donation only happens at the surface. Instead of permanent chemical conversion, H<sub>2</sub> molecules go through physical adsorption at sulfur vacancies or catalytic sites, where they donate electrons to the MoS<sub>2</sub> lattice. Nevertheless, this process still modulates the electronic states and optical absorption of MoS<sub>2</sub>, providing a stable platform for sensing under ambient conditions [15].

These hydrogen-induced variations in the optical properties of MoS<sub>2</sub> can be effectively monitored through absorbance measurements. In optical fibre-based sensor configurations, the evanescent field generated by total internal reflection (TIR) interacts with the MoS<sub>2</sub> layer deposited on the exposed fibre region. Any adsorption-induced modulation in MoS<sub>2</sub> electronic states alter its absorbance, and these variations can be directly captured by the optical fibre transducer [30]. Generally, the optical characteristics are governed by the complex refractive index,  $\bar{n}$ , in Equation 5.

$$\bar{n} = n + ik \quad (5)$$

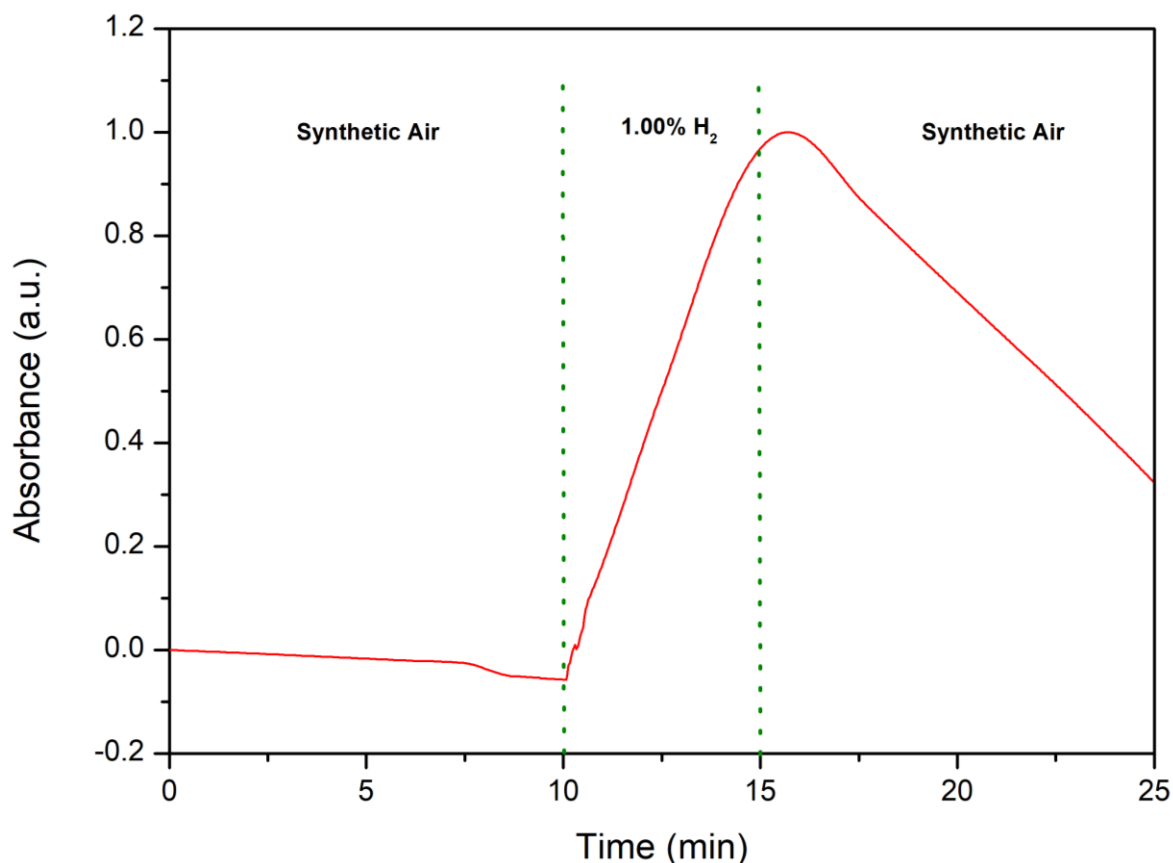
Here,  $n$  denotes the real part, governing the phase velocity of light, and  $k$  is the extinction coefficient, associated with absorption. Upon H<sub>2</sub> exposure, both  $n$  and  $k$  increase due to enhanced free-carrier interactions. This results in stronger light-matter coupling and higher optical losses. The absorption coefficient  $\alpha$  is related to  $k$  as in Equation 6.

$$\alpha = \frac{4\pi k}{\lambda} \quad (6)$$

When the extinction coefficient,  $k$ , increases due to H<sub>2</sub> exposure,  $\alpha$  also increases [31]. This means that more of the incident light is attenuated by the MoS<sub>2</sub> film due to the increased absorption coefficient. Equation 7 displays the relation between absorbance,  $A$ , with the absorption coefficient,  $\alpha$ .

$$A = \log_{10} \frac{I_0}{I} = \alpha d \quad (7)$$

where  $I_0$  is the incident light intensity,  $I$  is the transmitted light intensity and  $d$  is the thickness of the absorbing layer. As  $\alpha$  increases due to H<sub>2</sub> exposure, absorbance increases proportionally [32]. This could be observed as a higher absorbance peak in spectroscopic measurements.



**Figure 8.** Dynamic optical absorbance response of the MoS<sub>2</sub> coated POF under cyclic exposure to 1.00 % H<sub>2</sub> and synthetic air.

The corresponding graph is presented in Figure 8, which illustrates the MoS<sub>2</sub> coated fibre under 100 % synthetic air and 1 % H<sub>2</sub> exposure.

Under H<sub>2</sub> exposure, MoS<sub>2</sub> surfaces exhibited enhanced absorbance due to electron donation from dissociated H<sub>2</sub> molecules. These electrons increased the free-carrier concentration, which modified the refractive index and strengthened optical absorption. Conversely, when the environment was switched back to synthetic air (a mixture of N<sub>2</sub> and O<sub>2</sub>), the absorbance decreased toward its baseline level. This recovery indicates that once the H<sub>2</sub> exposure was removed, the sensor response gradually returned to its baseline level, demonstrating stable and repeatable performance under ambient conditions.

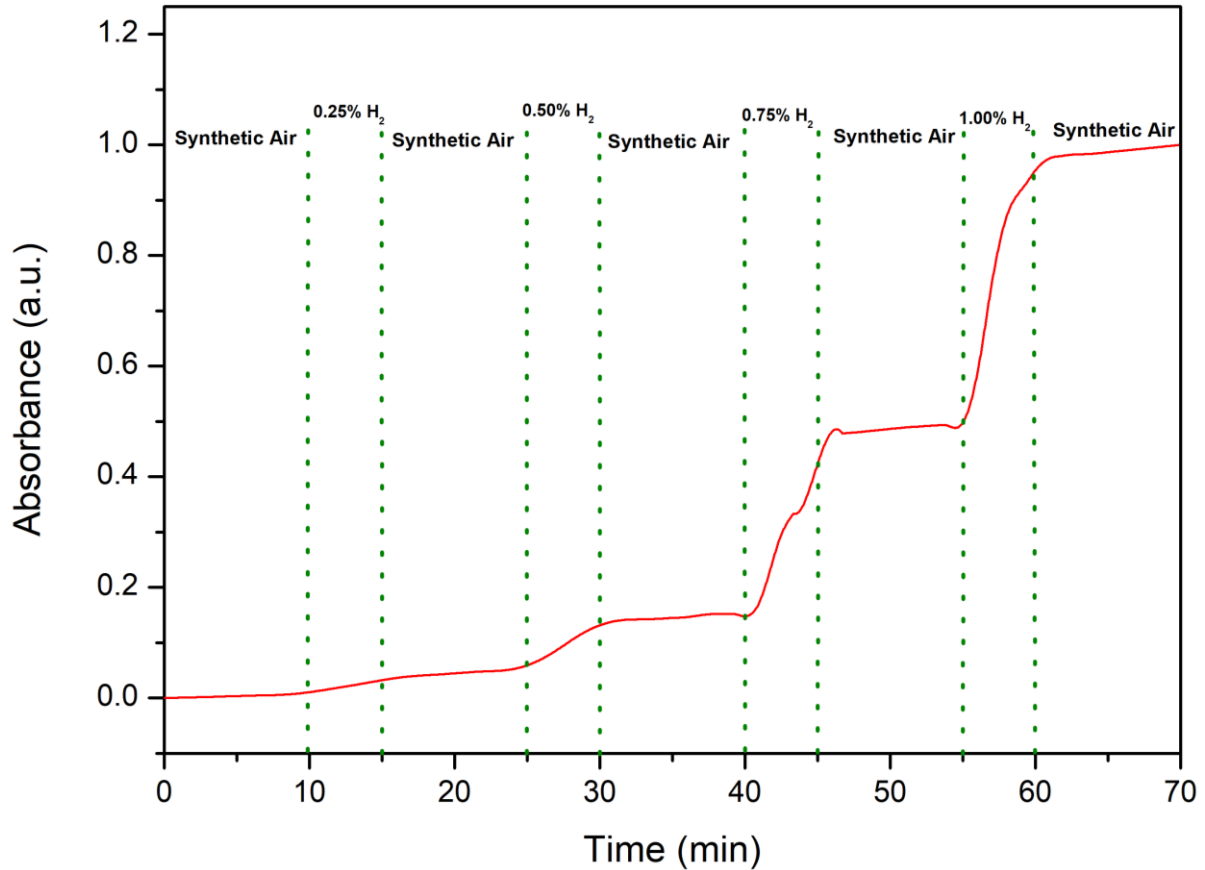
The absorbance profile in Figure 9 illustrates the sensor response over multiple exposure–recovery cycles. During each cycle, H<sub>2</sub> was introduced after baseline stabilization in synthetic air. An increase in absorbance followed by partial recovery was observed. The response pattern was repeatable but demonstrated a cumulative adsorption effect, suggesting incomplete H<sub>2</sub> desorption. This effect

became more prominent in subsequent cycles, confirming strong MoS<sub>2</sub> – H<sub>2</sub> surface interactions.

#### Recovery Limitations and Proposed Improvement Strategies

To address the observed cumulative adsorption and incomplete recovery behaviour, one promising approach is the development of MoS<sub>2</sub>-based composite sensing layers. The incorporation of secondary materials, such as noble metal catalysts or metal oxides, modifies the local electronic structure and facilitates charge redistribution. Pt-decorated MoS<sub>2</sub> has been shown to enhance H<sub>2</sub> dissociation and promote efficient carrier transfer across the MoS<sub>2</sub>/ Pt interface, resulting in improved reversibility [33].

Similarly, MoS<sub>2</sub>/WO<sub>3</sub> composite systems form heterojunctions with favourable band alignments, where charge transfer improves adsorption–desorption balance and recovery behaviour compared to pristine MoS<sub>2</sub>. These studies indicate that heterointerface-induced charge redistribution weakens excessively strong gas–surface binding and enhances reversibility during repeated sensing cycles [34].



**Figure 9.** The dynamic optical response of the sensor over full synthetic air-H<sub>2</sub>-air cycles.

In addition to material modification, post-deposition annealing treatment represents an effective strategy to improve recovery behaviour. Thermal annealing has been widely reported to remove residual solvents and surface-adsorbed water, reduce defect-related trap states, and improve crystallinity, thereby promoting faster and more reversible adsorption processes in semiconductor-based gas sensors [35].

Accordingly, future work will focus on MoS<sub>2</sub>-based nanocomposite integration and optimization of post-deposition annealing conditions to mitigate cumulative adsorption effects while preserving the

advantages of room-temperature, optical, and fibre-based H<sub>2</sub> sensing.

Figure 10 shows the cumulative absorbance variation of the sensor as a function of H<sub>2</sub> concentration in pure synthetic air: 0.25 % (2500 ppm), 0.5 % (5000 ppm), 0.75 % (7500 ppm), and 1.0 % (10000 ppm). A clear linear relationship was observed, as described by the regression equation  $y = 0.3084x + 0.0017$  with a correlation coefficient  $R^2 = 0.9476$ . The slope of the calibration curve indicated an optical sensitivity of 0.3084 a.u./% H<sub>2</sub>, demonstrating that the sensor quantitatively detected H<sub>2</sub> concentrations within the tested range with good linearity.

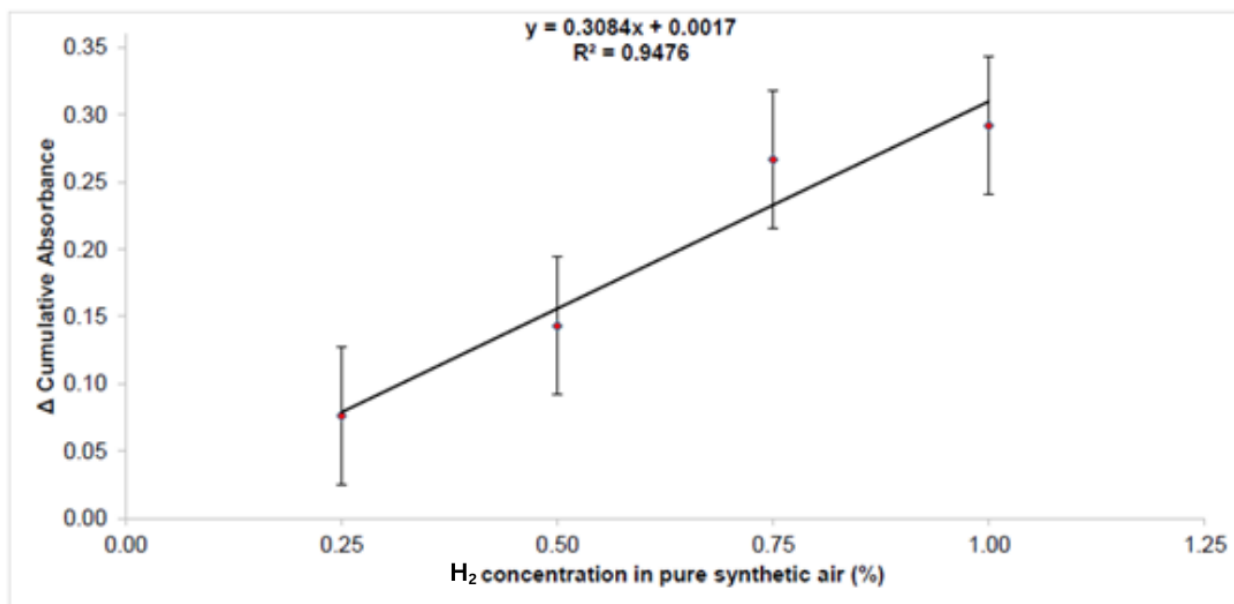


Figure 10. Cumulative absorbance changes vs H<sub>2</sub> concentration.

## CONCLUSION

In this study, an optical H<sub>2</sub> sensor was successfully developed by integrating chemically synthesized MoS<sub>2</sub> thin films with a side-polished POF. The MoS<sub>2</sub> coating was deposited using a simple and low-temperature CBD process.

The sensor demonstrated a clear increase in optical absorbance upon exposure to H<sub>2</sub>, with a linear relationship between absorbance and H<sub>2</sub> concentration in the range of 0.25 - 1.00 %. The optical sensitivity was determined to be 0.3084 a.u./% H<sub>2</sub> with a strong linearity ( $R^2 = 0.9476$ ), indicating reliable quantitative detection across the tested concentration range. Dynamic optical response revealed repeatable sensor responses across multiple exposure and recovery cycles, though with partial recovery due to cumulative adsorption effects. It was observed that at 0.25 % H<sub>2</sub> concentration, the cumulative absorbance increased by 0.0217 a.u. In contrast, at 1 % H<sub>2</sub>, the cumulative absorbance showed a much larger increase of 0.4558 a.u. This indicates that higher H<sub>2</sub> concentrations resulted in a stronger absorbance response.

Material characterization confirmed the successful formation of crystalline MoS<sub>2</sub> with appropriate morphology and composition. The sensing mechanism was attributed to surface adsorption of H<sub>2</sub> molecules, electron donation to the MoS<sub>2</sub> layer, and resulting changes in its electronic and optical properties.

This work demonstrates the potential of using MoS<sub>2</sub> coated optical fibres for compact, low-power,

and EMI-immune H<sub>2</sub> sensing platforms. Further improvements in recovery behaviour and selectivity may pave the way for real-time monitoring applications in H<sub>2</sub> energy systems and industrial safety settings.

## ACKNOWLEDGEMENTS

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