

Effect of Pt Loading on the Colorimetric Hydrogen-Sensing Properties of MoO₃ Nanorods

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ABSTRACT: Colorimetric hydrogen sensors enable simple visual detection of H₂ leakage without electrical signal readout. In this study, Pt-decorated MoO₃ nanorods were prepared, and the effect of Pt loading on their colorimetric H₂-sensing characteristics was investigated. Pt decoration enabled a clear H₂-induced color change at room temperature, while increasing the Pt loading accelerated the response but decreased the color difference. These contrasting effects of Pt loading were discussed based on the colorimetric sensing mechanism and the catalytic and optical roles of Pt. Considering both color difference and response speed, 3 wt% Pt-MoO₃ was selected for subsequent sensing evaluation. It exhibited H₂ selectivity, humidity tolerance, and sensor-to-sensor reproducibility and successfully detected H₂ leakage visually in a gas-line leakage test. These results demonstrate the importance of Pt loading in controlling the colorimetric H₂-sensing characteristics of Pt-MoO₃ and provide useful guidance for the development of colorimetric H₂ sensors.

KEYWORDS: *Colorimetric sensor, Hydrogen detection, MoO₃ nanorods, Pt loading, Spillover*

1. INTRODUCTION

Hydrogen is receiving increasing attention as a clean energy carrier because of its high gravimetric energy density and carbon-free utilization [1]. However, its wide flammability range, low ignition energy, and rapid diffusion pose safety risks during production, storage, transportation, and utilization. Reliable H₂ detection is therefore required for the safe use of hydrogen-based energy systems. Various H₂ sensing technologies have been developed, including chemiresistive, electrochemical, catalytic combustion, and optical sensors [2,3]. Among them, colorimetric sensors detect H₂ through a visible color change without continuous electrical signal readout, providing a simple and intuitive indication of gas leakage [4].

Transition-metal oxides exhibiting changes in optical properties upon H₂ exposure have been investigated as

colorimetric sensing materials [5]. Among them, MoO₃ is particularly attractive because its optical properties vary with its electronic and chemical states, resulting in a clear color change in the presence of H₂ [6,7]. However, the direct interaction between H₂ and MoO₃ is kinetically limited at low temperatures. Thus, noble-metal catalysts are incorporated to promote H₂ dissociation and hydrogen spillover onto MoO₃. Pt and Pd have been widely used for this purpose [8,9]. Accordingly, Pt-decorated MoO₃ has been investigated for visual H₂ detection [10].

The one-dimensional morphology of MoO₃ nanorods (NRs) facilitates gas access and transport through the sensing material [11]. Combined with the catalytic role of Pt, these characteristics make Pt-MoO₃ NRs suitable for colorimetric H₂ sensing. Pt loading can affect both the magnitude and kinetics of the H₂-induced color change in Pt-MoO₃ NRs. Understanding these Pt-loading-dependent characteristics is therefore important for selecting the Pt content according to the required sensing performance. However, few studies have systematically investigated these characteristics in Pt-MoO₃ NRs.

In this study, Pt-decorated MoO₃ NRs with different Pt loadings were prepared and evaluated for colorimetric H₂ sensing at room temperature. The effects of Pt loading on color difference and response speed were systematically investigated. The H₂-induced coloration mechanism was

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examined based on changes in the chemical states and optical absorption of Pt-MoO₃, and the contrasting effects of Pt loading on these two sensing characteristics were discussed in relation to this mechanism. Considering both characteristics, a Pt loading of 3 wt% was selected for further evaluation. Its H₂-sensing performance was examined in terms of concentration dependence, selectivity, humidity tolerance, and sensor-to-sensor reproducibility. Finally, its applicability to visual H₂ leakage detection was evaluated using a gas-line leakage test. This study provides insight into how Pt loading influences the colorimetric sensing characteristics of Pt-MoO₃ NRs and offers a basis for controlling Pt content to meet the required sensing performance.

2. EXPERIMENTAL

2.1 Synthesis of MoO₃ NRs

MoO₃ NRs were synthesized using a hydrothermal method [12]. Molybdenum powder (2.0 g, -250 mesh, 99.9%, Thermo Fisher Scientific Inc.) was dissolved in 20 mL of H₂O₂ solution (35 wt%, Thermo Fisher Scientific Inc.) with stirring at 500 rpm for 20 min. The solution was heated at 230°C for 12 h in a 90 mL Teflon-lined autoclave. The product was washed with deionized water and ethanol by centrifugation, dried at 60°C overnight, and calcined in air at 300°C for 2 h.

2.2 Preparation of Pt-MoO₃ NRs

Pt-decorated MoO₃ NRs were prepared by a pyrolysis-assisted deposition method [13]. Chloroplatinic acid hexahydrate (0.1 g, Sigma-Aldrich) was dissolved in 2 mL of deionized water by sonication for 40 min. MoO₃ NRs (0.1 g) were dispersed in 10 mL of methanol ($\geq 99.8\%$, Thermo Fisher Scientific Inc.), and the Pt solution was added to obtain Pt loadings of 1, 3, and 5 wt%. The mixtures were stirred at 800 rpm for 24 h, freeze-dried for 12 h, and calcined in air at 300°C for 2 h. The samples were denoted as 1Pt-MoO₃, 3Pt-MoO₃, and 5Pt-MoO₃ based on Pt loading.

2.3 Characterization

The morphology and microstructure were examined by field-emission scanning electron microscopy (FE-SEM, Gemini 500, Zeiss) and high-resolution transmission electron microscopy (HR-TEM, Titan G2 Cube 60-300, FEI) equipped with energy-dispersive X-ray spectroscopy (EDS). The Pt contents were quantitatively determined by inductively coupled plasma mass spectrometry (ICP-MS, NexION-350X, PerkinElmer). The crystal structures were analyzed by X-ray

diffraction (XRD, LabX XRD-6100, Shimadzu) using Cu K α radiation ($\lambda = 0.1541$ nm). The specific surface area (SSA) was determined by Brunauer-Emmett-Teller analysis of nitrogen adsorption isotherms (BELSORP MINI X, MicrotracBEL). Surface chemical states were analyzed by X-ray photoelectron spectroscopy (XPS, Sigma Probe, Thermo VG Scientific) using an Al K α source. Optical absorption was measured using an ultraviolet-visible-near-infrared (UV-Vis-NIR) spectrophotometer (V-770, JASCO).

2.4 Sensor Fabrication

The synthesized powder (0.1 g) was mixed with polydimethylsiloxane (PDMS, Sylgard 184 Silicone Elastomer Kit, Dow Corning) at a weight ratio of 1:15 using a paste mixer (AR-100, Thinky) at 1000 rpm for 10 min. A curing agent was added at a PDMS-to-curing-agent weight ratio of 10:1, followed by mixing for 3 min. The mixture was spin-coated onto a 50 × 50 mm² glass substrate at 500 rpm for 60 s and cured at 70°C for 30 min. The colorimetric sensor was cut into 10 × 10 mm² pieces for colorimetric sensing measurements. For the H₂ leakage test, the film was cut into 10 × 50 mm² strips and used as tape-type colorimetric sensors.

2.5 Gas-Sensing Measurements

Colorimetric sensing measurements were conducted in an 8 mL quartz chamber. All analyte gases were supplied as N₂-balanced gas mixtures and further diluted with N₂ (99.999%, Hankook Special Gases) to the required concentrations. H₂ sensing was evaluated over a concentration range of 0.5–5 vol%, and selectivity was examined against CH₄ (5 vol%), NH₃ (100 ppm), acetone (5 ppm), ethanol (5 ppm), and CO (5 ppm). The total gas flow rate was fixed at 500 mL min⁻¹, and all measurements were performed at room temperature ($\sim 25^\circ\text{C}$). The quartz chamber was placed inside a dark enclosure to minimize variations in external lighting conditions. The color change of the sensor was monitored in real time using a digital microscope (HiMax Tech.) positioned approximately 3 cm above the sensor surface. Illumination inside the enclosure, camera exposure, and white-balance settings were kept constant throughout the measurements to ensure consistent image acquisition. The acquired images were processed using a custom-developed image-analysis program. The color change was quantified using the color difference (ΔE) in the International Commission on Illumination (CIE) L*a*b* color space according to the following equation:

$$\Delta E = \sqrt{(L - L')^2 + (a - a')^2 + (b - b')^2} \quad (1)$$

where L , a , and b represent lightness, the green–red coordinate, and the blue–yellow coordinate before gas exposure, respectively, and L' , a' , and b' represent the corresponding values after gas exposure. A ΔE value of 3 has been reported to represent a clearly visible color difference [14]. Accordingly, the response time was defined as the time required for ΔE to reach 3 after gas exposure.

3. RESULTS AND DISCUSSION

3.1 Characterization of Pure MoO₃ and Pt-MoO₃ NRs

The morphologies of pure MoO₃ and Pt-MoO₃ NRs were examined by SEM (Fig. 1(a)). All samples exhibited similar one-dimensional NR morphologies, with no noticeable changes as Pt loading increased. TEM analysis of 3Pt-MoO₃

revealed nanoparticles attached to the MoO₃ NR surfaces (Fig. 1(b)). HR-TEM analysis revealed lattice fringes with interplanar spacings of 0.35 and 0.20 nm, corresponding to the (040) plane of orthorhombic MoO₃ [15] and the (200) plane of metallic Pt [16], respectively (Fig. 1(c)). EDS elemental mapping further showed the distribution of Pt over the MoO₃ NR surfaces (Fig. 1(d)). ICP-MS analysis determined Pt contents of 0.873, 3.24, and 4.58 wt% for 1Pt-MoO₃, 3Pt-MoO₃, and 5Pt-MoO₃, respectively. These results confirm the successful decoration of metallic Pt nanoparticles on the MoO₃ NRs.

XRD patterns showed that all samples exhibited the orthorhombic MoO₃ phase (JCPDS #05-0508) (Fig. 1(e)), consistent with the HR-TEM results. No noticeable shifts in the major diffraction peaks were observed with increasing Pt loading, suggesting that Pt was not incorporated into the MoO₃ lattice. No diffraction peaks corresponding to Pt were

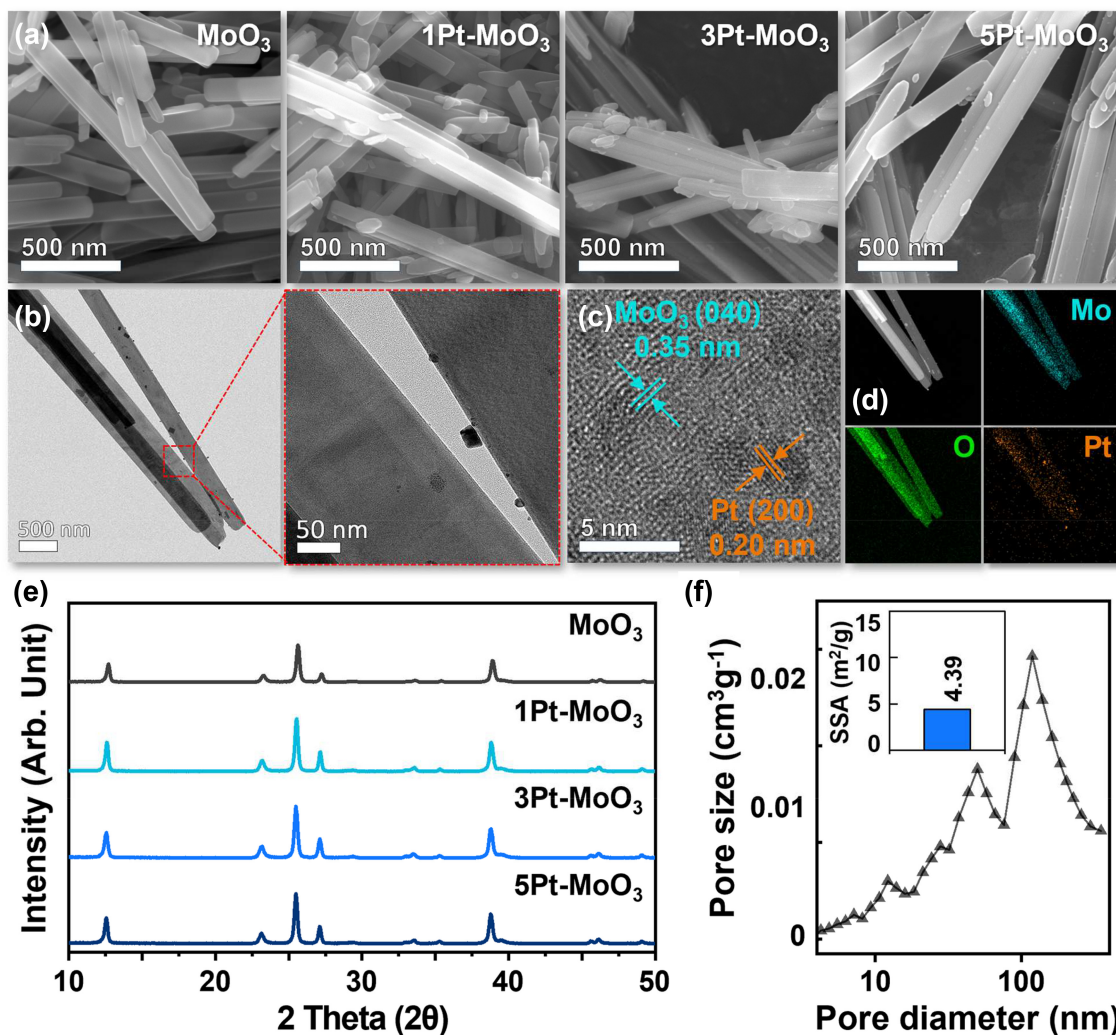


Fig. 1. Structural and morphological characterization of Pt-MoO₃ NRs. (a) FE-SEM images of MoO₃, 1Pt-MoO₃, 3Pt-MoO₃, and 5Pt-MoO₃. (b) TEM, (c) HR-TEM, and (d) EDS elemental mapping images of 3Pt-MoO₃ NRs. (e) XRD patterns of all samples. (f) Pore size distribution and SSA (inset) of 3Pt-MoO₃ NRs.

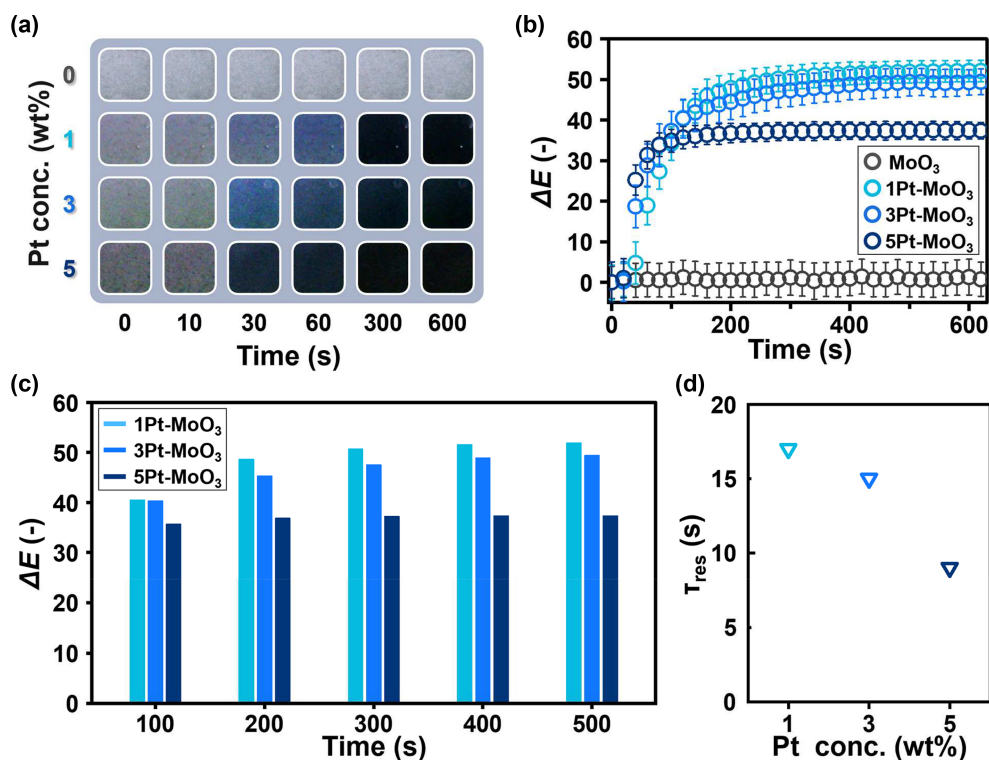


Fig. 2. Colorimetric responses of Pt-MoO₃ NRs to 5 vol% H₂ at room temperature. (a) Time-dependent optical images. (b) Time-resolved changes in ΔE . (c) ΔE values at selected exposure times. (d) Response time (τ_{res}) as a function of Pt loading.

observed, likely due to the low Pt content. These results indicate that Pt decoration did not alter the crystal structure of the MoO₃ NRs.

Pore-size analysis of 3Pt-MoO₃ revealed pores spanning a broad size range from several to tens of nanometers (Fig. 1(f)), which can be attributed to void spaces formed between the assembled NRs. The SSA of 3Pt-MoO₃ was 4.39 m² g⁻¹ (inset of Fig. 1(f)). These inter-rod void spaces provide pathways for gas transport through the sensing material, which is favorable for gas sensing.

3.2 Colorimetric Hydrogen Sensing Properties of Pt-MoO₃ Sensors

The colorimetric H₂-sensing properties of pure MoO₃ and Pt-MoO₃ sensors were evaluated using 5 vol% H₂ at room temperature. Pure MoO₃ initially appeared gray and showed little color change even after H₂ exposure for more than 10 min (Fig. 2(a)). In contrast, all Pt-MoO₃ sensors exhibited a clear color change to dark navy upon H₂ exposure and did not return to their original color after H₂ removal, demonstrating that Pt decoration is essential for the colorimetric H₂ response. The initial color of the Pt-MoO₃ sensors became progressively darker with increasing Pt loading.

The color changes were quantitatively evaluated using the

time-dependent ΔE (Fig. 2(b)). The results revealed contrasting effects of Pt loading on the magnitude and rate of the color change. The ΔE decreased in the order of 1Pt-MoO₃, 3Pt-MoO₃, and 5Pt-MoO₃ over the measured exposure times (Fig. 2(c)), indicating that higher Pt loading resulted in a smaller color difference.

In contrast, the response time decreased with increasing Pt loading (Fig. 2(d)). Based on the time required to reach $\Delta E = 3$, the response time decreased from approximately 17 s for 1Pt-MoO₃ to 15 and 9 s for 3Pt-MoO₃ and 5Pt-MoO₃, respectively. Thus, increasing Pt loading resulted in a smaller color difference but a faster response, demonstrating its contrasting effects on the colorimetric sensing characteristics of Pt-MoO₃.

3.3 Colorimetric Sensing Mechanism of Pt-MoO₃ Sensors

To examine the H₂-induced coloration mechanism of Pt-MoO₃, the surface chemical states and optical properties of 3Pt-MoO₃ were analyzed before and after H₂ exposure. The Mo 3d XPS spectrum [17,18] of the pristine sample consisted of Mo⁶⁺ and Mo⁵⁺ components with relative fractions of 66.6 and 33.4%, respectively (Fig. 3(a)). The O 1s spectrum [19] showed lattice oxygen (O_L, 81.8%), oxygen vacancies (O_v,

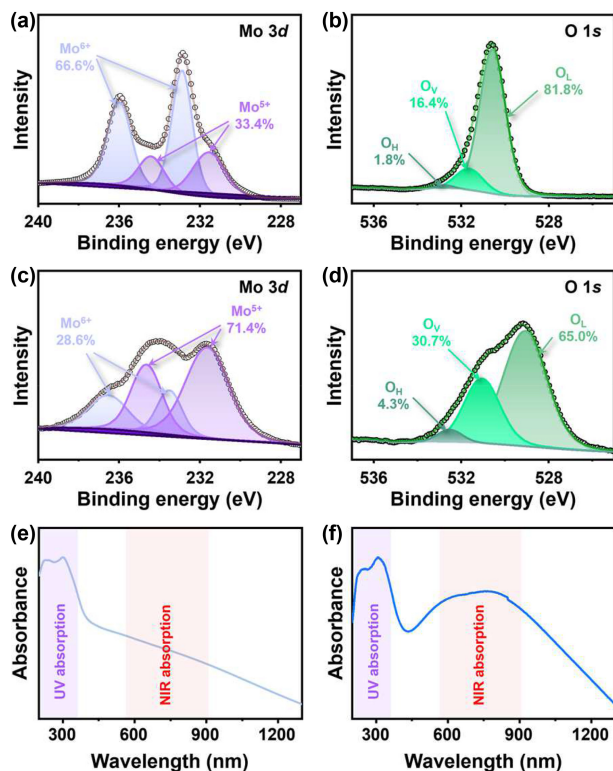


Fig. 3. Surface chemical and optical analyses of 3Pt-MoO₃ NRs. (a, b) Mo 3d and O 1s XPS spectra before H₂ exposure. (c, d) Mo 3d and O 1s XPS spectra after H₂ exposure. UV-Vis-NIR absorbance spectra (e) before and (f) after exposure to 5 vol% H₂.

16.4%), and surface hydroxyl

species (O_H, 1.8%) (Fig. 3(b)). Upon H₂ exposure, the Mo⁵⁺ fraction increased to 71.4%, while the Mo⁶⁺ fraction decreased to 28.6% (Fig. 3(c)). Concurrently, the O_V and O_H fractions increased to 30.7 and 4.3%, respectively, while the O_L fraction decreased to 65.0% (Fig. 3(d)). These changes indicate that H₂ exposure reduces MoO₃, accompanied by increases in oxygen vacancies and surface hydroxyl species.

The H₂-induced chemical changes were accompanied by a pronounced change in optical absorption. Compared with the pristine sample (Fig. 3(e)), the H₂-exposed sample showed substantially increased absorption across the visible and NIR regions (Fig. 3(f)). This increase can be attributed to reduced Mo species and to defect-related electronic states formed upon H₂ exposure. In particular, intervalence charge transfer between adjacent Mo⁵⁺ and Mo⁶⁺ species can contribute to absorption in the visible region [20], accounting for the characteristic navy coloration. Taken together, the XPS and optical results indicate that H₂ exposure reduces MoO₃, increasing Mo⁵⁺ and oxygen vacancies and consequently altering its electronic structure and optical absorption. These changes produce the colorimetric H₂ response of Pt-MoO₃.

Table 1. Colorimetric H₂-sensing characteristics of MoO₃ NRs with different Pt loadings.

No.	Material	H ₂ conc. (vol. %)	Response time (s)	Maximum ΔE
1	MoO ₃	5	-	-
2	1Pt-MoO ₃	5	17	51.9
3	3Pt-MoO ₃	5	15	49.4
4	5Pt-MoO ₃	5	9	37.3

Pt nanoparticles facilitate this process by promoting H₂ activation at room temperature [21]. Because direct H₂ activation on MoO₃ is limited, Pt promotes H₂ dissociation and the transfer of activated hydrogen species to the MoO₃ surface through hydrogen spillover [8]. This accounts for the negligible color change of pure MoO₃ and the pronounced coloration of Pt-MoO₃ upon H₂ exposure. The faster response with increasing Pt loading can therefore be reasonably attributed to enhanced H₂ activation and subsequent hydrogen transfer to MoO₃.

In contrast, increasing Pt loading decreased ΔE . As shown in Fig. 2(a), the initial color became progressively grayer with increasing Pt loading. This change is likely associated with the optical contribution of Pt nanoparticles [22], as it differs from the characteristic navy coloration induced by H₂ exposure. The darker initial color reduces the optical contrast between the initial and H₂-exposed states, resulting in a smaller ΔE at higher Pt loadings. Therefore, increasing Pt loading accelerates the colorimetric response but decreases the resulting color difference. Considering these contrasting effects, 3Pt-MoO₃ was selected for further evaluation because it retained a relatively large ΔE while exhibiting a shorter response time than 1Pt-MoO₃ (Table 1).

3.4 Gas Sensing Performance of the 3Pt-MoO₃ Sensor

The H₂-sensing performance of the selected 3Pt-MoO₃ sensor was further evaluated under various conditions. Fig. 4(a) shows the time-dependent ΔE as the H₂ concentration was decreased from 5 to 0.5 vol%. As the H₂ concentration decreased, the color change became smaller and slower. Nevertheless, at 0.5 vol% H₂, ΔE gradually exceeded the visible color-difference criterion ($\Delta E = 3$) upon prolonged exposure. This demonstrates that 0.5 vol% H₂ can be visually detected after prolonged exposure.

The selectivity of the 3Pt-MoO₃ sensor was evaluated against CH₄, NH₃, acetone, ethanol, and CO (Fig. 4(b)). H₂ produced a pronounced color change, whereas all interfering gases showed only minor changes, even after prolonged exposure. The ΔE values of the interfering gases remained

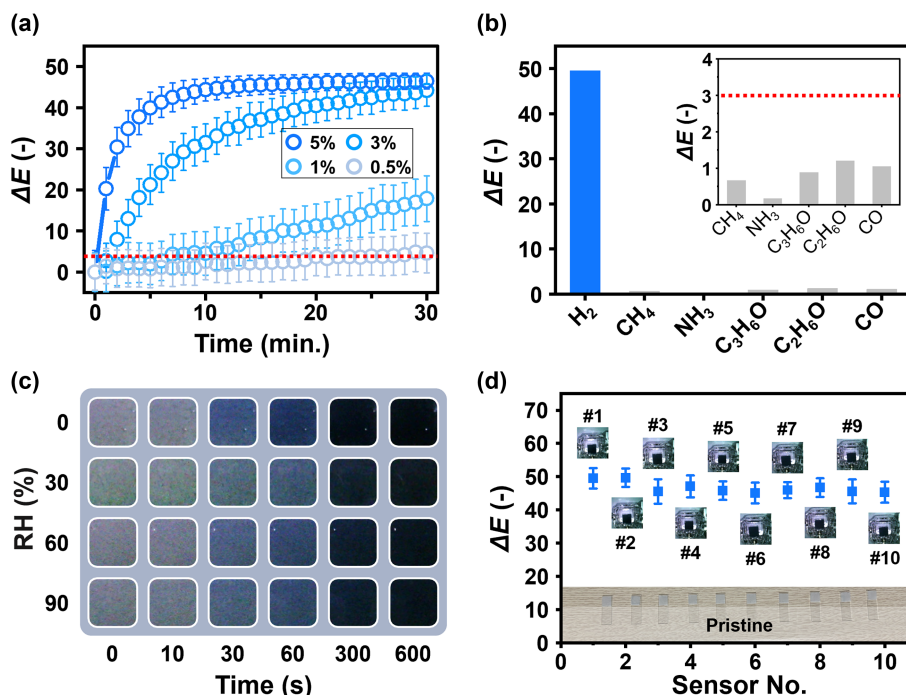


Fig. 4. Colorimetric H₂ sensing performance of 3Pt-MoO₃ NRs. (a) Time-resolved ΔE changes at 0.5–5 vol% H₂. (b) Selectivity against interfering gases. (c) Time-dependent optical images under 5 vol% H₂ at RH 0–90%. (d) Sensor-to-sensor reproducibility under 5 vol% H₂.

below 3, demonstrating that the visible color change was highly selective to H₂ under the tested conditions.

The effect of relative humidity (RH) on the H₂ response was examined over an RH range of 0–90% (Fig. 4(c)). The sensor exhibited comparable color changes upon H₂ exposure across the tested RH range, showing that its colorimetric response was not significantly affected by humidity. This humidity tolerance is favorable for H₂ detection under varying environmental conditions.

Finally, sensor-to-sensor reproducibility was evaluated using ten 3Pt-MoO₃ sensor pieces (Fig. 4(d)). All ten sensors exhibited similar ΔE values and visually consistent color changes upon H₂ exposure. The small variation among the sensors confirms the reproducibility of the fabricated colorimetric sensors. Collectively, these results demonstrate that 3Pt-MoO₃ maintains reliable colorimetric H₂-sensing performance across different H₂ concentrations, interfering gases, humidity conditions, and individual sensor units.

3.5 H₂ Leakage Detection Using the 3Pt-MoO₃ Colorimetric Sensor

The practical applicability of the 3Pt-MoO₃ sensor was examined using a simple H₂ leakage test (Fig. 5). The sensor film was attached near a gas-line fitting, which was slightly loosened to create a controlled leakage point, and 5 vol% H₂ was subsequently supplied through the gas line. A commercial

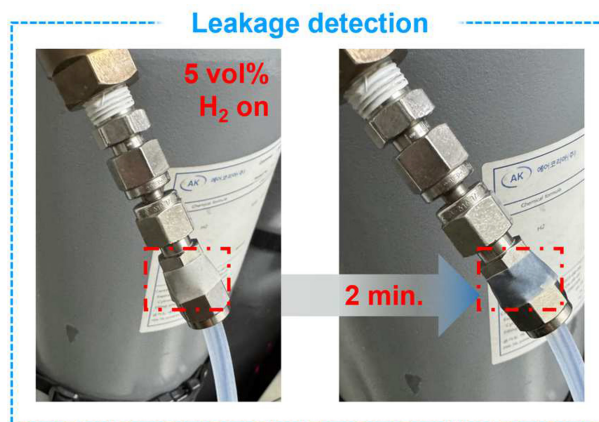


Fig. 5. Demonstration of visual H₂ leakage detection using a 3Pt-MoO₃ sensor before and 2 min after 5 vol% H₂ leakage from a gas-line fitting.

H₂ sensor was used to monitor the surrounding

H₂ concentration and ensure safety during the experiment. Before H₂ exposure, the sensor appeared gray. Upon leakage from the fitting, the sensor region near the leakage point changed to dark navy within 2 min, allowing the leakage to be readily identified by visual inspection without additional signal readout. This demonstration shows that the 3Pt-MoO₃ sensor can be directly attached near potential leakage points in H₂ gas lines to provide a simple visual indication of leakage.

4. CONCLUSIONS

Pt-decorated MoO₃ NRs with different Pt loadings were investigated for colorimetric H₂ sensing at room temperature. Pt decoration was essential for inducing a clear color change upon H₂ exposure. Increasing the Pt loading accelerated the response but decreased ΔE , showing contrasting effects on response kinetics and color difference. XPS and UV-Vis-NIR analyses indicated that the colorimetric response originates from H₂-induced reduction of MoO₃, accompanied by increased Mo⁵⁺ and oxygen vacancies. The faster response at higher Pt loadings was attributed to enhanced H₂ dissociation and hydrogen spillover by Pt, whereas the decrease in color difference was associated with the darker initial color resulting from the optical contribution of Pt nanoparticles. Considering both color difference and response time, 3Pt-MoO₃ was selected for further evaluation. It exhibited H₂ selectivity, humidity tolerance, and sensor-to-sensor reproducibility and also enabled visual detection of H₂ leakage in a gas-line leakage test. Overall, these results demonstrate the importance of Pt loading in determining the colorimetric sensing characteristics of Pt-MoO₃ and provide useful guidance for developing colorimetric sensors for visual H₂ leakage detection.

CRedit Authorship Contribution Statement

Seungwoo Park: Investigation, Methodology, Writing - original draft. **Nguyen Thao Trinh Do:** Formal analysis, Visualization. **Ji-Wook Yoon:** Writing - review & editing, Supervision.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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