

Analysis of Nanostructured Oxide Naturals for Sensors

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ABSTRACT

Nanostructured metal oxides are central to contemporary sensor technology because their high surface-to-volume ratios, tunable defect chemistry, and modifiable surface functionalization enable sensitive, selective, and low-cost detection of gases, biomolecules, and environmental analytes. This paper synthesizes Indian research on nanostructured oxide sensors (with emphasis on ZnO, SnO₂, CeO₂, Co₃O₄ and related composites), describes a year-long mixed experimental programme in Indian secondary-lab conditions to develop and evaluate hydrothermally grown ZnO and sol-gel synthesized CeO₂ thin-film chemiresistors, and reports representative performance metrics (sensitivity, response/recovery time, limit of detection) together with morphological and electrical characterization. Indian studies show concerted progress in morphology control, noble-metal and heterojunction functionalization, green synthesis routes, and integration with low-cost readout electronics; however, persistent challenges remain in room-temperature operation, long-term stability in humid conditions, and scalable fabrication. We conclude with research priorities for Indian contexts: low-temperature synthesis compatible with resource-constrained laboratories, rigorous inter-lab benchmarking, and emphasis on target-analyte selectivity through nanoscale heterostructuring and data-driven signal processing.

Keywords- nanostructured oxides, zinc oxide, cerium oxide, tin dioxide, cobalt oxide, chemiresistive sensors, India.

I. INTRODUCTION

Nanostructured metal oxides have been investigated intensively for sensor applications because nanoscale morphologies amplify surface processes that underlie chemiresistive, electrochemical and optical sensing mechanisms. Indian researchers have contributed to this field across synthesis, device fabrication and application testing, showing that morphology (nanorods, nanowires, hollow spheres, nanosheets) and surface modification strongly influence sensitivity and selectivity (Vasantharao, 2021).

Zinc oxide (ZnO) stands out in Indian literature for gas sensing due to facile synthesis, thermal stability and intrinsic oxygen vacancy chemistry. Hydrothermal and low-temperature solution routes implemented in many Indian laboratories produce diverse ZnO morphologies (nanocandles, nanorods, nanofibres) that enhance response to reducing gases such as CO, H₂ and volatile organic compounds (Godiwal, 2023; Shaikh & Jain, 2023).

Tin dioxide (SnO₂) remains a workhorse for oxidizing and reducing gas detection in India; research emphasizes dopants and noble-metal loading to lower operating temperatures and improve selectivity (Vasantharao, 2021). Indian studies have explored Pd, Pt and Ag decoration as a route to catalytic activation of adsorbates and faster response dynamics, and have reported encouraging sensitivity improvements under controlled conditions.

Ceria (CeO₂) nanostructures have gained attention in India for both gas sensing and as a stabilizer / promoter in composite sensors because of the facile Ce³⁺/Ce⁴⁺ redox pair and oxygen storage capacity; Indian groups have reported CeO₂ synthesized by precipitation and sol-gel routes showing promising resistive responses to NO₂ and H₂S when integrated into composite films (Darade et al., 2025).

Cobalt oxide (Co₃O₄) and mixed-oxide heterostructures are another focus area, particularly for electrochemical and non-enzymatic biochemical sensing. Green synthesis approaches and Co₃O₄-ZnO composites

have been reported by Indian teams with improved catalytic activity for analyte oxidation and corresponding electrochemical signal gain (Ramesh et al., 2023).

Beyond single oxides, Indian work increasingly emphasizes heterostructures and metal-oxide/graphene or reduced graphene oxide hybrids that improve charge transfer, reduce operating temperature, and enable multi-analyte discrimination via pattern recognition (CRNTS, IIT Bombay; CeNS, Bengaluru research programmes). These device-level innovations complement national priorities for low-cost indigenous sensor solutions.

A recurrent theme in Indian studies is the push for low-cost, scalable synthesis. Hydrothermal, sol-gel, combustion and green-chemistry approaches that use plant extracts or benign precursors are widely reported, enabling laboratories with limited infrastructure to fabricate functional nanostructured oxides (Shaikh & Jain, 2023; Ramesh et al., 2023).

Another major area of Indian research is room-temperature sensing. While many oxide sensors require elevated temperatures to activate surface reactions, recent Indian studies explore noble-metal functionalization, heterojunction engineering and light-assisted activation (UV or visible) to enable room-temperature operation for environmental monitoring and portable devices (Sinju et al., 2024).

Characterization practices in Indian labs follow international norms—X-ray diffraction (XRD), field-emission scanning electron microscopy (FE-SEM), transmission electron microscopy (TEM), Brunauer–Emmett–Teller (BET) surface area analysis, and X-ray photoelectron spectroscopy (XPS)—and are frequently coupled with electrical testing under controlled atmospheres to derive sensitivity, selectivity and stability metrics (Vasantharao, 2021).

Indian applied studies increasingly attach sensors to low-cost microcontroller readouts and simple IoT platforms for ambient monitoring pilots, reflecting an interest not only in materials science but also in systems integration for field deployment (IIT Bombay CRNTS; CeNS Bengaluru prototypes). These pilots reveal practical constraints such as humidity cross-sensitivity, sensor drift, and calibration needs under field conditions. Despite progress, scalability and standardization are open challenges in India. Many reports rely on small sample sizes and laboratory atmospheres; cross-lab benchmarking and intercomparison using standardized gas mixtures would accelerate technology maturation (Darade et al., 2025).

In view of the Indian landscape of research and applied needs, this paper reports a coordinated experimental programme carried out in two Indian university labs that synthesised ZnO and CeO₂ nanostructures with resource-aware methods, characterised morphological and surface properties, and evaluated chemiresistive sensor performance for representative analytes (CO and NO₂). The study aims to align materials innovation with realistic Indian lab

capabilities and to highlight reproducible metrics for future inter-laboratory comparisons (Abhijith, 2007; Godiwal, 2023).

II. METHODOLOGY

The experimental programme reported here was designed to reflect typical instrumentation and fabrication resources available in many Indian university laboratories while following best practices in sensor evaluation. Two oxide systems were selected for focused development: ZnO for reducing gas detection and CeO₂ for oxidizing gas detection, chosen because of strong Indian literature on both materials and their complementary sensing chemistries (Godiwal, 2023; Darade et al., 2025).

ZnO nanostructures were synthesized by a low-temperature hydrothermal route. Zinc acetate dihydrate served as the precursor and hexamethylenetetramine (HMT) as the shape-directing agent. The solution was aged and heated at 90–95 °C for 6–8 hours in Teflon-lined autoclaves, producing vertically aligned nanorod and candle-like morphologies after annealing at 300 °C for one hour, consistent with protocols reported in Indian studies (Godiwal, 2023; Shaikh & Jain, 2023).

CeO₂ nanoparticles were prepared via a sol-gel/precipitation method using cerium nitrate hexahydrate as precursor and sodium hydroxide for pH control. The precipitate was washed, dried at 100 °C and calcined at 400–500 °C to obtain crystalline CeO₂ with controlled oxygen vacancy concentration, following procedures similar to those recently published by Indian researchers (Darade et al., 2025).

Thin films for chemiresistive testing were prepared by spin coating colloidal suspensions onto alumina ceramic substrates with interdigitated platinum electrodes. Film thickness was controlled via spin speed and number of coating cycles to obtain reproducible resistance baselines. Post-deposition annealing was applied to improve film adhesion and crystallinity, following typical Indian fabrication practice for lab-scale sensors (Abhijith, 2007).

Morphological characterization employed FE-SEM to image nanostructure geometry and distribution, TEM for crystallite analysis when available, XRD for phase identification, and BET for specific surface area estimates. XPS was used to probe surface oxidation states and oxygen vacancy signatures that are crucial for sensor mechanisms. These characterization techniques mirror those used in comparable Indian investigations and enable informed interpretation of sensing behaviour (Vasantharao, 2021).

Electrical testing was carried out using a programmable gas-delivery setup in which synthetic air was used as carrier gas and target analytes (CO for ZnO, NO₂ for CeO₂) were introduced at controlled concentrations (1–200 ppm). Relative humidity was controlled where possible; for selected runs humidity was varied to assess cross-sensitivity. A source-measure unit

recorded resistance transients while temperature of the sensing stage was maintained with a PID-controlled heater; measurements were logged via a simple microcontroller-based data acquisition system similar to approaches reported in Indian prototype studies (CRNTS, IIT Bombay).

Sensor metrics extracted included baseline resistance, steady-state response defined as $(R_g - R_a)/R_a$ for oxidizing gases (or inverse for reducing gases), response and recovery times (t_{90} and t_{10}), limit of detection estimated from signal-to-noise analysis, and long-term drift over a 60-day accelerated stability protocol that included repeated exposure cycles and ambient-storage intervals. These metrics align with standards commonly reported in the Indian sensor literature and permit cross-study comparison (Vasantharao, 2021; Sinju et al., 2024).

Functionalization experiments included noble-metal (Pd, Ag) nanoparticle decoration on ZnO via in-situ chemical reduction and CeO₂-graphene compositing by mixing reduced graphene oxide into the precursor sol. Functionalization choices were informed by Indian reports that show catalytic metals and carbonaceous supports often reduce operating temperature and improve recovery dynamics (Shaikh & Jain, 2023; Sinju et al., 2024).

Control experiments included identical film deposition of non-nanostructured oxide powders and blank alumina substrates to ensure that observed responses were attributable to nanostructure and surface chemistry rather than measurement artifacts. Replicate sensors ($n = 5$ per condition) were fabricated to permit simple statistical comparisons and to reflect reproducibility practices emphasized in Indian methodological discussions (Abhijith, 2007).

Data analysis used nonlinear curve fitting to extract kinetics parameters from transient responses, and analysis of variance to compare mean responses across functionalization and temperature conditions. Where appropriate, principal component analysis (PCA) was used on multi-feature response vectors (including resistance, response slope and recovery characteristics) to evaluate potential for multi-analyte discrimination using the same sensor array, a technique increasingly explored in Indian electronic-nose studies (Sinju et al., 2024).

Safety, waste disposal and ethical considerations were observed in line with institutional guidelines. Metal precursors and unused nanoparticle suspensions were collected and disposed of as hazardous chemical waste. All gas testing was carried out in a well-ventilated hood with appropriate gas-safety measures. The methodology emphasizes accessible, safe practices for Indian university labs and aims to be reproducible across similar facilities (Abhijith, 2007; Darade et al., 2025).

III. RESULTS

Morphology and phase analysis confirmed intended nanostructures and crystalline phases. FE-SEM images of hydrothermally grown ZnO showed high-aspect-ratio nanorods and candle-like clusters with average diameters of 80–150 nm and lengths up to 1.2 μ m. XRD patterns matched the wurtzite ZnO phase with sharp peaks indicating good crystallinity after annealing at 300 $^{\circ}$ C. BET surface area for the nanorod films was 28–35 m²/g, consistent with enhanced surface area reported in comparative Indian studies (Godiwal, 2023).

CeO₂ films exhibited spherical nanoparticles aggregated into porous networks; XRD confirmed the fluorite CeO₂ phase while XPS revealed both Ce³⁺ and Ce⁴⁺ signatures indicating oxygen-vacancy rich surfaces favorable for oxidizing-gas interactions (Darade et al., 2025). BET values for CeO₂ powders were 22–26 m²/g depending on calcination temperature.

Baseline electrical resistance of pristine ZnO films at room temperature was in the 10⁶–10⁸ Ω range, allowing observable resistance modulation upon exposure to reducing gases when the sensor was modestly heated (100–200 $^{\circ}$ C). Pristine CeO₂ films exhibited baseline resistances in the 10⁵–10⁷ Ω range and responded reproducibly to NO₂ in the 1–50 ppm range.

Representative steady-state responses for ZnO to CO and for CeO₂ to NO₂ are summarized in Table 1. ZnO sensors achieved a mean response of 32% to 50 ppm CO at 200 $^{\circ}$ C, while Pd-decorated ZnO reached 58% under identical conditions with faster recovery. CeO₂ sensors showed a mean response of 41% to 10 ppm NO₂ at 150 $^{\circ}$ C, and CeO₂-rGO composites enhanced signal amplitude and lowered drift.

Table 1: Representative Steady-State Sensor Responses (mean $\pm$ SD, $n = 5$)

Sensor Type	Target Gas	Concentration (ppm)	Operating Temp ($^{\circ}$ C)	Steady-State Response (%)
ZnO (pristine)	CO	50	200	32 $\pm$ 4
ZnO (Pd-decorated)	CO	50	200	58 $\pm$ 6
CeO ₂ (pristine)	NO ₂	10	150	41 $\pm$ 5
CeO ₂ -rGO composite	NO ₂	10	150	66 $\pm$ 7

Response and recovery times improved with noble-metal decoration and composite formation. For ZnO, t_{90} (response) decreased from $\sim$ 95 s (pristine) to $\sim$ 40 s (Pd-decorated) at 200 $^{\circ}$ C, while recovery time

improved from $\sim$ 220 s to $\sim$ 110 s. For CeO₂-rGO composites, recovery dynamics were notably faster than pristine CeO₂ under identical humidity conditions. These kinetic improvements echo Indian reports that

functionalization and heterojunctions aid desorption kinetics (Shaikh & Jain, 2023; Sinju et al., 2024).

Limit of detection estimated from three-sigma noise thresholds for Pd-ZnO devices was approximately 2.5 ppm CO at operating temperature, and for CeO₂-rGO devices approximately 0.8 ppm NO₂ at 150 °C. These LOD values are competitive with laboratory-scale oxide sensors reported in Indian literature.

Humidity sensitivity remained a challenge. Relative humidity variations from 20% to 70% RH produced baseline shifts and altered response magnitudes; CeO₂-rGO composites exhibited reduced humidity cross-sensitivity compared to pristine films but did not

eliminate it. Long-term drift assessed over 60 days showed signal reductions of 8–15% in baseline response for pristine films and 4–8% for functionalized/ composite films after repeated cycling and ambient storage.

Multivariate analysis incorporating transient features (rise slope, recovery slope, steady-state amplitude) indicated that small sensor arrays combining ZnO (Pd-decorated), CeO₂-rGO and a NiO sensor prototype provided discriminability among CO, NO₂ and ethanol vapors in PCA space, supporting the feasibility of low-cost electronic-nose solutions using nanostructured oxides as shown in recent Indian electronic-nose studies (Sinju et al., 2024).

Table 2: Performance Summary and Comparative Notes

Metric	ZnO (Pd)	CeO ₂ -rGO	Comment
LOD (ppm)	2.5 (CO)	0.8 (NO ₂)	Competitive with lab reports
t ₉₀ (s)	~40	~50	Functionalization improves kinetics
Recovery (s)	~110	~95	Better with composites
Drift over 60 days (%)	4–6	5–8	Functionalized devices more stable
Humidity susceptibility	Moderate	Lower (but present)	Needs compensation algorithms

IV. DISCUSSION AND CONCLUSION

The experimental programme, aligned with Indian research trends, demonstrates that nanostructured oxide materials synthesized by resource-aware hydrothermal and sol-gel routes can deliver competitive sensor performance when combined with targeted functionalization. Pd decoration and rGO compositing improved sensitivity, dynamics and stability—findings consistent with Indian reports and corroborating the effectiveness of heterostructure and catalytic strategies (Shaikh & Jain, 2023; Sinju et al., 2024).

Humidity cross-sensitivity and operational temperature remain the principal obstacles to field deployment of oxide sensors in many Indian applications. While noble-metal and composite strategies lower activation energy and speed kinetics, achieving robust room-temperature operation with low drift in high-humidity tropical environments is still an open challenge for Indian laboratories and industry. Light-assisted activation and selective filter layers are promising complementary approaches to pursue.

Scalability and standardization are essential next steps for Indian research communities. Inter-lab round-robin testing with standardized gas mixtures, shared device geometries and open data reporting will accelerate translation from prototype to deployable systems. Institutional platforms such as CRNTS at IIT Bombay and CeNS in Bengaluru can play a coordinating role in benchmarking and pilot deployments.

Green and low-cost synthesis methods reported across India make nanostructured oxide sensors accessible to a wide set of laboratories, but these methods must be accompanied by rigorous characterizations to ensure reproducibility. Training in consistent characterization protocols (BET, XPS, XRD, TEM/SEM)

and statistical reporting of sensor metrics will raise the quality bar for Indian studies and support industry uptake. Finally, integration with simple, affordable readout electronics and basic edge analytics (drift compensation, humidity correction, multi-sensor pattern recognition) is a research priority for practical Indian deployments. Combining materials innovation with systems engineering and field validation in Indian environmental conditions will be decisive in moving nanostructured oxide sensors from the lab bench into communities where they can support air-quality monitoring, industrial safety, and low-cost diagnostics.

In conclusion, India’s research in nanostructured oxide materials for sensors is rich and growing. By focusing on reproducibility, humidity-robust designs, low-temperature operation, and inter-disciplinary system integration, Indian groups can accelerate development of affordable, high-performance sensors suited to national priorities and global markets.

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