



A low-cost colorimetric HKUST-1 sensor for ppm-level humidity detection

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ARTICLE INFO

Keywords:

HKUST-1
Colorimetry
Humidity
Gas sensor
MOF

ABSTRACT

Accurate detection of trace moisture is critical in various industries, from microelectronics to petrochemicals. While electrical humidity sensors are prevalent, optical methods offer superior sensitivity for low relative humidity (RH) detection, particularly in demanding applications requiring ppb sensitivity in harsh environments. This work explores the potential of HKUST-1, a metal-organic framework (MOF), as a colorimetric humidity sensor for ppm level detection across a broad range (0–80 % RH). By spraying HKUST-1 onto polyester substrates and coupling it with a low-cost, commercially available LED-photodiode module, we developed a simple, portable sensing platform. The observed color transitions, from dark blue to turquoise, correlate with the MOF's moisture adsorption mechanism, achieving a detection limit of 18 ppm H₂O (< 0.05 % RH). Comprehensive characterization, including interferent gas studies, revealed significant color changes upon exposure to NH₃, CO, and ethanol, while CO₂, CH₄, and N₂O showed minimal interference. One-week stability tests demonstrated consistent sensor performance and full reversibility upon exposure to dry air at room temperature. This low-cost, sensitive, and easily fabricated HKUST-1-based sensor offers a promising alternative for humidity monitoring in diverse industrial applications.

1. Introduction

Low relative humidity (ppm) sensors are at the heart of many industries, from high-density integrated circuits to natural gas petrochemicals, where economic profits are directly impacted by moisture contamination [1]. Portable monitoring solutions predominantly rely on electrical sensors (capacitance, resistance/impedance) [2,3]. However, converting absorbed water molecules into electrical signals often exhibits lower sensitivity than acoustic and optical methods [4]. Current applications, such as transformer oil (5–25 ppm), plutonium trays (4–30 ppm) or hydrogen for cooling generator rotors (< 10 ppm), demand sensors capable of reaching the ppb regime. These applications often involve harsh environments with high temperatures and pressures. While several stable and sensitive low-humidity sensors exist (Table 1), each presents unique advantages and disadvantages [4]. Expanding the range of available sensor technologies is therefore essential to promote affordability and widespread use. This requires research focused on developing new approaches and materials.

Metal-Organic Frameworks (MOFs) are growing recognition as gas

sensing material redefining the standards of precision, sensitivity, and selectivity [5,6]. Metal-Organic Frameworks constitute a high porosity periodic structure formed by ligands coordinated with transition metals throughout one, two, or three dimensions. The versatility of MOFs to satisfy diverse applications depends on ligand size, functional groups, and metal centers [7]. Given that, properties like pore size, reactivity, conductivity, or color could be easily tailored. For instance, Liu et al. [8], incorporated defects and switched the metal centers of a two-dimensional conductive MOF to reduce response-recovery time during moisture measurements.

In contrast to other organic materials, MOFs like metal oxide sensors [9], exhibit remarkable resistance to temperature and long-term stability. Yaghi et al. justified that with the strong bonds of the MOF skeleton (C-C, C-H and M-O) [7]. Differing from metal oxides, the unattainable porosity and surface area of MOFs [10] could improve limits of detection and response time as well as selectivity through chemical reactions [6]. Using various MOFs, Qin et al. [11] designed a microbalance electronic nose to measure o-p-m xylene mixtures with a limit of detection of 1 ppm. In a different approach, Davey et al. combined ZIF-8

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<https://doi.org/10.1016/j.snb.2025.137973>

Received 22 February 2025; Received in revised form 5 May 2025; Accepted 13 May 2025

Available online 14 May 2025

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with a dye to detect static, visible color shifts at hundreds of ppm of CO₂ [12].

Multiple properties of MOFs, such as luminescence, refractive index, magnetism, resistance are reported to shift in the presence of a gas [12–14]. However, compared to these, the colorimetric properties of MOFs have been barely explored [6]. A colorimetric sensor consists of a material that alters its color in the presence of a targeted gas - in the visible range. Leveraging smartphones cameras [15,16] and off-the-shell components [17] this class of sensor has demonstrated to be an inexpensive, user-friendly, and portable option for manufactory and packaging control [18]. The d-d transitions of the metal and the chromophore groups of the ligands frequently result in a range of colorful MOFs. Gases can interact with unoccupied metal sites or these groups, inducing a notable color change [6]. Setting a significant milestone in this field, HKUST-1 stands as the pioneering MOF that alone (without a dye) undergoes a color change in moisture presence [19].

HKUST-1, also named Cu₃(BTC)₂ or MOF-199, is built by binuclear copper-clusters coordinated via four carboxylate groups of the organic linker to form a paddle-wheel unit. Its unit cell is defined by an array of 32 Cu²⁺ ... Cu²⁺ paddlewheels connected in three dimensions by 1,3,5-benzene tricarboxylate (BTC) [20]. It is one of the most studied MOFs and it is already produced on an industrial scale by BASF SE. Building upon research, HKUST-1 is reported to turn from dark blue to turquoise in between 0 % and 10 % of relative humidity [19]. Nevertheless, its interactions with other gases are not critically questioned and HKUST-1 applicability demands an extensive investigation to achieve efficient monitoring.

The cutting-edge sensing capabilities of Metal-Organic Frameworks (MOFs) have opened new horizons in the realm of colorimetric gas detection, promising superior sensitivity, selectivity, and stability for the precise monitoring and detection of a wide array of gases, thereby revolutionizing the landscape of affordable gas sensing technologies. In this work, we identify the limits and potential of HKUST-1 as colorimetry ppm levels monitoring humidity alternative. This material promises to become a cheap low relative humidity sensor. Technical requirements

in its manufacturing are not so rigorous, so its mass production could devalue to a few euros the price of a device. The response of HKUST-1 covered not only ppm level, but a wide range from 0 % to 80 %, enabling this material suitable for many applications. The behavior of the material during H₂O streams was totally reversible at room temperature and without further activation elements than dry air. We carried out a comprehensive characterization, including the assessment of interferents such as CH₄, CH₂O, CO₂, ethanol, CO, N₂O and NH₃. We observed both reversible and irreversible behaviors during the cross-sensing experiments. So, the affinity of HKUST-1 for water in compared to these gases was discussed. Finally, we conducted long-term experiments to identify the practical performance.

2. Experimental section

2.1. Synthesis of HKUST-1

HKUST-1 was synthesized via a ball-milling method using liquid-assisted grinding in a Planetary Micro Mill PULVERISETTE 7, operating at room temperature. We adapted the synthesis procedure proposed by Timothy Steenhaut et al. [26]. Specifically, 0.59 g of copper(II) acetate monohydrate (98 %, Alfa Aesar) and 0.41 g of trimesic acid (98 %, Alfa Aesar) were placed into a 15 mL grinding container along with ten zirconium balls (5 mm diameter). Subsequently, 7.5 mL of ethanol (≥ 99.5 %, Sigma-Aldrich) was added to the mixture. The reaction proceeded through the following sequence: four cycles of 5 min each at 600 rpm in one direction, with 2-min pauses between cycles, followed by four identical cycles in the opposite direction. The resulting product was washed with 50 mL of a 1:1H₂O/ethanol solution by stirring and then filtered. To obtain a HKUST-1 paste suitable for spraying, the sample was sonicated for 5 min after the addition of 15 mL of ethanol.

2.2. Characterization of HKUST-1

The crystal structure was confirmed by X-ray diffraction

Table 1
Sensing performance of low humidity sensors reported in literature and this work.

Sensitive Element	Principle	Instrumentation	Ranges	LoD*	Response time	Interferences questioned (+/-)	Longer experiment Shown	Sources
[Eu(BTC)]-MOF	Capacitance	LCR Meter	10–2000 ppm (0.03–6.36 % RH) **	0.84 ppm (0.003 % RH) **(+) , 10 ppm (0.032 % RH) **(-)	270 s (0–100 ppm)	100–2000 ppm of CH ₃ OH ⁽⁺⁾ , ethanol ⁽⁺⁾ , Acetonitrile ⁽⁻⁾ , Acetone ⁽⁻⁾ , Toluene ⁽⁻⁾ , Hexane ⁽⁻⁾	4 weeks	[21]
Nickel (II) Iodide on Al ₂ O ₃	Impedance	Impedance Analyzer	0.4–11 % RH; 11 – 75 % RH	1 % RH ⁽⁻⁾	Not Specified	600 ppm of CH ₃ OH ⁽⁻⁾ , ethanol ⁽⁻⁾ , Acetone ⁽⁻⁾ , Formaldehyde ⁽⁻⁾	90 min	[22]
Polypyrrole	Quartz Crystal Microbalance (Mass load)	Ultra-high Frequency Counter	15–900 ppm; 900–5413 ppm (0.048–2.86 %; 2.86–17.15 % RH) **	51.1 ppm (0.16 % RH) ⁽⁻⁾	48 s (0–510 ppm)	Not Specified	30 min	[23]
PANi@PVB	Surface Acoustic waves (Mass, Viscoelastic and Electroacoustic load)	Frequency Counter	0–90 % RH	0.02 % RH ⁽⁺⁾ , 139 ppm (0.45 % RH) **(-)	1 s (11–98 % RH)	Temperature ⁽⁺⁾	5 h 7 months stored	[24]
BME 280***	Unknown	ESP32	0–100 % RH	3 % RH ⁽⁻⁾	1 s (63 % response)	Temperature ⁽⁺⁾ ****	-	[25]
HKUST-1	Optic/Colorimetric	Off-the-shelf LEDs-Photodetector	20–150 ppm (0.048–0.48 % RH)**; 5–80 % RH	18.04 ppm (0.057 % RH) **(-), 20 ppm (0.064 % RH) **(-)	691 s (0–150 ppm) 437 s (20–60 % RH)	50 ppm CO ⁽⁺⁾ , 5 ppm NH ₃ ⁽⁺⁾ , 50 ppm ethanol ⁽⁺⁾ , 4.5 ppm N ₂ O ⁽⁻⁾ , 1500 ppm CO ₂ ⁽⁻⁾ , 25 ppm CH ₄ ⁽⁻⁾	7 days	This Work

* Calculated ⁽⁺⁾/Experimental ⁽⁻⁾.

** Calculate at 298 K, 1013 mbar, 28,9645 g/mol for air.

*** Commercial Name.

**** Compensated.

(PANalytical XPert PRO MPD θ/θ) equipped with Cu anode ($\lambda = 1.5418 \text{ \AA}$) radiation, operating at 45 kV and 40 mA was used to record diffraction patterns over the 2θ range from 2° to 60° , with a step size of 0.0263° and a measuring time of 300 s per step). The structural porosity was characterized by Brunauer–Emmett–Teller (BET) using N_2 adsorption isotherms (Micromeritics Tristar 3000).

X-ray photoelectron spectroscopy analysis was performed with a TFA-XPS spectrometer (Physical Electronics, Inc., Chanhassen, USA) using a focused monochromatic Al K_{α} source (1486.68 eV, 200 W). Spectra were collected over a $400 \mu\text{m}$ -diameter area with a pass energy of 58 eV. Charge neutralisation was achieved using a low energy electron beam. The binding energy scale was referenced to the C 1s peak at 284.8 eV. Multipak software (v9.1.1) was used for spectral analysis.

In situ UV–vis absorption spectroscopy was performed using a Lab-sphere ILLUMIA-LITE from Pro-Lite Technology Iberia coupled to our customized gas chamber with a quartz optical window. During measurement, a white LED positioned beneath the HKUST-1 tag served as a broadband light source.

2.3. Preparation of HKUST-1 Tags for gas sensing

HKUST-1 was deposited onto 1 cm^2 polyester sheets (Overhead transparencies 3552, Avery Zweckform) using an airbrush (Airbrush ABPST01, Timbertech) equipped with a 0.3 mm nozzle and filled with 3.5 mL of HKUST-1 paste. A constant spraying pressure of 2 bar was applied. To isolate the sensing area, the backside of each polyester sheet was covered with double-sided tape (Marshall), thereby creating what we refer to as “tags”. The optimal amount of deposited HKUST-1 was determined by incrementally increasing the number of sprayed layers on the substrate. We observed that excessive deposition negatively impacted the sensor’s response time without significantly altering the intensity of the color variation (Fig. S11). This behavior suggests that the color change is primarily governed by surface interactions, with the inner layers contributing minimally. For subsequent experiments, we selected tags that contained approximately 1 mg of HKUST-1.

2.4. Experimental setup for gas sensor evaluation

The physicochemical changes induced by humidity were transduced into a digital signal using a commercial MAX30105 module. This module integrates three internal light-emitting diodes (LEDs) emitting in the green ($537 \pm 35 \text{ nm}$), red ($660 \pm 20 \text{ nm}$), and infrared ($880 \pm 30 \text{ nm}$) ranges. The reflected light was detected by a coupled broadband photodiode (640–980 nm, quantum efficiency $> 30 \%$). The module was configured [17] with an analogue-to-digital converter range of 8192, an averaging of 8 samples per measurement, a sampling frequency of 1600 Hz, and a fixed LED drive current of 12.55 mA with a $215 \mu\text{s}$ pulse width, ensuring stability and reproducibility. The module was controlled via an ESP8266 microcontroller using an I²C interface. The entire sensor system, comprising the HKUST-1 tag and the MAX30105 module, was placed inside a hermetic gas chamber (Fig. 1).

Experiments were carried out at room temperature (Average temperature during the experiments specified at each figure caption). Several mass flow controllers (Bronckhorts) were programmed to inlet precise mixtures. Two humidity sources were used to achieve a working range of 0–80 % RH. For high concentrations, a gas bubbler system directly pumped dry synthetic air into water, providing concentrations up to 80 % RH. Real time humidity, temperature and pressure were monitored using a commercial BME280 sensor (Bosch, work range $0\text{--}100 \pm 3 \%$ RH) [25]. For low concentrations, a premixed H_2O gas bottle provided concentrations up to 150 ppm in synthetic air (Abello Linde). In this case, the relative humidity within the chamber can be calculated using the measured temperature and pressure in conjunction with the known water vapor concentration. To achieve the tested concentrations of interference gases, we diluted premixed gas bottles of 100 ppm of CO, 4548 ppm of CO_2 , 50 ppm of CH_4 , 100 ppm of ethanol

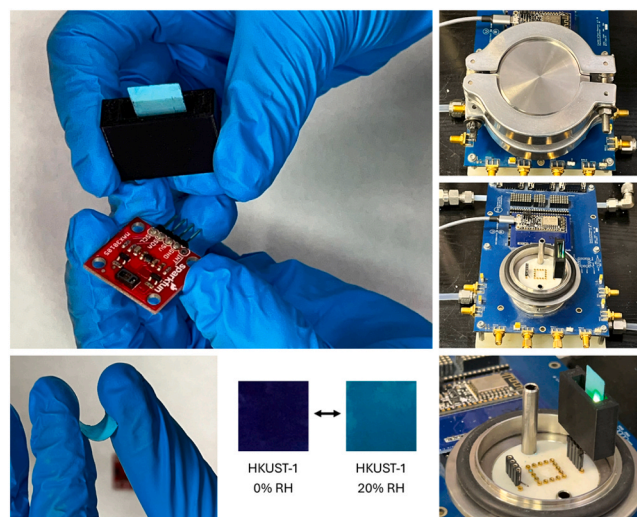


Fig. 1. Illustration of the deposited HKUST-1 onto a polyester sheet, showing the color change of HKUST-1 under different humidity conditions. Schematic of the sensor setup comprising the HKUST-1 tag and the commercial MAX30105 LED-photodiode module. Representation of the working principle of the optical read-out system. Photographs of the custom gas chamber opened and closed.

and 9 ppm of N_2O (all in synthetic air) and 200 ppm of NH_3 (in nitrogen), using synthetic air as dilution gas (Abello Linde). Prior to all experiments, the tags were activated by flushing with a continuous flow of dry air to ensure the removal of any pre-adsorbed moisture.

3. Results and discussion

3.1. Material characterization

The crystalline structure of the synthesized material was confirmed by X-ray diffraction (XRD). As shown in Fig. 2a, the diffractogram matches well with the simulated pattern of HKUST-1 (CCDC 987873), with sharp peaks at characteristic 2θ values. The absence of secondary phases and the narrow peak widths confirm high crystallinity and phase purity, validating the effectiveness of the liquid-assisted grinding (LAG) synthesis route. This structural quality is essential for ensuring consistent porosity and reproducible sensor performance.

Nitrogen adsorption–desorption isotherms (Fig. S12) revealed a Type IV profile [27] with a pronounced nitrogen uptake at low relative pressures, indicative of microporous character. The isotherm also exhibits H4 hysteresis, suggesting the coexistence of mesoporosity. The Brunauer–Emmett–Teller (BET) surface area was calculated to be $1009.64 \text{ m}^2/\text{g}$, with an average micropore size of 5.24 nm and a micropore volume of $0.35 \text{ cm}^3/\text{g}$. These values are characteristic of high-quality HKUST-1 and confirm the preservation of its porous architecture, critical for gas adsorption and diffusion.

Fig. 3a shows the evolution of the absolute difference of transmitted photon flux ($\Delta\phi$ relative to the activated, dry state of the HKUST-1 tag, as it was exposed to 2.5 % RH increments every 15 min, up to a final RH level of 20 %. A spectrum was recorded every 4 seconds. Two distinct absorption bands clearly observed at 590 nm and 680 nm. These bands evolved with increasing RH, and a perceptible color with even small humidity (2.5 % RH) changes. Fig. 3b shows the transmitted photon flux as a function of RH at selected wavelengths near these bands (extended in Fig. S13). These results indicate a non-linear relationship between color and RH. Although these two bands fall within the emission bands of the green and red LEDs of the MAX30105 module, an LED with an emission band centered at either 590 nm or 680 nm would likely provide a stronger response. At very low RH concentrations (nominally 0 %), $\Delta\phi$ around the red LED emission band is positive and transitioned

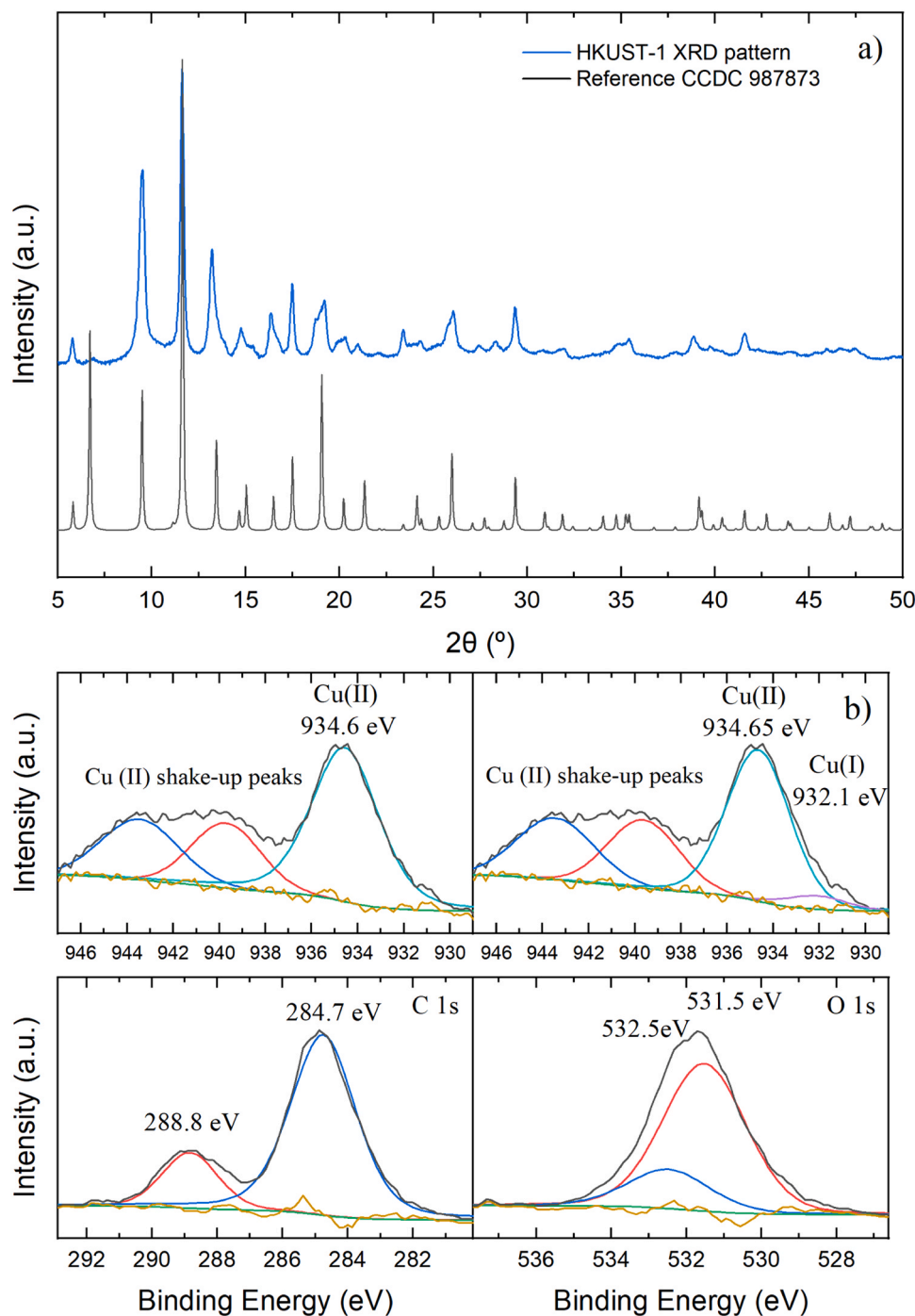


Fig. 2. (a) XRD pattern of synthesized HKUST-1, showing excellent agreement with the simulated pattern (CCDC 987873) and confirming high crystallinity and phase purity. (b) . XPS spectra of the sample. Top left: Cu 2p region excluding Cu(I), highlighting dominant Cu(II) and shake-up satellites. Top right: Cu 2p region including Cu(I). Bottom left: C 1s spectrum confirming the BTC linker, with signals from aromatic and carboxylate carbon. Bottom right: O 1s spectrum showing coordinated carboxylate oxygen and minor physisorbed species.

to negative at higher RH levels.

The origin of this color change has been widely discussed in literature. Historically, it has been attributed to ligand-to-metal charge transfer (LMCT) and d–d transitions within the Cu(II) paddle-wheel units of HKUST-1, where two Cu^{2+} ions are bridged by carboxylate groups [28]. However, recent studies propose that structural defects may play a critical role, particularly those involving partial reduction of Cu(II) to Cu(I) [29,30]. These defects reduce the local symmetry of the paddle-wheel units and introduce new electronic transitions, thereby increasing absorption in the visible range.

To probe this hypothesis, X-ray photoelectron spectroscopy (XPS) was employed. The Cu 2p region (Fig. 2b) consistently shows strong peaks around 934 eV with well-defined shake-up satellites between 940 and 945 eV, confirming a predominant Cu(II) oxidation state [31]. Although a faint feature near 931 eV can be forced via constrained fitting procedures, it lacks sufficient intensity and definition to be considered conclusive evidence of Cu(I) (Fig. 2 b). Thus, we interpret our XPS results as indicating that any potential Cu(I) species exist only in trace amounts or are transient, possibly below the detection threshold of the technique. Taken together, the UV–vis and XPS data support a dual

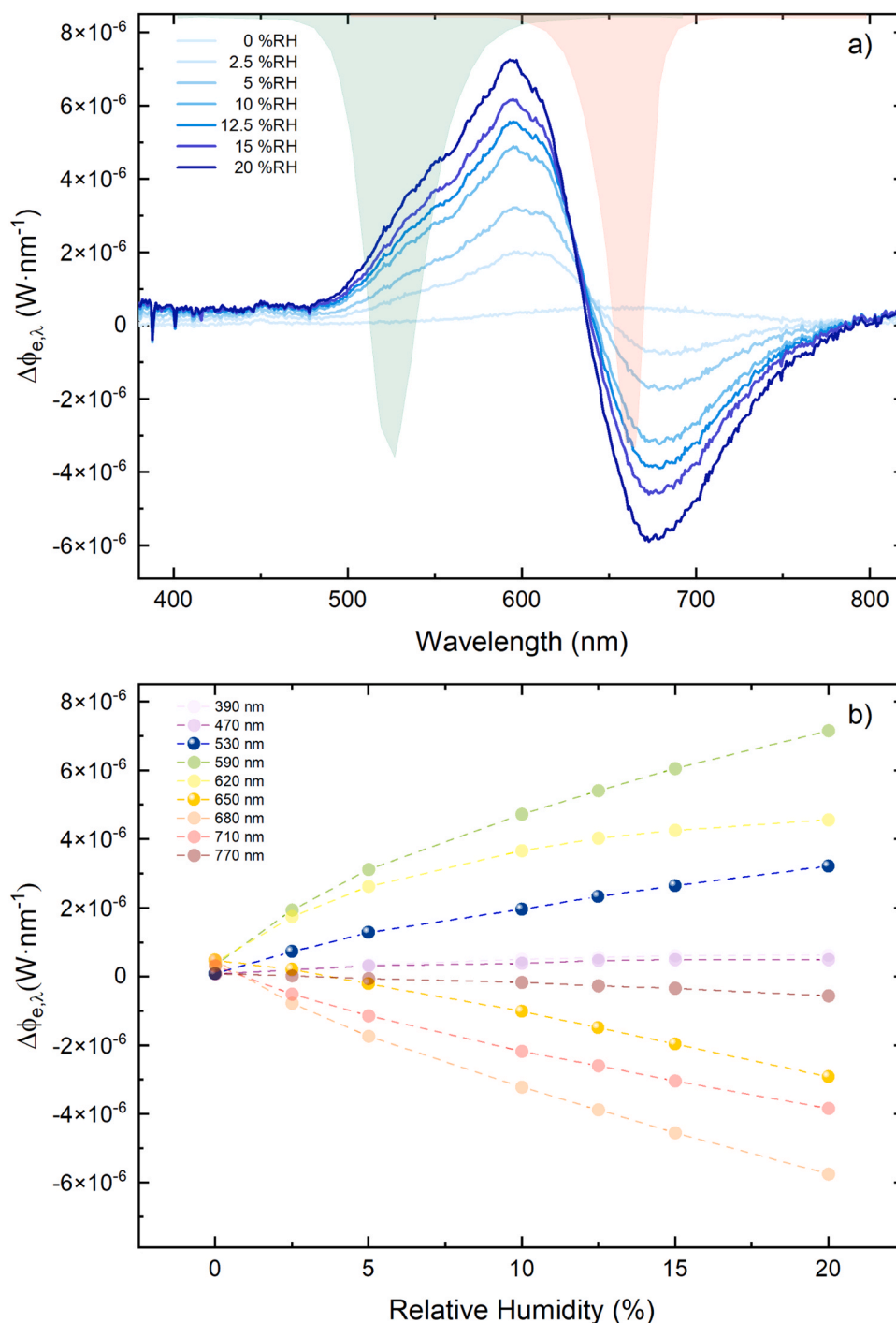


Fig. 3. (a) Evolution of the absolute difference in transmitted photon flux ($\Delta\phi$) relative to the activated dry state of HKUST-1 as a function of increasing relative humidity (RH) up to 20 %. Two distinct absorption bands at 590 nm and 680 nm evolve with humidity. (b) Transmitted photon flux at selected wavelengths as a function of RH. The emission bands of the MAX30105 module's green and red LEDs are indicated for reference.

interpretation of the color mechanism. The dominant contribution arises from the coordination environment of Cu(II) centers, which is altered upon water adsorption [27,28]. However, subtle optical effects, such as band shifts or intensity variations, may be modulated by local defect states involving partial Cu(II) reduction. These processes, although challenging to isolate spectroscopically, are consistent with the material's high color sensitivity to humidity and its reversible behavior, discussed in the following sections.

In addition to copper, the XPS spectra for carbon and oxygen confirmed the expected chemical environments within the framework.

The C 1s spectrum revealed distinct peaks associated with aromatic carbons (C–C/C=C) at ~ 284.7 eV, carboxylate groups (O–C=O) at ~ 288.8 eV. No trace from adventitious carbon (e.g., C–O or C=O) at ~ 286.2 eV was detected. These features are fully consistent with the chemical structure of the BTC linker in HKUST-1 and confirm the presence of intact organic moieties within the framework (Fig. 2c).

The O 1s spectrum showed a main peak centered around ~ 531.5 eV, which is typically assigned to the oxygen atoms from the carboxylate groups coordinated to copper. A secondary component at ~ 532.5 eV was also observed and is attributed to non-coordinated or physisorbed

oxygen species, such as adsorbed water or surface hydroxyl groups (Fig. 2d).

3.2. Sensor performance across humidity ranges

Then, the sensor (MOF tag + MAX30105) was placed in the custom chamber and tested under a humidity ramp from 5 % up to 80 % RH using the gas bubbler set up. Prior to this, the sensor was activated and stabilized under dry synthetic air for around 8 hours. During the experiment, the RH was monitored with a BME280 sensor placed inside the chamber (note that the limit of detection is roughly 3 %). Fig. 4 shows the evolution of the photodiode signal under red and green LED illumination. The signals for both LEDs were normalized between 0 % and 100 % for illustration purposes, as the signal corresponding to the red LED was much higher than the green LED (Fig. S14). As shown, both signals exhibit three distinct regimes (Fig. 4, insert): (i) Up to 11 % RH, the green signal has a positive slope, whereas the red signal has a negative and minor slope; (ii) from 11 % to 25 % RH, the slope of the green LED signal decreases, while the red LED signal increases with a positive slope; and (iii) from 25 % RH onwards, where both signals decrease in slope, lose some linearity, and show some degree of color saturation at high RH concentrations. Both signals are fully consistent with the UV-vis optical characterization (Fig. 3), which predicted the two most distinctive trends: (i) higher sensor response (slope) at lower RH concentrations; and (ii) a transition from a negative to a positive slope for the red LED signal (660 ± 20 nm) at very low RH concentrations.

To understand the observed regimes, we draw an analogy between our humidity transient experiments and a water vapor adsorption isotherm at approximately 298 K. Our sensor monitors the color evolution of HKUST-1, which is directly related to the amount of adsorbed water vapor within the MOF. This optical approach offers a significant advantage over conventional gravimetric methods by providing much higher resolution at very low vapor pressures (below $p/p_0 = 0.002$). In contrast, typical water vapor adsorption isotherms have limited resolution for relative vapor pressures around 0.01, hindering accurate analysis in this crucial low-pressure region.

In this regard, some authors have identified two energetically distinct adsorption processes within the low-pressure region [28,32,33]. The first regime (0–10 % RH) is attributed to the coordination of water

molecules with the unsaturated copper sites, as mentioned earlier. In the second regime (10–25 % RH), the filling of small and large pores within the MOF framework is believed to be the primary driver of the observed color changes. However, other authors suggested that the color change up to 25 % RH is solely due to the direct interaction of water molecules with unsaturated copper sites [27,32].

The fact that we clearly observed two distinct regimes within 0–25 % RH (specially with green LED signal) is compatible with this latter hypothesis. Water adsorption kinetics depend on the accessibility of Cu sites and HKUST framework has three different cavities with pore diameters of 1.4, 1.1 and 0.5 nm. The larger cavities are readily accessible to water molecules (Regime I), while the smallest cavity likely hinders adsorption kinetics (Regime II). In this context, the saturation region (Regime III) could be attributed to the subsequent filling of pores by water molecules. Concerning the desorption of water molecules, our transient measurements (Fig. 5) demonstrate that HKUST-1 fully recovers its original color after the cessation of water vapor exposure. To further evaluate the reversibility and robustness of the HKUST-1 sensor, we performed hysteresis measurements using non-sequential cycles of increasing and decreasing relative humidity levels. A total of 20 humidity pulses (10 forward and 10 backward) were applied in randomized order at identical concentrations to eliminate sequence bias. The sensor exhibited minimal hysteresis, with desorption signals closely overlapping the adsorption curve, as shown in Fig. 5b. The average signal deviation between forward and backward responses was within 3.5 %, comparable to the experimental noise, confirming the fully reversible nature of water adsorption in HKUST-1 and the absence of memory effects. These results reinforce the sensor's reliability for repeated humidity sensing without requiring reactivation.

To extend the study to a lower range of relative humidities and determine the limit of the detection of our sensor, we conducted experiments using a calibrated gas bottle (150 ppm in synthetic air) with low water vapor concentration. The HKUST-1 tag was first activated under a constant stream of dry air for 2 h. Subsequently, a sequence of 30-minute pulses of water vapor, ranging from 20 to 150 ppm (0.05–0.51 % RH at an average temperature of 24.15 °C and pressure of 102923 Pa), was introduced into the chamber. Fig. 5a shows the recorded response by the sensor. In this case, only the green LED signal is presented because, as indicated previously, the response in the low-pressure regime was higher. An initial drift was likely caused by an incomplete activation procedure. As can be observed, the photodiode signal is fully reversible, and the signal increment respect to the baseline is proportional to the water vapor concentration (insert of Fig. 5a) with a linear trend (R-Square 0.971). The limit of detection (Eq. (1)) obtained was 18.04 ppm of H₂O. This is close to the experimental limit of detection (20 ppm), which is the lowest analyte concentration that produces a measurable change in the signal relative to the baseline within the noise limits of our setup.

$$LoD = \frac{3.3 \cdot \text{Standard deviation of the signal noise}}{\text{Slope of linear fit}} \quad (1)$$

The capacity of our sensor to measure low humidity concentrations is related with its high surface area and intrinsic porosity and is comparable to most advanced low-humidity sensors reported in the literature [4]. However, our approach utilizes a significantly simpler and more cost-effective configuration: a tag with a MOF printed on top as the sensing element and a commercially available module with integrated LEDs and photodiode.

3.3. Evaluation of interferent gases

To evaluate potential interfering gases, we conducted comprehensive cross-interference experiments with N₂O, NH₃, ethanol, CO, CH₄ and CO₂. The experiments (Fig. 6) involved a long activation period with dry synthetic air, followed by pulses of the potential interfering gas at concentrations significantly exceeding those found in a typical

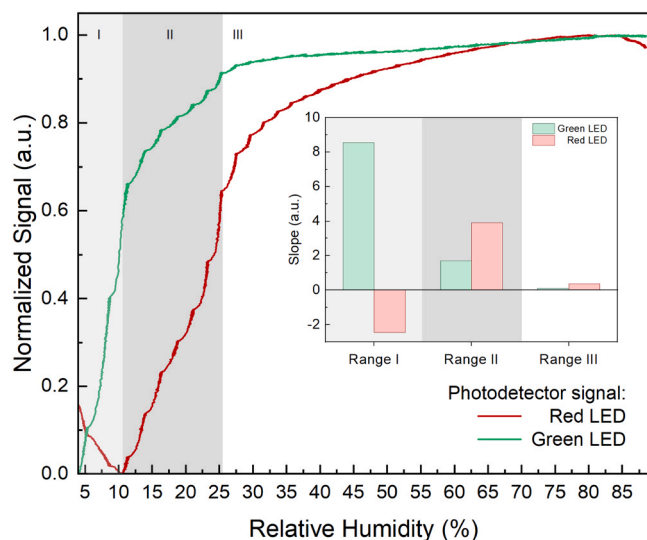


Fig. 4. Correlation between the normalized HKUST-1 sensor signal and relative humidity (RH), measured by a BME280 sensor during a humidity ramp from 5 % to 80 % RH. Three distinct regimes are highlighted by different shades of gray. Insert: Linear fits of the data within each regime, indicating the corresponding slopes.

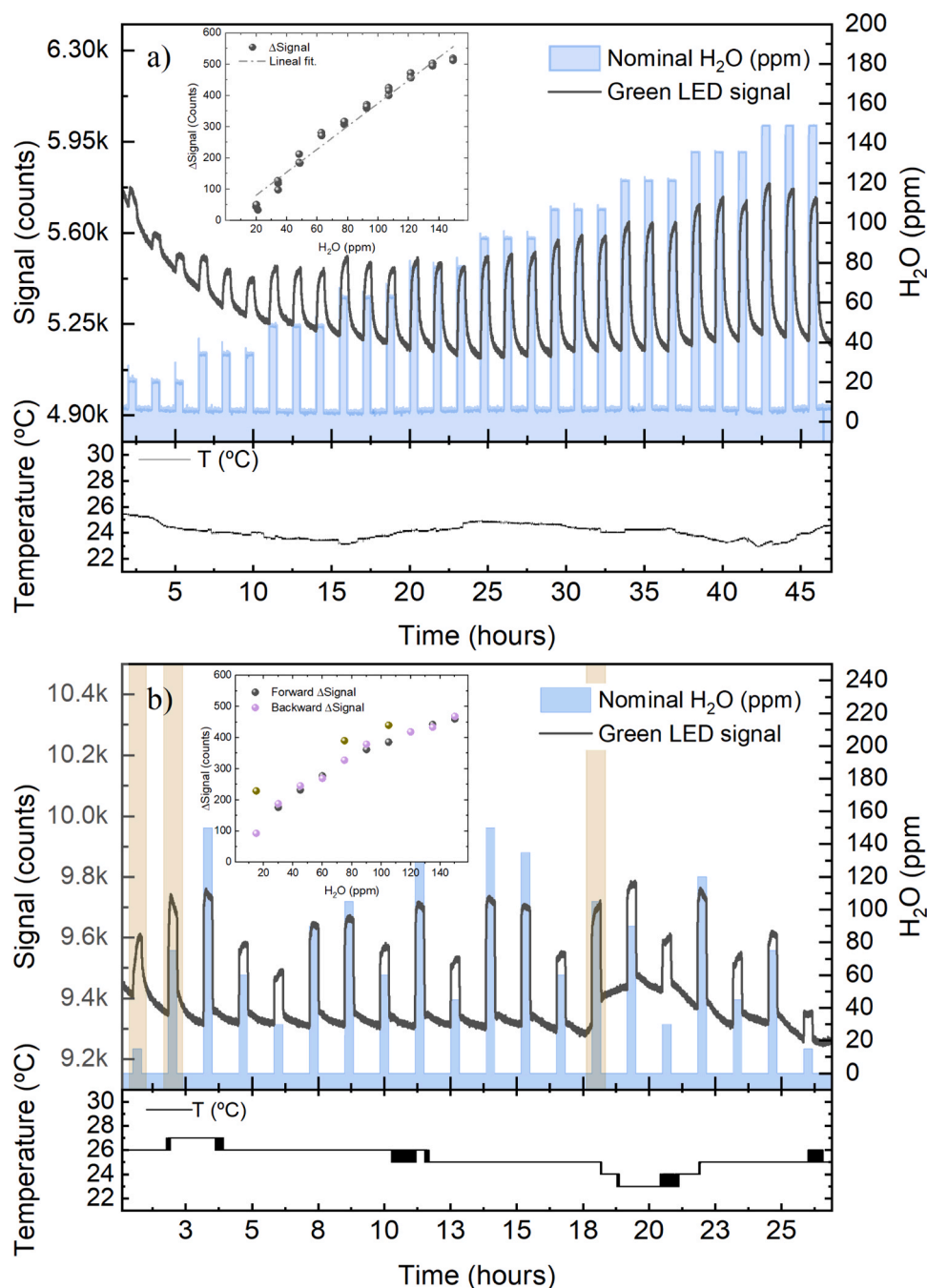


Fig. 5. (a) Transient response of the HKUST-1 sensor to periodic step changes in H₂O concentration from 20 to 150 ppm. Measurement was performed at an average temperature of 24.15 °C and pressure of 102.923 kPa. Inset: Linear fit of the photodiode green Δ signal versus H₂O concentration. (b) Sensor response to a hysteresis test with non-sequential exposure to increasing and decreasing H₂O concentrations. Inset: Forward and Backward Photodiode green Δ signal versus H₂O concentration.

atmosphere. This approach allowed us to assess their individual influence on the sensor response without competition from water vapor.

In dry conditions, we observed significant color changes in the sensor upon exposure to NH₃, CO, and ethanol (Fig. 6a). Previous studies have shown that ammonia interacts with HKUST-1 in a manner similar to water vapor, involving acid-base interactions, but with potentially greater structural perturbations [20]. No color shift was observed with CO₂, CH₄, and N₂O.

Carbon monoxide also induced a noticeable color change, albeit with lower binding energies compared to ammonia. The interaction mechanism with CO is distinct from that of ammonia and water, involving electrostatic interactions and σ and π back-donation [20]. Finally,

ethanol exhibited unique behavior. Upon exposure, the sensor signal showed a sharp initial rise followed by a slower, gradual increase without reaching saturation. Moreover, the color change was not fully reversible. This behavior could be attributed to the dynamic diameter of ethanol (0.44 nm) and its interaction with the hierarchical pore structure of HKUST-1 (0.5 nm, 1.1 nm, and 1.4 nm) [34]. The varying pore sizes may lead to differential accessibility of copper sites within the framework, potentially explaining the observed signal profile and incomplete reversibility. This observation is consistent with reports of non-fully reversible ethanol adsorption in HKUST-1 [35].

In the second stage of the experiment, we introduced a mixture of 75 ppm of H₂O and the same concentration of the interfering gas

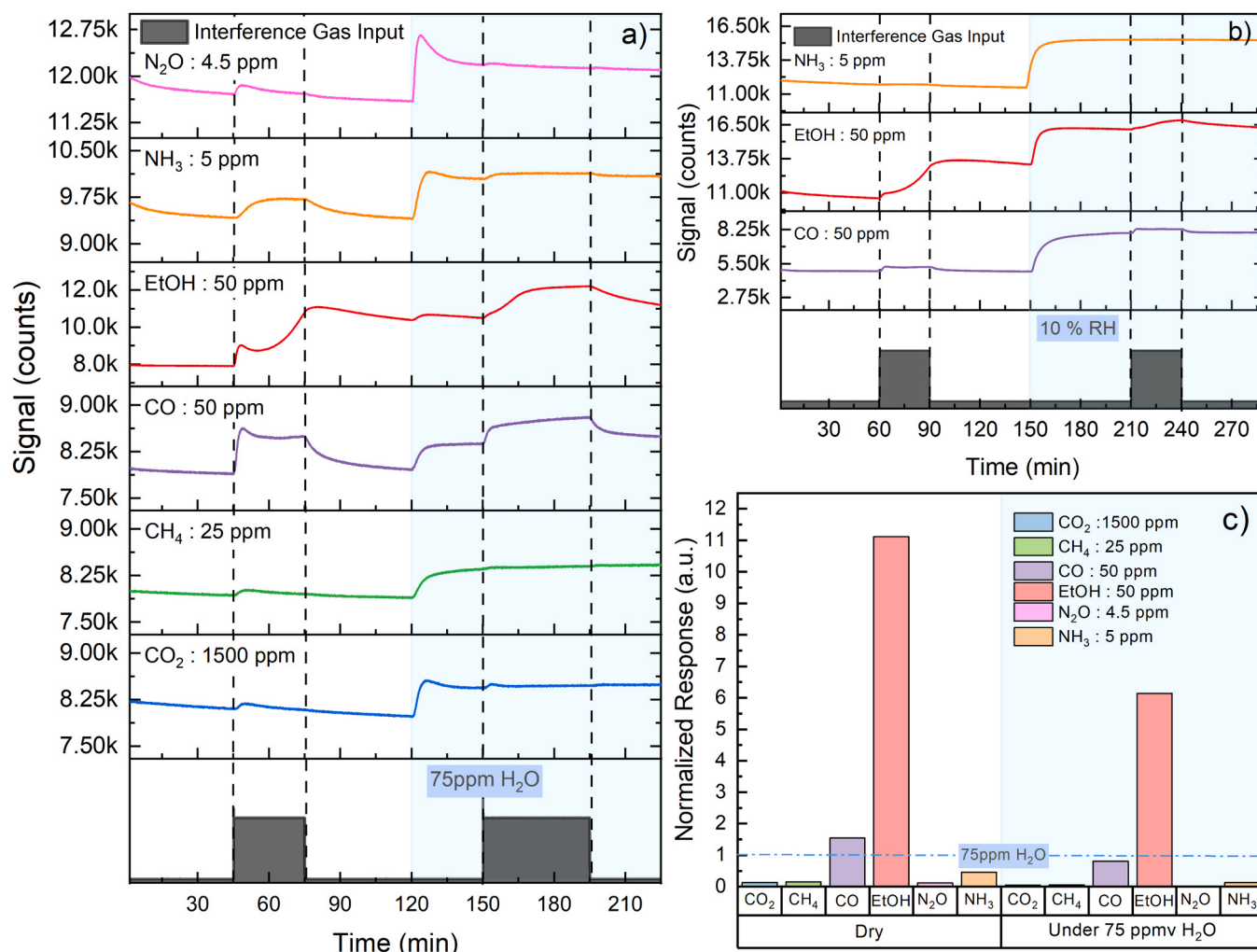


Fig. 6. Comparison of the HKUST-1 sensor green signal response to CO₂, CH₄, CO, ethanol, NH₃, and N₂O under dry and humid conditions. (a) Humid conditions: 75 ppmv H₂O (equivalent to 0.24 % RH) at 25.26 °C and 102.419 kPa. (b) Humid conditions: 10 % RH. (c) Relative signal increase for each gas compared to the signal generated by 75 ppm of H₂O.

(Fig. 6a). The presence of water vapor led to competition for copper sites, as evidenced by a decrease in the relative signal change for NH₃, CO, and ethanol during their respective pulses. Notably, the ammonia signal was almost completely suppressed. Upon removal of the interfering gases, the sensor signal returned to the baseline in the humid environment. This suggests that under these conditions, only ethanol and CO could potentially cause a false humidity reading at very low relative humidity (< 1 % RH). To further investigate the competition between water vapor and the interfering gases, we conducted another cross-interference experiment using a mixture with 10 % RH (Fig. 6b). At this higher humidity level, the interference from ammonia was eliminated, while ethanol and CO continued to show some interference. For reference, Fig. 6c and Fig. S15 show the normalized response of the sensor to the interferent gas, relative to its response to water at 75 ppm and 10 % RH, respectively.

3.4. Repeatability, stability, reproducibility and kinetic response of the sensor

The repeatability and reliability of the sensor were first evaluated through two separate 7-day experiments under distinct humidity regimes. In the first case, 30-minute humidity pulses ranging from 0–150 ppm H₂O were introduced every 4 hours, and in the second, the pulses spanned 20–60 % RH under the same timing conditions (Fig. 7).

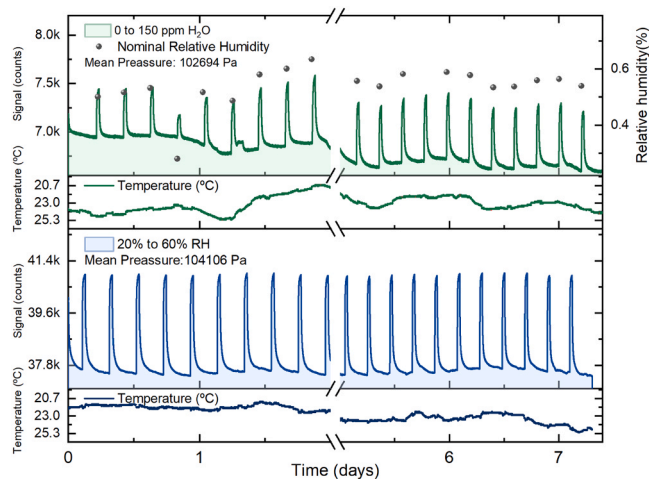


Fig. 7. One-week stability test of the HKUST-1 sensor. Top: Response to periodic 150 ppm H₂O pulses in synthetic air (green LED signal), with corresponding relative humidity (black dots) and temperature. Bottom: Response to periodic 40 % RH increments in a 20 % RH carrier gas (red LED signal), with temperature. Stable and reversible behavior was maintained, with minor baseline shifts in the ppm range attributed to temperature dependence.

The coefficient of variation (CV) was calculated to assess repeatability: a CV of 16.11 % was obtained for the low humidity range (0–150 ppm) and 1.91 % for the moderate humidity range (20–60 % RH), both confirming good measurement precision (CV < 20 %). A one-way ANOVA

analysis grouping the Δ Signal responses by experimental day revealed statistically significant variations across days (p-values = 0.0017 for 0–150 ppm and 0.00014 for 20–60 % RH). Although day-to-day variations were observed, the consistently low CV values suggest that the

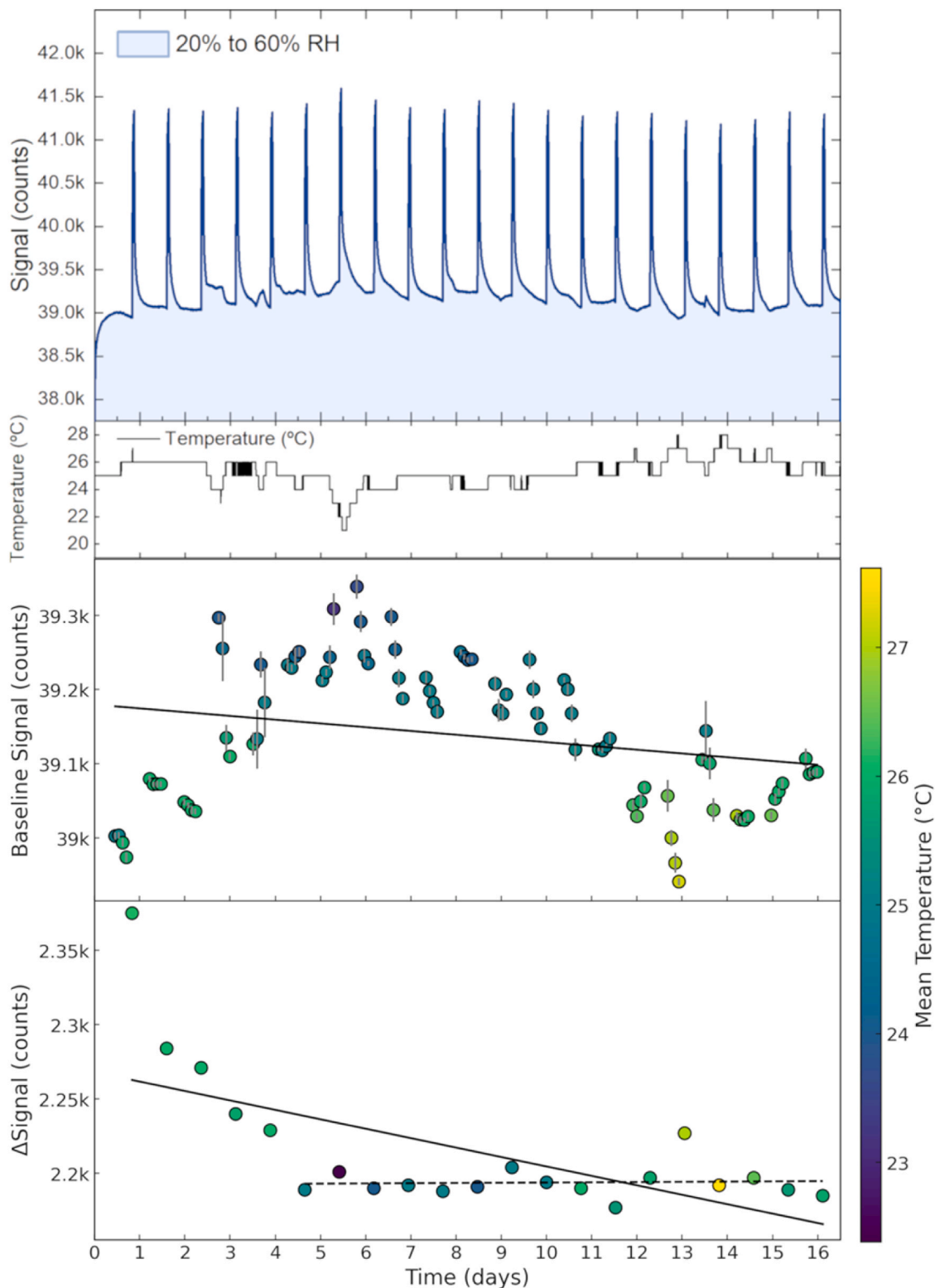


Fig. 8. Long-term performance of the HKUST-1-based humidity sensor under cyclic exposure to 20–60 % RH over 16 days. Top panel: Raw photodiode signal showing sensor response to 1 h humidity pulses every 16.5 h and the corresponding temperature. Middle panel: Baseline drift analysis, calculated as the average signal during the 20 % RH period preceding each pulse. Bottom panel: Δ Signal analysis, defined as the difference between the pre-pulse baseline and the response during each humidity pulse, used to evaluate response consistency and sensor robustness over time.

variability remained minor relative to the signal magnitude (See SI6 section for further details). Particularly at low humidity levels, the Δ Signal showed a visible dependence on temperature, reinforcing the need to interpret sensor signals in terms of relative humidity (%RH) rather than absolute H₂O concentration (ppm), due to the temperature dependency of air's water-holding capacity according to the Clausius-Clapeyron relation.

To further validate the sensor's long-term robustness, we conducted a 16-day extended experiment under moderate humidity conditions (20–60 % RH), introducing 1 h humidity pulses every 16.5 h (Fig. 8). In the baseline stability plot (middle panel), each point represents the average signal over a 2-hour window, taken from the 10-hour dry period preceding each humidity pulse (excluding the final hour before the pulse). This approach was designed to capture the undisturbed 20 % RH behavior of the sensor while avoiding any memory effects from the previous humidity cycle. A gradual downward drift in the baseline signal is observed over time, closely mirroring ambient temperature fluctuations, as shown by the color scale. This is consistent with the effect of temperature on relative humidity: in our experimental setup, humidity levels were generated by bubbling synthetic air through a water-filled wash bottle, meaning the amount of water vapor introduced into the system is inherently dependent on the temperature of the air and the ambient environment. As a result, even when the absolute water content remains approximately constant, the %RH varies with temperature, thereby altering the sensor response. Hence, the baseline drift is attributed to humidity dynamics driven by temperature, not to a direct thermal effect on the sensor itself. Statistical analysis confirms this interpretation, with a strong negative Pearson correlation between baseline signal and temperature ($r = -0.896$, $R^2 = 0.803$, $p \approx 1.15 \times 10^{-30}$).

In the Δ Signal plot (bottom panel), each point corresponds to the difference between the RED signal immediately before the humidity pulse (2 data points prior to the onset) and the signal measured after 10 data points into the pulse. This Δ Signal quantifies the sensor's optical response to the humidity step. Across the full dataset, the Δ Signal shows a small downward drift (-6.34 counts/day) and a coefficient of variation (CV) of 2.05 %. However, this variation is mainly concentrated in the early part of the experiment. From the sixth pulse onward, the response stabilizes into a consistent plateau, with negligible drift ($+0.15$ counts/day), a CV of only 0.48 %, and no statistically significant correlation with temperature ($r = +0.163$, $R^2 = 0.026$, $p = 0.547$). These results confirm that the sensor response is highly repeatable and largely unaffected by thermal variations under steady-state conditions. A detailed description of the analysis methodology is provided in [Supporting information Section SI7](#), and a summary of the key statistical parameters is presented in [Table SI7](#).

To evaluate sensor-to-sensor reproducibility, measurements from three independent devices (each consisting of an HKUST-1 tag and a separate MAX30105 readout module) are presented in [Supplementary information Fig. SI8](#). The results show that absolute photodiode signals vary between devices, primarily due to the manual deposition process of the HKUST-1 layer and inherent differences among the commercial readout modules, both of which introduce variability. However, when the signals are normalized to each sensor's minimum response value (i. e., the signal at 0 ppm H₂O in the fully activated dry state), the relative responses become more comparable. Despite this improvement, additional refinements, particularly in deposition control and readout standardization, are necessary before the sensors can be considered truly reproducible.

Finally, [Fig. SI9](#) presents the response and recovery times of the sensor at different humidity increments. For a step change from 20 % to 60 % RH, the sensor required 437 s to reach 90 % of the maximum signal and 2000 s to fully recover. For a concentration increase from 0 ppm to 150 ppm H₂O, the response and recovery times were 691 and 788 s, respectively. The sensor responds more rapidly at higher relative humidity, where hydrogen bonding facilitates faster adsorption kinetics.

In contrast, at lower humidity levels (ppm range), diffusion becomes the limiting factor, resulting in slower response dynamics. In both cases, recovery takes longer than response, suggesting stronger retention of water molecules within the framework once adsorbed.

It is important to emphasize that these measured times include the filling and stabilization period of the test chamber, which is dictated by its volume and gas flow rate [36]. To contextualize this effect, we compared our results with a commercial BME280 sensor, which, despite its datasheet response time of 1 s ($\tau_{90\%}$) [25], exhibited a $\tau_{90\%}$ of 451 s under identical conditions. This indicates that the observed response times are not solely intrinsic to the sensing material but are largely influenced by the experimental setup.

This comparison underscores the importance of interpreting response times within the context of the measurement system. In our configuration, the gas exchange limitations mask the true kinetics of the HKUST-1 sensor. Therefore, while these values provide a useful performance benchmark, further improvements in sensor integration and chamber design could significantly reduce overall response time. These findings conclude the evaluation of the sensor's dynamic behavior and reinforce the material's potential for reliable low-humidity detection in real-world applications.

4. Conclusions

In this work, we demonstrated the potential of HKUST-1 as a colorimetric sensor for ppm-level humidity detection, covering a broad range from 0 % to 80 % relative humidity (RH). The material exhibited fully reversible behavior at room temperature, using only dry air for regeneration, making it highly promising for humidity monitoring in industrial and environmental applications. The optical response of HKUST-1 was found to be highly sensitive to humidity variations, with distinct absorption bands evolving with RH, enabling precise detection at low concentrations. Through a comprehensive study of cross-sensitivity, we observed significant reversible color changes upon exposure to NH₃, CO, and ethanol, while gases like CO₂, CH₄, and N₂O induced negligible response. Competitive adsorption experiments revealed that at higher humidity levels (> 10 % RH), water vapor effectively suppressed interference from ammonia, whereas ethanol and CO could still cause minor perturbation, particularly under very dry conditions (< 1 % RH). Long-term stability tests over one and two weeks demonstrated consistent performance, with minimal drift attributable to environmental factors such as temperature variations rather than material degradation. The sensor maintained good repeatability, with a coefficient of variation below 20 % in the ppm range and even lower at moderate RH, validating its robustness for real-world deployment. Furthermore, despite intrinsic variability due to manual MOF deposition and commercial readout module differences, standardized signal normalization showed that comparable relative responses could be achieved between different devices, highlighting a feasible path toward scalable sensor fabrication. Finally, although response and recovery times are currently influenced by the chamber filling dynamics rather than intrinsic material limitations, our HKUST-1-based sensor showed competitive or even faster performance compared to commercial sensors under identical experimental setups. This suggests that system-level optimizations could further enhance response times without compromising sensitivity.

CRedit authorship contribution statement

Fàbrega Cristian: Writing – review & editing, Validation, Supervision, Resources, Project administration, Methodology, Formal analysis, Conceptualization. **Xuriguera Martín Elena:** Validation, Supervision, Resources. **Martínez Medina Elizabeth:** Writing – original draft, Validation, Investigation, Formal analysis, Conceptualization. **González Gómez María:** Writing – review & editing, Writing – original draft, Visualization, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Kovač Janez:** Validation, Investigation.

Martínez López Mònica: Validation, Supervision, Resources, Project administration, Methodology, Conceptualization. **Casals Olga:** Validation.

Declaration of Generative AI and AI-assisted technologies in the writing process

During the preparation of this work the authors used Chat GPT and Gemini in order to enhance the writing process. After using this tool/service, the authors reviewed and edited the content as needed and take full responsibility for the content of the publication.

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.

Acknowledgements

This work was supported in part by the Agència per la Competitivitat de l'Empresa (ACCIO) under Grant ACE034/21/000057 (Innotec) and Grant ACE088/23/000028 (Nuclis R+D).

Supplementary Information

includes nitrogen adsorption data confirming the porosity and surface area of HKUST-1, effects of layer thickness on response time, activation and optical response analysis, and additional synthesis details. It also presents XPS results, raw LED signals, reproducibility and cross-interference data, as well as repeatability and response/recovery time studies. This material is available free of charge via the Internet.

Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at [doi:10.1016/j.snb.2025.137973](https://doi.org/10.1016/j.snb.2025.137973).

Data availability

Data will be made available on request.

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