

First- and Second-Order Individual Gas Sensors as the Next Generation of Reliable Analytical Instruments

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Abstract:

Conventional gas sensors frequently underperform in field conditions due to their designs as single-output, “zero-order” devices that cannot differentiate between target analytes, chemical interferents, and sensor drift. To overcome these limitations, we are developing more trusted gas sensors with independent responses to function as first- and second-order devices. Inspired by proven traditional analytical instruments, we show how their underlying mathematical principles are translated into next-generation sensor designs to improve the “3S” attributes of Sensitivity, Selectivity, and Stability. We show the importance of hardware–analytics co-design where machine learning only fully succeeds when sensor outputs contain sufficient independent information. We show our two first-order sensing platforms such as (i) radio-frequency sensors utilizing multi-frequency dielectric excitation of metal oxides and (ii) photonic sensors employing multi-wavelength illumination of nanostructured materials. Our second-order sensors incorporate different modulation techniques into these first-order platforms. These next-generation gas sensors are the revolution from “low-cost commodity” to “high-value differentiated layer” sensors to enable their trusted, widespread environmental and industrial monitoring.

Key words: sensitivity, selectivity, stability, first-order sensor, second-order sensor

1. Introduction

For decades, pellistors, electrochemical, and semiconducting metal oxide (SMOX) sensors have supported industrial and residential safety by detecting high levels of toxic and combustible gases [1, 2]. However, these legacy sensors (a.k.a. sensor elements) are single-output (a.k.a. zero-order) sensors, unable to differentiate gases that produce similar signals or distinguish a gas response from drift [3, 4].

Emerging applications need accurate detection of diverse gases in complex backgrounds (Fig. 1), making limitations of legacy sensors a major bottleneck [5-8]. To solve this challenge, gas sensors based on first- and second-order detection principles are being developed [9-15], inspired by traditional analytical instruments that use independent response variables for multigas differentiation in complex environments [3]. A first-order detector has one independent response variable; gases can be differentiated by different response patterns as a function of that variable. A second-order detector has two independent response variables for robust analysis in complex backgrounds. These principles, established in traditional analytical instrumentation, inspire rethinking of gas-sensor designs (Fig. 1) with enhanced “3S” attributes of Sensitivity, Selectivity, and Stability.

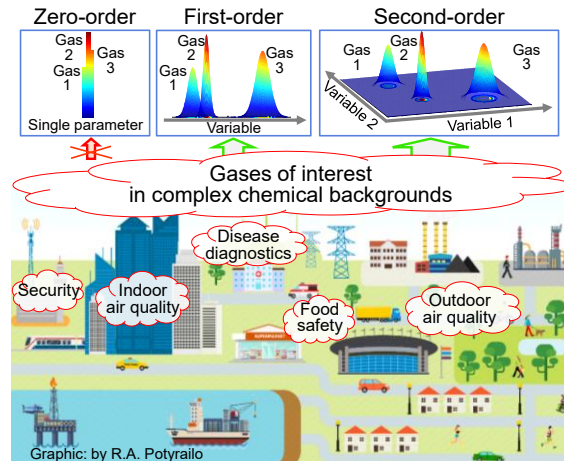


Fig. 1. Efficiency of zero-, first-, and second-order gas sensors in complex chemical backgrounds.

2. Design Rules of First- and Second-Order Gas Sensors

Inspired by mathematics of traditional analytical instruments, first- and second-order gas sensors are being built by teams worldwide by applying four design aspects (Fig. 2a): (1) transducer is built to operate with independent variables, (2) excitation conditions are designed to induce independent responses in transducer / sensing material system, (3) sensing material is devised with variable responses to multiple volatiles

under variable excitation, and (4) edge-based data analytics (a.k.a. machine learning) provides desired “3S” performance improvements.

Over the decades, different sensors were demonstrated with diverse independent variables in their responses and different energies were applied for excitation of sensing materials. Until recently, these results were demonstrated in the laboratory conditions using desktop analyzers followed by off-line processing of multivariate data on desktop computers (Fig. 2b). Early laboratory equipment and results have paved the way toward next-generation gas sensors.

Fig. 2c shows examples of recent advancements in microelectronics that allowed the creation of edge-based sensor readers such as application-specific integrated circuits (ASICs) for impedance measurements and edge-based micro-processors with high computing power capability and low electrical power demands. These and many other hardware innovations drive next-generation gas sensors into trusted field deployments.

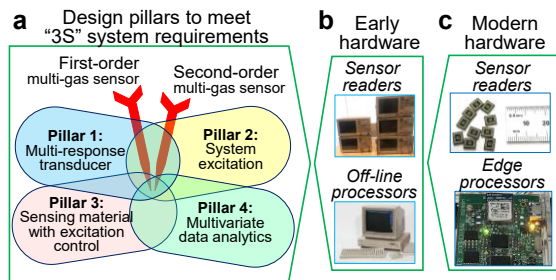


Fig. 2. Building next-generation gas sensors: (a) four design aspects (four pillars), (b) examples of early laboratory desktop analyzers and off-line processors that paved the way to next-generation gas sensors, (c) examples of recent innovations in microelectronics that drive new sensors to trusted field deployments.

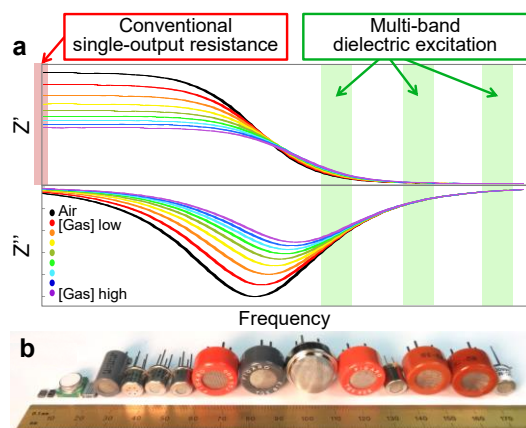


Fig. 3. SMOX gas sensors: (a) legacy single-output resistance and multi-band dielectric excitation responses, (b) all three generations of SMOX sensors that follow new dielectric excitation scheme.

3. Radio-Frequency First- and Second-Order Gas Sensors

SMOX materials showed new performance via a “dielectric excitation” scheme, i.e., impedance spectral readout of Z' and Z'' on the shoulder of their dielectric relaxation peak [13] (Fig. 3). By selecting certain operation frequencies of n-type and p-type SMOX materials, dielectric excitation coupled with suitable data analytics improved desired sensitivity, selectivity and stability.

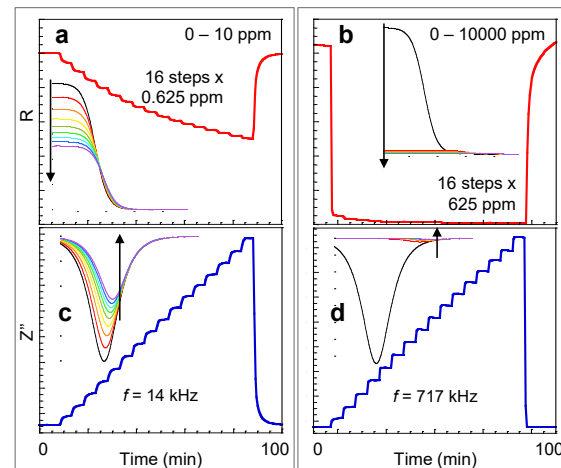


Fig. 4. Monitoring of different concentration ranges of CH_4 using resistance (a,b) and dielectric excitation (c,d) measurements: 0–10 ppm (a,c), and 0–10,000 ppm (b,d). Dielectric excitation Z'' at (c) 14 kHz and (d) 717 kHz. Insets, (a,b) Z' and (c,d) Z'' spectra.

Known non-linearity of SMOX chemiresistors is due to their power law response, degrading sensitivity at high gas levels [16]. Dielectric excitation scheme yields a linear response [13]. Fig. 4a and 4b depict resistance upon exposures to steps of CH_4 gas over 0–10 ppm and 0–10,000 ppm, showing degraded sensitivity at high gas levels. Dielectric excitation shows frequency-selected response linearity (Fig 4c and 4d).

Following these intriguing results, tests with different types of SMOX sensing materials and elements and numerous volatiles were reported by research teams worldwide [17–20], demonstrating a general rule of linear responses of SMOX materials with their dielectric excitation.

Fig. 5 compares dielectric excitation of sensors by desktop and ASIC impedance analyzers with illustrative gases. Both configurations had similar linearity and similar limits of detection of volatiles. The ASIC advantages were in $\sim 10^6$ -fold reduction in volume and $>10^3$ -fold power reduction versus the desktop analyzer [13, 21].

Dynamic range of gas sensing is another important factor. Compared to SMOX resistors with their 2–4 decades of dynamic range [22], dielectric excitation expanded dynamic range of gas detection to more than six decades [13].

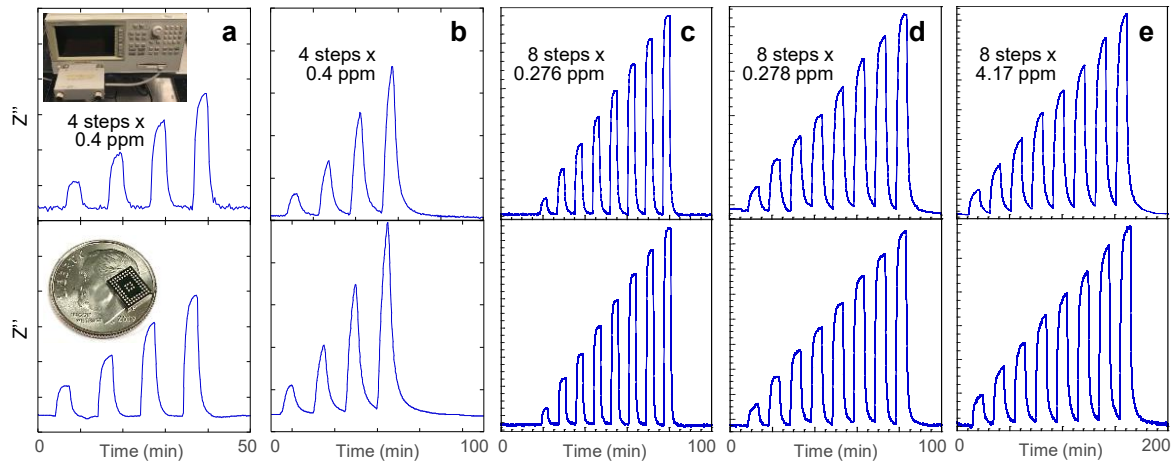


Fig. 5. Dielectric excitation measurements of responses of SMOX sensors to various volatiles measured by desktop (top graphs) and ASIC impedance analyzers (bottom graphs): (a–e) dichloropropane, dimethyl methylphosphonate, benzene, toluene, hydrogen, formaldehyde, and carbon monoxide, respectively.

Dielectric excitation of SMOX sensors differentiated diverse and closely related individual gases and their mixtures [13]. Fig. 6 shows results of differentiation between methane CH_4 , ethane C_2H_6 , and propane C_3H_8 . Such differentiation is important in determination of the biogenic or thermogenic origins of sources of environmental emissions. Different Z' and Z'' response patterns (Fig. 6a–d) allowed differentiation of these gases using a simple machine learning technique such as principal components analysis (PCA) [7] (Fig. 6e).

Numerous gases were differentiated with their gas concentration ratios of up to 200,000:1. Individual gases were differentiated and quantified in binary and ternary mixtures using different multivariate data analytics tools [13].

Stability of gas sensors can be affected by different environmental conditions, leading to drift of sensor baseline and sensitivity [23]. Self-compensation against ambient temperature was demonstrated with cycling from $-25\text{ }^\circ\text{C}$ to $50\text{ }^\circ\text{C}$.

While resistance showed known temperature-induced baseline offsets and variable sensitivity (Fig. 7a), the Z' response at $\sim 2.7\text{ MHz}$ was not temperature-affected (Fig. 7b) [24].

In another test, a baseline drift was observed over duration of a test and a multivariate curve resolution (MCR) machine learning technique (Fig. 8) was applied for multi-frequency baseline correction and noise reduction [25].

Fig. 8a shows the raw sensor response to NO_2 at one of the frequencies over 45 h of testing, where a substantial baseline drift was observed after the first 15 h of the test. For the MCR model development, only the first replicate of the multiple responses to NO_2 was utilized.

Thus, multi-frequency readout with an ASIC impedance analyzer coupled with the MCR algorithm resulted in automatic baseline correction and noise reduction without preselection of operation frequencies (Fig. 8b).

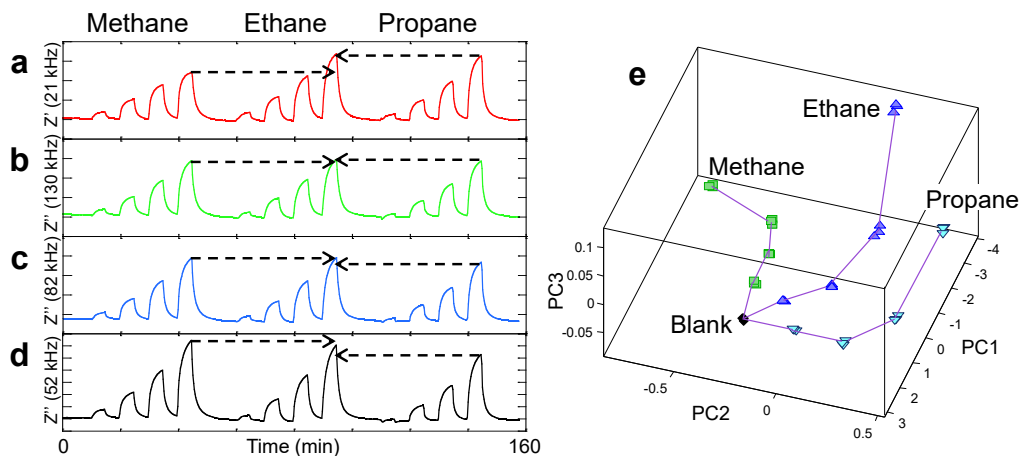


Fig. 6. SMOX sensor differentiates CH_4 , C_2H_6 , and C_3H_8 gases: (a–d) dynamic responses to three gases at different frequencies, (e) pattern recognition using PCA score plot. Each gas, 0.25, 0.5, 0.75, 1.0 % vol.

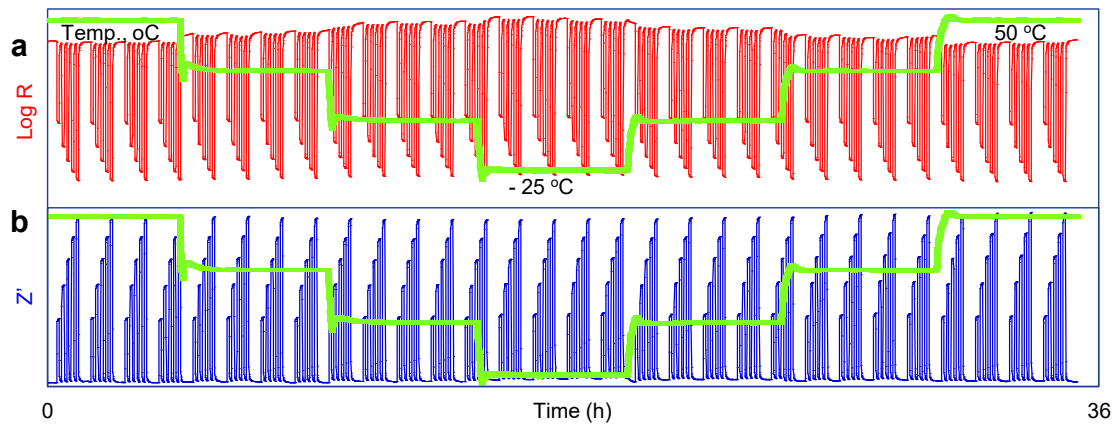


Fig. 7. Effects of ambient temperature from -25 °C to 50 °C on (a) resistance and (b) dielectric responses of a SMOX sensor upon exposures to CH₄ (50, 100, 150, 200, and 250 ppm).

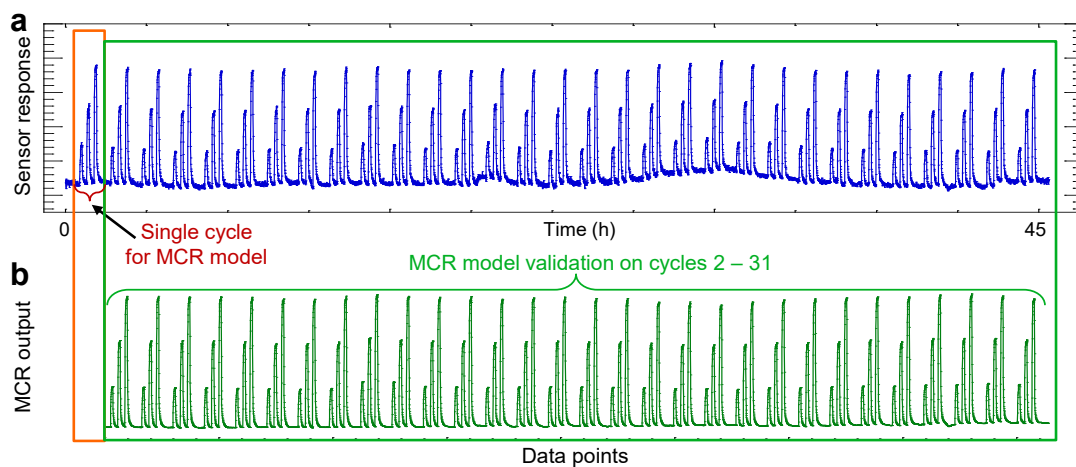


Fig. 8. Multi-frequency MCR self-correction of baseline drift and noise reduction of a SMOX sensor under its dielectric excitation with an ASIC impedance analyzer during multiple cycles of NO₂ exposures over 45 h in ambient lab conditions: (a) raw sensor response; for MCR model development, only the first replicate of multiple responses to NO₂ was utilized, (b) steady baseline and reduced sensor noise by MCR algorithm. NO₂: 80, 160, 240 ppb.

Using impedance measurements at different frequencies also provided the ability to correct for effects of partial poisoning of SMOX sensors by siloxane vapors because of different effects of siloxane vapors on impedance spectra [26].

Gas sensor nodes with dielectric excitation were tested in multiple field campaigns, showing <15% drift 407 days after the initial calibration. Dielectric excitation sensors were initially commercialized for CH₄ detection in the Oil and Gas industry [27].

In building second-order sensors, we operated SMOX sensors with their dielectric excitation and implemented thermal and radiant modulation [28]. Dielectric excitation provided a frequency-dependent response, while temperature and radiation modulation introduced additional gas-specific transient signatures that further increased response dimensionality. These results provide a practical route for building high-information-content gas sensors for emerging applications [10, 28, 29].

4. Photonic First- and Second-Order Gas Sensors

Our photonic sensors are inspired by iridescent butterfly scales, which are natural metamaterials [30]. We discovered that iridescence of *Morpho* butterflies is attractive for gas sensing [11] because it is produced by micro-scales with an open-air nano-architecture that allows volatiles to interact with all its regions.

We created design rules of photonic nanomaterials for multi-gas differentiation and fabricated nanomaterials to operate at room and ~300 °C temperatures [12, 31-33]. Fig. 9 shows examples of natural and our fabricated nanostructured gas sensors.

To evaluate gas responses, we measure the differential optical reflectance spectral response $\Delta R(\lambda)$ of the sensing nanostructure upon exposure to gases [11, 12]. Such measurements accentuate spectral differences due to gas response and cancel common spectral features before and during gas exposure.

The $\Delta R(\lambda)$ spectra of *Morpho* scales to polar vapors (water, methanol, and ethanol) showed substantial spectral differences in the violet–blue regions (Fig. 10a–c), critical for multi-vapor differentiation using PCA (Fig. 10d) [11].

Bioinspired sensors were further fabricated for room and high temperature operation. Selectivity of fabricated nanostructured sensors was tested in numerous scenarios of increasing complexity.

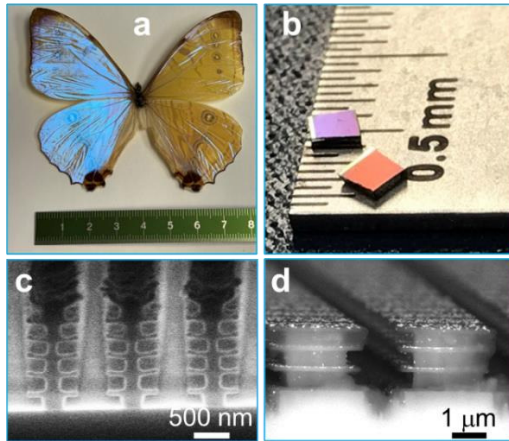


Fig. 9. Photonic nanostructured gas sensors: structural color of (a) natural *Morpho* butterfly and (b) fabricated sensors for (c) room and (d) high temperature sensing.

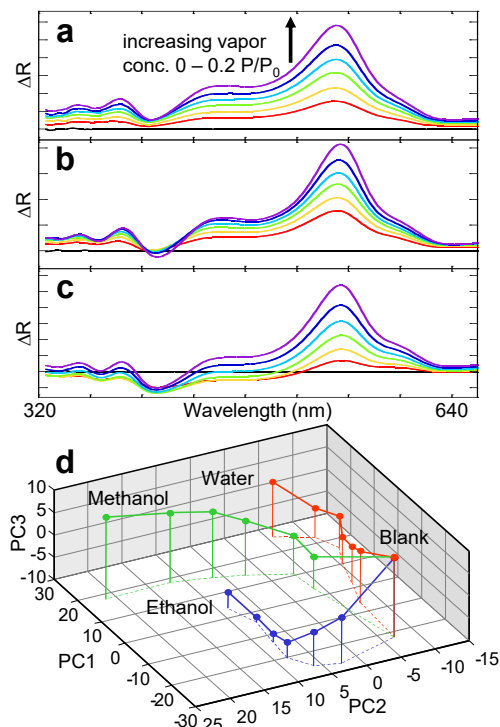


Fig. 10. *Morpho* nanostructure differentiates vapors: ΔR spectra upon (a) water, (b) methanol, and (c) ethanol exposures, concentrations of vapors were 0–0.2 P/P_0 (P is vapor partial pressure and P_0 is saturated vapor pressure), (d) pattern recognition using PCA score plot.

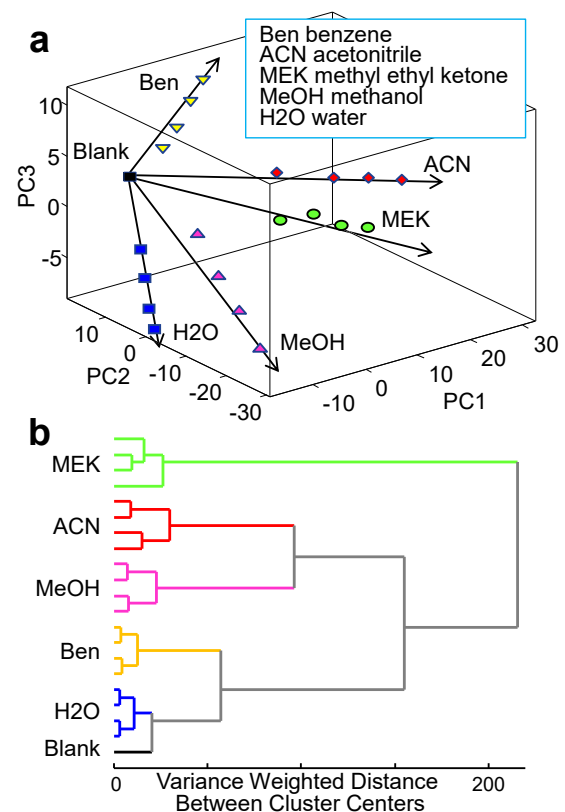


Fig. 11. Fabricated nanostructure differentiates vapors at room temperature using: (a) PCA score plot, (b) HCA dendrogram. Concentrations of vapors: 0 - 0.1 P/P_0 .

Tests with fabricated nanostructures at room temperature with vapors of diverse nature and polarity showed good multi-gas differentiation [12]. Besides PCA (Fig. 11a), hierarchical cluster analysis (HCA) machine learning technique was used (Fig. 11b). These sensors also resolved and quantified mixtures of methanol and ethanol with variable water vapor background [12].

High-temperature tests with gases of importance to solid oxide fuel cells [31, 33] were performed using H_2 and CO , with CO_2 , CH_4 , and H_2O as interferences at high concentrations. The PCA and HCA results differentiated all volatiles (Fig. 12).

Multi-wavelength illumination also allowed drift correction. In one such test, we used CO_2 as an analyte gas (22%, 44%, 67%, and 89%) mixed with H_2O vapor (5 % and 10 % RH). Over ~18 h of laboratory test, a noticeable baseline drift was observed. To eliminate drift effects, we utilized two transfer functions. One transfer function did not include appropriate wavelengths with effects of drift, predicting CO_2 concentrations well in the presence of H_2O vapor but did not perform well in the presence of the sensor baseline drift (Fig. 13a). The second transfer function included appropriate wavelengths with diverse effects of drift, resulting in dramatic improvement in prediction of CO_2 (Fig. 13b).

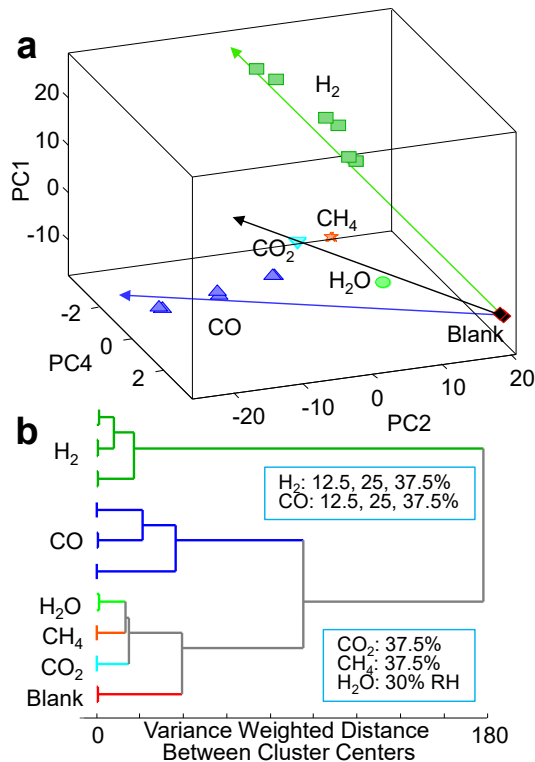


Fig. 12. Fabricated nanostructure differentiates H₂ and CO gases of interest and rejects CO₂, CH₄, and H₂O as interferences at 300 °C using: (a) PCA score plot, (b) HCA dendrogram.

To boost multi-gas resolution, we showed three sensor designs as second-order devices. In all three designs, the first independent variable in response was optical wavelength across the visible spectral range. The second independent variable was either illumination angle, light polarization, or sensor temperature [15].

Using illumination at multiple wavelengths and angles, we achieved an extraordinary sensor-response dispersion with 10 vapors. A *Morpho* nanostructure was illuminated at three angles θ of 20°, 40°, and 60° (Fig. 14) [15]. Fig. 14a-c

depicts spectral responses of the sensor to ten vapors as a function of wavelength λ and angle θ . The visual spectral diversity at different illumination angles was two-fold, with three general spectral regions of ~360–430 nm, ~430–490 nm, and ~490–600 nm with their respective inflection points and changing relative spectral intensities for each vapor with illumination angle. Fig. 14d shows the PCA score plot from data with combined three angles, differentiating ten vapors with seven principal components. This seven-dimensional (7-D) response dispersion is higher compared to 5-D dispersion with sensor arrays or individual first-order sensors [34, 35].

5. Conclusions and Path Forward

This paper provides an overview of approaches to increase the information content of gas sensor elements (a.k.a. sensors) without major changes to established fabrication processes of sensor systems that use these sensor elements [36].

Our two described types of sensors operate with dielectric excitation of metal oxides or with multi-wavelength illumination of nanostructured materials. These sensors are complementary in the nature of sensing materials and range of detected volatiles.

With radio-frequency sensors that utilize multi-frequency dielectric excitation of metal oxides, we show that using available ASIC impedance analyzers instead of resistance provides multi-gas differentiation, rejection of interferences, improved stability, and simplified calibration, all without an increase of system size and cost. With photonic sensors that utilize multi-wavelength illumination of nanostructured materials, we provide examples of multi-gas differentiation, rejection of environmental interferences, and improved stability.

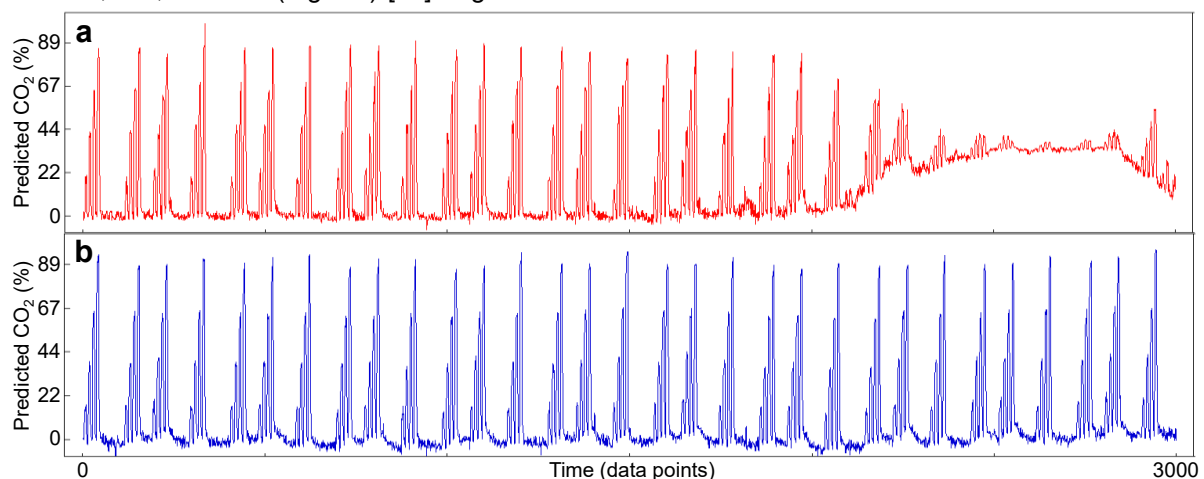


Fig. 13. Prediction of CO₂ concentrations in the presence of H₂O vapor and uncontrolled sensor drift using (a) first and (b) second transfer function. 3000 data points were collected over ~ 18 h of test.

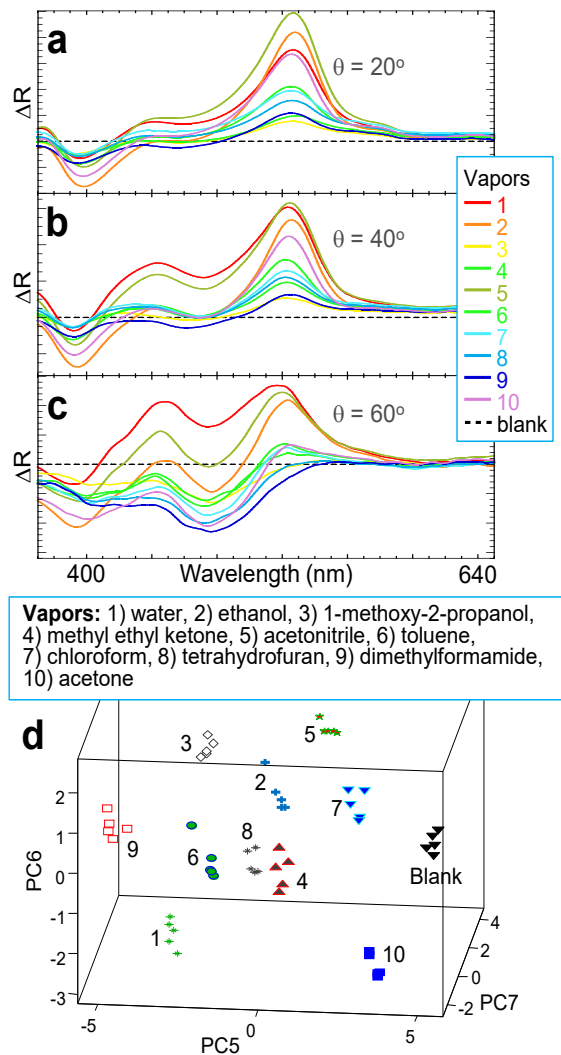


Fig. 14. Response of Morpho nanostructure to ten vapors as a function of λ and θ : (a-c) $\Delta R(\lambda, \theta)$ spectra at three angles, (d) PCA score plot with data analyzed with all three angles.

Our future work will focus on multigas quantification in complex backgrounds for industrial monitoring and diagnostics and for many other applications. Fig. 15 shows examples of our upcoming hardware and sensing materials. We are working to implement our recently designed and fabricated advanced ASIC impedance analyzers [37] (Fig. 15a) for an extended measurement frequency range. Our extraordinary 7-D response dispersion achieved with an individual photonic sensor inspired us to fabricate high-aspect ratio metamaterials (Fig. 15b) that we will couple with the second-order interrogation for exquisite multi-gas sensing. Finally, availability of micro-packaged multi-wavelength light sources originally developed for other applications (Fig. 15c) will propel miniaturization efforts of our photonic sensors.

Contemporary hardware now creates a practical opportunity to implement compact, energy-efficient, and affordable sensor systems [13, 19, 29, 38]. Unlike single-output, zero-order gas sensors, which provide limited response dimensionality, these new hardware designs generate multidimensional data better suited to modern machine learning tools. This hardware-analytics co-design opens a systematic path to real-time multi-analyte differentiation and rejection of interferences, drift correction, calibration transfer, and improved stability.

Our strategic approach of expanding the dimensionality of sensor response to the first- and second-order devices described in this overview will enable next-generation sensors to perform on par with traditional orthogonal-output analytical instruments, but with a continuous real-time data stream and unobtrusive, energy-efficient designs.

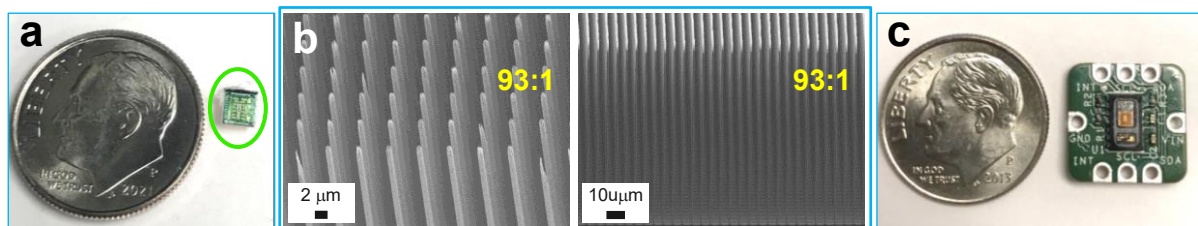


Fig. 15. Illustrative opportunities for future advances in first- and second-order individual gas sensors: (a) our recently designed and fabricated advanced ASIC impedance analyzer for extended frequency range of measurements, (b) electron microscopy images of nano-fabricated high-aspect ratio metamaterials, (c) micro-packaged multi-wavelength light source applicable for photonic sensors.

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