

Review

MoO₃- and WO₃-Based Chemiresistive Sensors for Triethylamine Detection: Material Engineering, Sensing Mechanisms and Performance Enhancement

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Abstract

Triethylamine (TEA) is a common industrial contaminant and an important indicator of seafood spoilage. Therefore, determination of rapid and selective TEA is of great significance. This review article critically compares tungsten oxide (WO₃)- and molybdenum oxide (MoO₃)-based chemiresistive sensors by relating their crystal structure, surface chemistry, defect states, morphology, and interfacial electronic properties to TEA-sensing performance. Pristine WO₃- and MoO₃-based sensors generally operate at approximately 133–325 °C and provide sub-ppm detection, whereas doping, noble-metal sensitization, heterojunction formation, and light activation can reduce the operating temperature to 100–180 °C and extend detection into the low-ppb range. WO₃-based sensors have exhibited a response of 1100 to 20 ppm TEA at 160 °C, with an estimated detection limit of 5 ppb, whereas modified MoO₃-based sensors have also achieved decent detection limit of 1.7 ppb. WO₃ is particularly responsive to phase, facet, work-function, and catalytic-interface engineering, whereas α-MoO₃ benefits from its anisotropic structure, variable Mo valence, and favorable Lewis acid–base interactions with amines. Noble metals enhance gas sensing through catalytic and electronic sensitization, dopants regulate adsorption and defect chemistry, and n-n or p-n heterojunctions amplify resistance changes through depletion-layer modulation. Despite considerable advances in sensitivity, humidity interference, high power consumption, slow recovery, baseline drift, and limited long term stability remain unresolved. Future advances may require standardized performance assessment, operando mechanistic studies, humidity-resistant low-power devices, and validation under realistic seafood-storage and industrial conditions.

Keywords: triethylamine; tungsten trioxide; molybdenum trioxide; chemiresistive gas sensor



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1. Introduction

Triethylamine (TEA) is a volatile organic amine which is extensively used as a solvent, catalyst, curing agent, corrosion inhibitor, and synthetic intermediate in various industrial processes [1–3]. Its high volatility, flammability, and irritant properties raise industrial and environmental safety concerns in the event of accidental release or prolonged exposure [4–6]. Volatile amines are important indicators of seafood spoilage, while TEA

is frequently employed as a model analyte for evaluating gas-sensor performance [7–10]. Accordingly, the rapid detection of TEA at low concentrations is essential for industrial monitoring, environmental assessment, and food-quality control. Conventional analytical approaches used for amines and organic pollutants, including gas chromatography–mass spectrometry and spectroscopic or chemometric methods, provide strong analytical capability but often require specialized instrumentation, trained personnel, and multistep data processing [11–14]. Chemiresistive sensors provide a simpler alternative by converting gas–surface interactions into measurable changes in electrical resistance [15]. Metal oxide semiconductors are attractive materials because of their chemical robustness, low material cost, straightforward fabrication, and compatibility with miniaturized devices [16–18]. Tin oxide (SnO₂) and zinc oxide (ZnO) remain widely used sensing materials [2,5]. However, their response to reducing volatile compounds often arises from broadly reactive surface oxygen species, which can produce cross-sensitivity and pronounced humidity dependence. WO₃ and MoO₃ provide alternative promising sensing platforms because their phase composition, metal valence, oxygen deficiency, Lewis acidity, and surface electronic structure can be adjusted over comparatively broad ranges [19–23]. These properties of WO₃ and MoO₃ enable TEA adsorption and charge-transfer behavior to be tuned through surface chemistry and bulk or interfacial transport, rather than solely by increasing the surface area.

Although WO₃ and MoO₃ are both n-type transition-metal oxides, their structures exhibit distinct TEA-sensing behavior. WO₃ consists mainly of distorted, corner-sharing WO₆ octahedra and can adopt several polymorphs with different facet energies, defect populations, and work functions. Its sensing response is therefore particularly sensitive to crystal phase, exposed facets, grain boundaries, and W⁵⁺/W⁶⁺ redox chemistry. Orthorhombic α -MoO₃, in contrast, has an anisotropic layered structure containing chemically inequivalent oxygen sites. This arrangement favors direction-dependent transport, surface reduction involving Mo⁶⁺/Mo⁵⁺ states, and Lewis acid–base interactions between coordinatively unsaturated Mo centers and the electron-donating nitrogen atom of TEA [19–23]. Therefore, WO₃ is generally more responsive to phase, facet, and work-function engineering, whereas α -MoO₃ provides greater opportunities to regulate amine adsorption through its layered structure, oxygen-site diversity, and variable Mo valence. This distinction provides the central basis for comparing the two oxide families rather than treating them as interchangeable chemiresistive materials. In pristine n-type WO₃ and MoO₃, the sensing process begins with the adsorption of oxygen molecules, which capture electrons from the oxide and form a surface electron-depletion layer, thereby increasing the baseline resistance. The reaction of TEA with adsorbed oxygen species releases trapped electrons back into the conduction band, thereby lowering the sensor resistance. The magnitude and rate of this resistance change depend on the concentration and chemical nature of the reactive oxygen species, the adsorption strength of TEA, the activation of C–N and C–H bonds, and the width of the depletion layer. However, the predominant mechanism responsible for improving the sensing response varies with the material design. Noble metals enhance catalysis and spillover and can form Schottky junctions with the oxide. Dopants modify Lewis acidity, carrier density, electron trapping, and oxygen vacancies, while heterojunctions regulate depletion layers through interfacial charge redistribution. Morphology, porosity, facets, and grain boundaries further control gas diffusion, adsorption, and intergrain barriers. These mechanisms should be identified separately rather than described broadly as “synergistic effects” or “enhanced charge transfer.” In previous years, gasochromic studies of WO₃ showed that oxygen stoichiometry is a functional parameter rather than simply a structural defect. Several studies reported that the optical coloration of sub-stoichiometric WO_{3- γ} films depended strongly on oxygen content, W

valence, and deposition conditions, with similar trends in gasochromic and electrochromic behavior [24–28]. Although hydrogen-induced coloration differs from chemiresistive TEA sensing, these findings demonstrate that oxygen stoichiometry can modify the electronic structure and surface reactivity of WO_3 , thereby affecting sensor response and reversibility. Oxygen vacancies should therefore be assessed by their concentration, charge state, location, and stability rather than treated as an explanation for improved performance. WO_3 - and MoO_3 -based materials have also been investigated for ammonia, nitrogen dioxide, carbon dioxide, hydrogen sulfide, and other gases [29–40]. Previous reviews summarize morphology and interface engineering in WO_3 - and MoO_3 -based gas sensors [41–43]. Beyond chemiresistive detection, MoO_3 -based materials have also been developed as electrode modifiers for electrochemical sensing [44]. Gas-sensing performance has been enhanced through elemental modification, heterojunction formation, and integration with polymers or two-dimensional materials [45–47]. Nevertheless, many reported sensors still require high operating temperatures and are evaluated under simplified laboratory conditions. Water molecules compete for adsorption sites, modify surface oxygen chemistry, and may cause substantial signal attenuation. Over prolonged operation, grain growth, irreversible surface hydroxylation, noble-metal agglomeration, contact degradation, and changes in defect populations can gradually cause baseline drift and reduce sensing sensitivity [48]. These limitations are especially relevant to seafood monitoring, where temperature, humidity, and the composition of volatile amines can vary continuously during storage and spoilage. The publication trends for WO_3 - and MoO_3 -based sensors are summarized in Figure 1. Previous reviews have discussed WO_3 - and MoO_3 -based sensors across broad analyte classes [41–43,49,50], but a focused comparison of their TEA-sensing behavior remains limited.

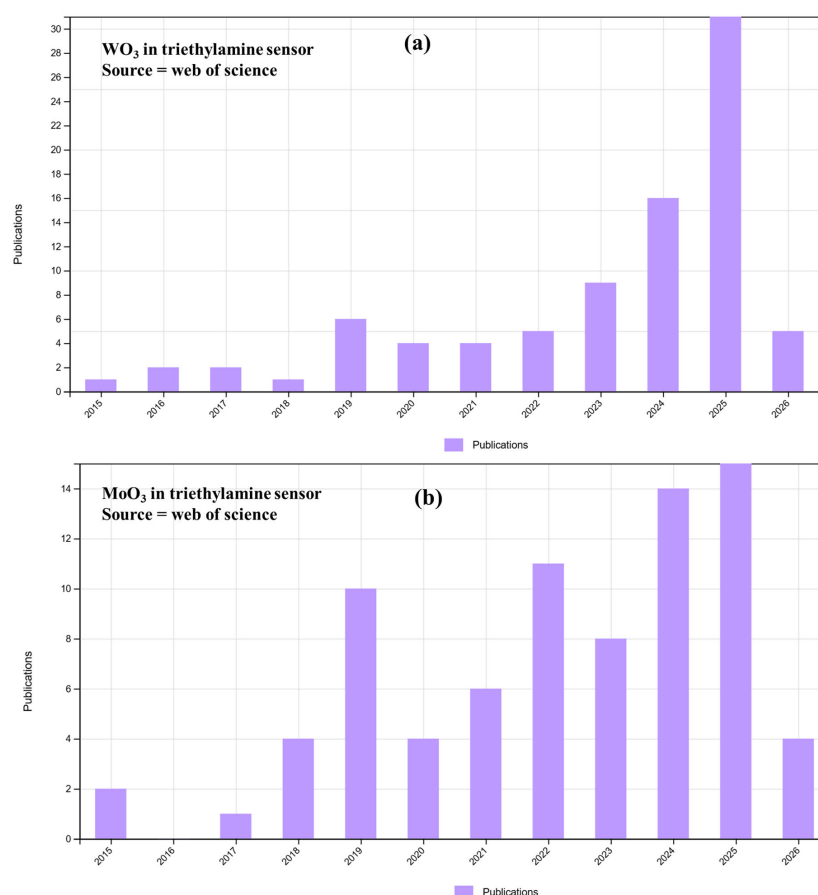
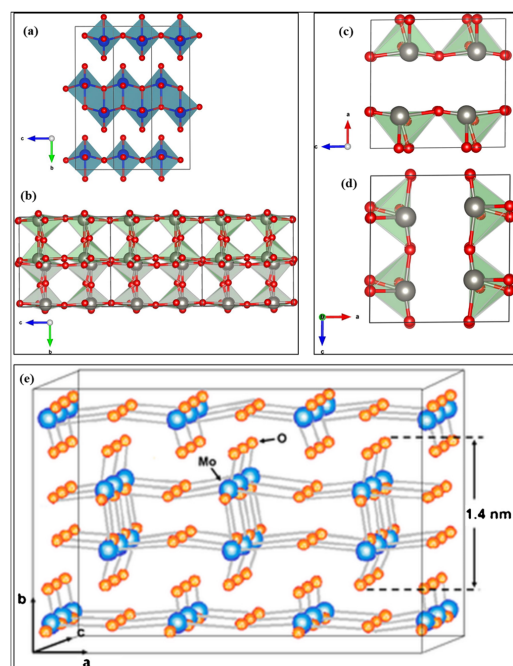


Figure 1. Bar graphs show the literature on (a) WO_3 - and (b) MoO_3 -based TEA sensors. Source: Web of Science.

This review relates synthesis-dependent structure, intrinsic oxide chemistry, and modification strategy to operating temperature, response, detection limit, response/recovery kinetics, selectivity, humidity tolerance, and long-term stability. The mechanistic differences between WO_3 and MoO_3 , distinct roles of dopants, noble metals, and heterojunctions, and the transition from conventional ceramic-tube sensors toward the fabrication of low-power, miniaturized, and flexible devices have been mentioned. The remaining challenges are considered in terms of mechanistic validation, standardized benchmarking, humidity-resistant operation, device aging, field calibration, and realistic industrial and seafood-storage applications.

2. Structure and Properties of WO_3 and MoO_3

The structures of MoO_3 and WO_3 are shown in Scheme 1 [18,20]. Scheme 1a–d compare the crystal frameworks and crystallographic orientations of α - MoO_3 and monoclinic WO_3 . Scheme 1a shows orthorhombic α - MoO_3 in which distorted MoO_6 octahedra form layers stacked along the b -axis. This layered arrangement produces pronounced structural anisotropy and may influence charge transport, gas diffusion and the accessibility of surface adsorption sites. In contrast, Scheme 1b presents monoclinic γ - WO_3 as a ReO_3 -related 3D framework composed of interconnected, distorted WO_6 octahedra. The three-dimensional WO_3 framework and its electrochromic-material context were comprehensively described by Granqvist [51]. Although Mo as well as W are surrounded by oxygen atoms in octahedral arrangements, these octahedra connect differently, resulting in the layered α - MoO_3 structure and the 3D monoclinic WO_3 framework. Scheme 1c,d show b -axis projections of the monoclinic WO_3 unit cell and illustrate the effect of crystallographic orientation. In Scheme 1c, the $(h\ 0\ 0)$ planes are oriented horizontally, placing the W atoms within closely packed planes parallel to the substrate. This configuration was associated with the preferred (200) orientation of WO_3 films annealed at 400 °C. In Scheme 1d, the $(0\ 0\ 1)$ planes are horizontal and the W atoms are distributed between two parallel sets of planes, corresponding to the preferred (002) orientation observed after annealing at 500 °C. The source study reported approximately 24 vol.% of (200) oriented crystallites at 400 °C and 27 vol.% of (002) oriented crystallites at 500 °C. These observations demonstrate that thermal processing can modify the preferred orientation without changing the underlying monoclinic phase. Such orientation-dependent surface terminations may affect oxygen-vacancy formation, TEA adsorption and resistance modulation, although the sensing response also depends on morphology, stoichiometry and interfacial structure. Scheme 1e further illustrates the anisotropic layered structure of orthorhombic α - MoO_3 . The framework is composed of distorted MoO_6 octahedra assembled into double layers. Within each double layer, the octahedra form corner-sharing rows along the a -axis and edge-sharing zigzag rows along the c -axis. The double layers are stacked along the b -axis and are held together by comparatively weak van der Waals interactions, whereas strong covalent and ionic Mo–O bonds stabilize the structure within each layer [20]. The indicated distance of approximately 1.4 nm corresponds to the crystallographic repeat along the b -axis ($b \approx 13.858\ \text{\AA}$), whereas an individual layer has a thickness of approximately 0.7 nm [20]. This pronounced structural anisotropy facilitates the formation of ultrathin α - MoO_3 flakes with a high surface-to-volume ratio. In TEA chemiresistive sensors, such a layered morphology may improve access to surface and edge adsorption sites, thereby enhancing the modulation of surface charge and electrical resistance upon exposure to TEA.



Scheme 1. Structures of the (a) orthorhombic α - MoO_3 , and (b) monoclinic γ - WO_3 phases. Projection of the monoclinic WO_3 unit cell along the b -axis, showing (c) the $(h\ 0\ 0)$ planes and (d) the $(0\ 0\ 1)$ planes oriented horizontally. (e) Layered crystal structure of orthorhombic α - MoO_3 in which the blue and orange spheres denote Mo and O atoms, respectively. Reproduced with permission [18,20].

The X-ray diffraction (XRD) technique is widely used to investigate the crystallinity and preferred orientation of nanostructured materials. In Figure 2a, the broad feature centered near 24° for the as-deposited and 300°C annealed WO_3 films indicates a predominantly amorphous, short-range-ordered structure. Annealing at 400°C promotes the formation of monoclinic WO_3 (ICDD 01-072-0677) with a preferred (200) orientation. At 500°C , the monoclinic phase is preserved, but the dominant orientation shifts to (002), together with an increase in coherent domain size from approximately 50 to 65 nm [18]. In contrast, Figure 2b exhibits sharp reflections characteristic of orthorhombic α - MoO_3 (ICDD 00-005-0508). Such crystallinity and texture evolution can critically regulate active-surface exposure, grain-boundary transport, and ultimately the gas-sensing response. Raman spectroscopy may also provide significant information on local metal–oxygen bonding and short-range structural disorder. Studies of WO_3/MoO_3 thin films and amorphous MoO_3 have shown that Raman features may be sensitive to phase, crystallinity, and photochromic structural changes [52,53].

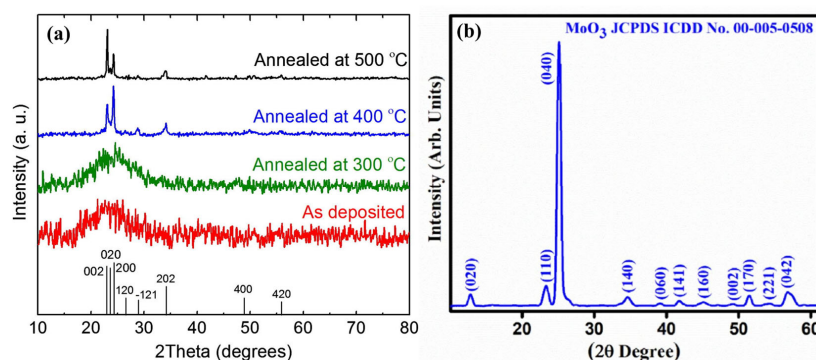


Figure 2. XRD patterns of (a) WO_3 thin films in the as-deposited state and after annealing at 300, 400, and 500°C and (b) sputter-grown nanocrystalline MoO_3 thin film. Reproduced with permission [18,32].

The properties of WO₃ and MoO₃ for TEA sensing are summarized in Table 1.

Table 1. Intrinsic properties and representative TEA sensing characteristics of pristine WO₃ and MoO₃.

Parameter	WO ₃	MoO ₃	Expected Role in TEA Sensing
Stable crystal structure	Monoclinic γ -WO ₃ , commonly indexed to $P2_1/n$ at room temperature	Orthorhombic α -MoO ₃ , commonly described using the equivalent $Pbnm/Pnma$ settings	Crystal symmetry determines exposed facets, oxygen coordination, and carrier transport [41,43].
Structural architecture	Three-dimensional network of distorted WO ₆ octahedra	Double layers of distorted MoO ₆ octahedra separated by weak van der Waals gaps	The layered structure of α -MoO ₃ produces greater transport and surface anisotropy.
Optical bandgap	Approximately 2.6–2.8 eV	Approximately 2.9–3.2 eV	The values depend on phase, stoichiometry, and defect concentration [41,43]. Degree of crystallinity also affects the bandgap, as reported for sputter-deposited (PVD) and CVD-deposited WO ₃ thin films [54–56].
Electronic characters	n-type; oxygen vacancies and W ⁵⁺ states contribute donor levels	n-type; oxygen vacancies and Mo ⁵⁺ states contribute donor levels	In both oxides, adsorbed oxygen withdraws electrons, whereas TEA oxidation returns electrons and lowers the resistance.
Lewis acidity and TEA interaction	Undercoordinated W ⁶⁺ centers act as Lewis-acid sites; their density varies with facet and defect structure	Exposed Mo ⁶⁺ centers provide Lewis-acid sites, complemented by terminal and bridging oxygen species	TEA can coordinate through the nitrogen lone pair, promoting selective adsorption and subsequent oxidation [47].
Principal intrinsic variables	Crystal phase, exposed facet, W ⁵⁺ /W ⁶⁺ ratio, oxygen-vacancy density, grain boundaries, and work function	Exposed facet, layer edges, inequivalent oxygen sites, Mo ⁵⁺ /Mo ⁶⁺ ratio, interlayer transport, and oxygen-vacancy density	These variables change together during synthesis and should be controlled when comparing sensing mechanisms.
Principal limitation	Relatively high operating temperature and humidity-sensitive surface chemistry	Limited humidity data, anisotropic transport, and possible slow recovery when TEA binds strongly	Long-term drift, calibration stability, and recovery under repeated exposure remain important for both materials.

3. Synthesis Methods for WO₃- and MoO₃-Based Materials

3.1. Hydrothermal Synthesis

Hydrothermal synthesis is a widely used method for preparing WO₃- and MoO₃-based nanomaterials. During synthesis, temperature, pH, reaction time, solvent composition, and the choice of mineralizing or structure-directing agent influence precursor hydrolysis, nucleation, crystal growth, and phase formation. Compared with conventional solid-state processing, this method can form hierarchical architectures at relatively moderate temperatures and also support direct deposition on conductive substrates or the in situ formation of multicomponent interfaces. The main advantage of this method is that it can tune the phase, morphology, exposed facets, and defect chemistry, rather than morphology alone. In WO₃, hydrothermal conditions also govern crystal growth and surface orientation. Chen et al. [57] varied the treatment temperature from 90 to 180 °C in an acidic sodium tungstate–tartaric acid system and obtained WO₃ structures with different morphologies and exposed crystal planes. This suggested that temperature-dependent nucleation and growth can be used to regulate surface characteristics relevant to gas adsorption.

Wang et al. [58] also adopted the approach to obtain Ag-modified WO₃ nanoflowers, as illustrated in Figure 3a. In another study, Sayed et al. [59] obtained hydrated WO₃ films on fluorine-doped tin oxide (FTO) substrates without a seed layer and found that deposition time controlled film thickness and morphology. Nevertheless, annealing at 550 °C for 4 h was required to obtain γ -monoclinic WO₃. Although hydrothermal deposition simplifies film formation and substrate integration, it cannot be considered a low-thermal-budget process if crystallization still requires post-deposition annealing. Jian et al. [60] prepared hierarchical urchin-like WO₃ at 180 °C for 4 h, with the particles assembled from needle-like nanorods (NRs). Such open architectures are favorable for gas diffusion, although their sensing performance should be distinguished from changes caused by crystallinity, facet exposure, or defect concentration. Hydrothermal processing also enables substantial phase and dimensional control in MoO₃. Khan et al. [61] prepared MoO₃ over a temperature range of 140–230 °C using sodium molybdate, hydroxylamine hydrochloride, and cetyltrimethylammonium bromide under acidic conditions. A pure hexagonal phase was obtained at 200 °C, indicating that hydrothermal temperature can direct phase evolution when precursor speciation and solution chemistry are fixed. In contrast, Zima et al. [62] obtained orthorhombic α -MoO₃ nanobelts from a peroxomolybdenum precursor at 180 °C for 6 h. These observations show that the final MoO₃ phase depends not only on temperature but also on precursor chemistry, acidity, additives, and the reaction pathway. The method has further been used to construct α -MoO₃/g-C₃N₄ composites [63] and ZnMoO₄ decorated MoO₃ heterostructures [64]. Hydrothermal synthesis can tailor phase, porosity, exposed facets, and hetero-interfaces. These features may improve TEA diffusion, adsorption, and charge transport. However, their effects are difficult to separate because crystallinity, particle size, defect density, and morphology often change together. Long reaction times, batch processing, washing, acidic media, and post-annealing may also limit reproducibility and scale-up. Controlled studies should therefore isolate key variables, report structural and fabrication parameters, and compare as-prepared, annealed, and modified samples.

3.2. Sol–Gel Synthesis

Sol–gel approach is a well-suited approach to the preparation of WO₃- and MoO₃-based films, porous oxides, and multicomponent materials because it enables precursor mixing at the molecular scale. Hydrolysis and condensation may be tuned through precursor, pH, solvent composition, complexing agents, aging time, and thermal treatment. These variables govern nucleation, network formation, porosity, crystallinity, and film integrity, whereas dopants or secondary phases can be introduced before gelation. The method is therefore particularly efficient for controlling composition and interfacial contact. In practice, the method is less straightforward than it appears. Incomplete condensation, residual organics, shrinkage, and cracking can undermine reproducibility, whereas prolonged aging and post-calcination increase energy consumption. For WO₃, sol–gel processing has been used to form uniform films and incorporate catalytic or electronically active components. In connection with this, Longato et al. [65] prepared WO₃ films containing 10 nm colloidal Pt nanoparticles (NPs) for gasochromic sensing. This approach illustrates the ability of solution processing to distribute a catalytic phase within an oxide film, which may improve gas activation and interfacial charge exchange. However, the separate synthesis of Pt NPs and annealing at 700 °C increase fabrication complexity and make it difficult to describe the process as either low-temperature or readily scalable. Wan et al. [66] also prepared sol–gel-derived WO₃ thin films for NO₂ sensing, supporting the compatibility of this method with resistive sensor fabrication. Shieh et al. [67] prepared WO₃ and W-Ti-O thin-film sensors by sol–gel dip-coating. Ti incorporation reduced the grain size and increased the space-charge-layer thickness, raising the reported sensitivity to 10 ppm NO₂ at 200 °C from 82 for WO₃

to 1768 for W-Ti-O. Sol-gel synthesis provides similar compositional flexibility for MoO_3 , but control of phase separation becomes increasingly important as material complexity increases. Rajhi et al. [68] used a one-pot route to prepare $\text{MoO}_3/\text{SiO}_2$ composites with tunable MoO_3 loading which formed orthorhombic MoO_3 nanoflakes dispersed within the silica matrix. Varying the oxide fraction allowed the dimensions of the nanoflakes to be modified, although excessive loading may reduce dispersion uniformity or promote segregation. Such matrix-supported structures can restrict particle coarsening and improve mechanical stability, but an electrically insulating SiO_2 phase may also hinder charge transport if its content or spatial distribution is not carefully controlled. Aghaei et al. [69] prepared a ternary $\text{MoO}_3\text{-Bi}_2\text{O}_3\text{-Bi}_2\text{MoO}_6$ composite by the sol-gel method. The coexistence of three crystalline phases may generate several interfaces and broaden the range of accessible redox or adsorption sites. At the same time, it complicates mechanistic interpretation because changes in performance may arise independently from phase composition, particle size, defect density, or interfacial band alignment. Matched binary and single-phase controls are therefore required before assigning an improvement to interfacial cooperation. Ates et al. [70] obtained orthorhombic MoO_3 from molybdenum isopropoxide and molybdenum trichloride, followed by calcination at 400 °C. Although phase-pure material was achieved, the three-week aging period represents a substantial kinetic limitation and reduces the practical value of the route for rapid production. Al-Namshah et al. [71] used the F127 triblock copolymer as a structure-directing agent to prepare mesoporous MoO_3 and subsequently incorporated 1–4 wt% multi-walled carbon nanotubes (MWCNTs). This strategy combines controlled porosity with a conductive additive, although the contribution of the nanotubes must be separated from changes in oxide crystallinity, pore structure, and interfacial contact.

3.3. Chemical Vapor Deposition

Chemical vapor deposition (CVD) is particularly useful for the fabrication of WO_3 - and MoO_3 -based films with controlled thickness, strong substrate adhesion, and direct electrical contact. Conventional CVD relies on volatile precursors, whereas aerosol-assisted chemical vapor deposition (AACVD) transports solution-derived precursor droplets into the heated reaction zone and therefore accommodates compounds with lower volatility. Film deposition can be regulated through substrate temperature, precursor concentration, carrier-gas composition, flow rate, deposition time, and oxygen partial pressure. These variables influence not only morphology and preferred orientation but also phase composition, oxygen stoichiometry, defect density, and electrical transport. The main limitations are the need for specialized equipment, elevated temperatures, precursor losses, reactor-wall deposition, and stringent control of gas-phase transport during scale-up. For WO_3 , AACVD deposits the oxide film directly onto the heated substrate from aerosolized precursor droplets, avoiding a separate powder-synthesis and transfer step while allowing substantial control over nanoscale architecture. Hajimazdarani et al. [72] deposited WO_3 films from H_2WO_4 -derived precursor solutions and found that precursor concentration influenced film coverage and morphology, as illustrated in Figure 3c. Triton X-100 improved aerosol stability and film uniformity, but annealing at 500 °C for 6 h was still required. The high thermal demand weakens the practical advantage of AACVD as a nominally rapid deposition route. Malik et al. [73] subsequently decorated AACVD-deposited WO_3 nanowires (NWs) with approximately 3 nm CeO_2 NPs. Changes in N_2 flow transformed the nanowire tips from pointed to flower-like structures, demonstrating the sensitivity of vapor-phase deposition to mass transport. However, separate NP preparation, drop casting, and post-annealing increased process complexity and may have produced less uniform interfaces than direct in situ heterostructure formation. Hot-filament CVD provides much

shorter deposition times but introduces a different energy trade-off. Godbole et al. [74] fabricated monoclinic WO_3 films with broccoli-like and pyramidal morphologies within 4–6 min and without post-calcination. Although rapid deposition is attractive, the filament temperature of approximately 1250 °C creates a substantial localized energy requirement, whereas the strong dependence of morphology on the reactive-gas ratio may hinder reproducibility over large substrates. Arzaee et al. [75] developed a one-step AACVD route using purified air rather than N_2 and obtained WO_3 films within 10 min at 450 °C. The films contained multiple exposed facets and appreciable oxygen-vacancy concentrations, and the overall processing time was reportedly reduced by approximately 13 fold relative to deposition followed by annealing. This is a significant step towards process intensification, but the simultaneous changes in exposed facets and defect density make their individual contributions difficult to separate. Because both are sensitive to the oxygen chemical potential, reproducible scale-up will require tighter control over local temperature and gas composition than is typically reported in morphology-focused laboratory studies. CVD has also been used to control the structure and oxygen stoichiometry of MoO_3 . Defect-modulated α - MoO_3 flakes were deposited at 850 °C and subsequently exposed to reductive treatment [76]. The α - MoO_3 framework was retained, but the combined deposition and reduction steps increased the thermal burden and may have generated spatially non-uniform oxygen-vacancy distributions. Chen et al. [77] demonstrated a higher-throughput hot-wire CVD route in which commercial MoSi_2 heating rods acted as both the Mo source and deposition substrate, yielding approximately 20 g of single-crystalline α - MoO_3 nanobelts per rod in ambient air. The high yield and lack of a protective atmosphere are attractive for scale-up. However, deposition relied on rod surfaces modified by prior carbothermal treatment, making substrate history a potential source of batch-to-batch variation that should be carefully controlled. Metal–organic CVD provides finer control over film crystallinity and orientation through the choice of precursor and deposition conditions. Guerrero et al. [78] used $\text{MoO}_2(\text{acac})_2$ to deposit crystalline α - MoO_3 films directly at approximately 400–560 °C without post-annealing. Highly oriented, needle-like films formed near 560 °C, showing that precursor decomposition and substrate temperature can direct film crystallization and orientation. However, the lengthy precursor synthesis, reduced-pressure operation, high deposition temperature, and specialized handling requirements may hinder scale-up and cost-effective production. The atmospheric-pressure carbonyl-CVD route was originally detailed by Gesheva, Ivanova, and co-workers in papers published during 1999–2001. These studies described the low-temperature deposition of MoO_3 and WO_3 films from $\text{Mo}(\text{CO})_6$ and $\text{W}(\text{CO})_6$, respectively, and examined the influence of process and annealing temperatures on crystallinity, vibrational signatures, and optical properties [55,79–81]. The as-deposited films were often amorphous or weakly crystalline, whereas annealing produced crystalline oxide phases. Amorphous films may contain a high density of coordinatively unsaturated and defect-related sites, but their poorly defined conduction pathways and tendency to crystallize during heating can cause baseline drift and limited long-term reproducibility. Polycrystalline films generally provide more stable transport and better-defined structure–property relationships, although annealing can reduce surface area or remove reactive defects. The preferred state therefore depends on whether adsorption-site density or electrical and thermal stability controls the sensing response. CVD is the most valuable approach when direct device integration is the main objective. The key challenge is to translate its control over film deposition into reproducible TEA sensing at practical power levels. This will require low-temperature, annealing-free deposition of heterostructures and large-area validation of film and device uniformity.

3.4. Magnetron Sputtering

Magnetron sputtering is a physical vapor deposition (PVD) method suitable for directly depositing dense, adherent WO_3 - and MoO_3 -based sensing films on electrodes, microheaters, and other device substrates. The Ar-plasma ejects species from a metal or oxide target, whereas reactive O_2 flow enables oxide formation and adjustment of oxygen stoichiometry. Target power, Ar/ O_2 ratio, working pressure, substrate temperature, target-to-substrate distance, and deposition time determine film thickness, grain size, crystallinity, preferred orientation, surface roughness, and oxygen-vacancy concentration. These parameters are critical for chemiresistive sensors because they affect baseline resistance, adsorption-site density, depletion-layer modulation, and long-term stability. Ngoc et al. [54] showed that structural ordering in sputtered WO_3 films altered the optical bandgap, whereas Zhu et al. [48] demonstrated that magnetron sputtering conditions strongly influenced the stability of WO_3 thin-film gas sensors. Singh et al. [32] also prepared sputter-deposited nanocrystalline MoO_3 films for NO_2 sensing. Gao et al. [82] fabricated ZrO_2 - CeO_2 / WO_3 films by magnetron sputtering and developed a humidity-tolerant TEA sensor. Magnetron sputtering provides precise thickness control, good reproducibility, wafer compatibility, and binder-free sensor integration. Its main limitations are the need for vacuum equipment, relatively low deposition rates, target cost, and possible plasma-induced defects or residual stress. Post-deposition annealing may also be required to obtain the desired phase, but it can simultaneously alter grain size and oxygen deficiency. Sputtering studies should therefore report both deposition and annealing conditions and correlate them with film stoichiometry, crystallinity, electrical resistance, and device-to-device variation.

3.5. Microwave Method

Microwave-assisted synthesis provides rapid and volumetric heating, which can accelerate nucleation and shorten the preparation of WO_3 - and MoO_3 -based nanomaterials. Unlike conventional heating from the reactor walls, microwaves directly heat absorbing components within the reaction mixture. This can shorten reaction times and improve control over particle size, phase, and morphology, although the outcome depends on the mixture, reactor design, pressure, and batch size. For WO_3 , microwave processing may be used to control phase formation and crystallite growth. Hernandez-Uresti et al. [83] employed an additive-free microwave-assisted hydrothermal route and showed that the microwave treatment time and subsequent calcination controlled the transformation between hexagonal and monoclinic WO_3 . Avoiding organic additives can reduce surface contamination and simplify purification, but thermal treatment at temperatures as high as 700°C was still needed to obtain the targeted phases. The method therefore shortened the solution-phase reaction without eliminating the major thermal requirement. Yu et al. [84] prepared nanoscale $\text{WO}_3 \cdot 1/3\text{H}_2\text{O}$ within 20 min and observed improved crystallinity at higher microwave temperatures. This study demonstrates the kinetic benefit of microwave heating, although the inclusion of PdCl_2 in the precursor system introduces compositional complexity that should be considered when relating the final structure solely to the WO_3 hydrate. Microwave irradiation can also support the rapid construction of multicomponent materials. Claudino et al. [85] prepared WO_3 and W-doped BiVO_4 / WO_3 heterostructures using microwave-assisted solvothermal steps lasting approximately 12–24 min. The short reaction time is attractive, but the resulting material contains several potentially active features, including W substitution, WO_3 coupling, interfacial charge transfer, and defect formation. Appropriate single-component and binary controls are therefore needed to establish which modification governs the functional response. Periasamy et al. [86] combined ultrasonic pretreatment with microwave irradiation to produce highly crystalline orthorhombic WO_3 with nanorod and nanosheet derived morphologies, as illustrated in

Figure 3d. Ultrasonication influenced crystallite development by improving precursor dispersion before microwave treatment. Nevertheless, precipitation, ultrasonic processing, microwave irradiation, drying, and 4 h of calcination make the overall preparation more complex than a single-step microwave route. For MoO_3 , microwave processing provides similar advantages in rapid oxide formation and dimensional control. Mir et al. [87] synthesized MoO_3 NPs at 120 °C within 15 min and subsequently functionalized their surfaces with cetyltrimethylammonium bromide, sodium dodecyl sulfate, or thioglycolic acid. Surface functionalization can regulate dispersion and molecular adsorption, but calcination at 400 °C, followed by 12 h of surfactant treatment and 24 h of aging, substantially increases the actual processing time. Residual surfactants and differences in binding strength may also alter the surface chemistry, making it difficult to compare the intrinsic behavior of the resulting MoO_3 samples. Lin et al. [88] used a two-step microwave-assisted hydrothermal process to prepare MoO_3 nanobelts and reduced graphene oxide (GO)- MoO_3 composites. During the second microwave step, GO was reduced and coupled with MoO_3 , enabling rapid composite formation and improved interfacial contact. Nevertheless, changes in conductivity, oxide dispersion, surface area, and defect density occur simultaneously, so the contribution of the interface should not be inferred without matched MoO_3 , graphene, and physically mixed controls. Hsu et al. [89] followed a different route in which hydrothermally prepared WO_3 NPs and MoO_3 nanoribbons were subjected to prolonged wet mechanical grinding to form WO_3/MoO_x heterostructures and introduce oxygen vacancies into MoO_3 . In another study, rapid formation of $\alpha\text{-MoO}_3$ nanobelts was also demonstrated. In this regard, Zakharova et al. [90] prepared $\alpha\text{-MoO}_3$ nanobelts from a peroxomolybdic acid precursor at 160 °C within 20 min. The products were smaller than those obtained by conventional autoclave treatment, indicating that rapid microwave-driven nucleation restricted subsequent crystal growth. The use of concentrated H_2O_2 and metallic Mo as the precursor, however, introduces additional safety and handling requirements. Wang et al. [91] obtained highly crystalline orthorhombic $\alpha\text{-MoO}_3$ nanobelts after 10 min of microwave heating at 150 °C. Despite the short irradiation period, overnight precursor aging remained necessary and should be included when comparing its processing efficiency with other microwave-based routes. Microwave synthesis enables rapid nucleation and limits crystal growth, favoring small crystallites, exposed surfaces, and heterostructures. However, current studies mainly demonstrate synthetic flexibility rather than improved TEA detection. Aging, calcination, surfactant treatment, and multistep composite formation may also offset the time saved by microwave heating.

3.6. Electrodeposition Method

Electrodeposition enables the direct deposition of WO_3 - and MoO_3 -based films on conductive substrates under mild conditions and with simpler equipment than most vapor-phase methods. Film loading, morphology, porosity, and thickness can be tuned through the deposition conditions and electrolyte chemistry. However, uneven current distribution can produce thickness variations, nonconductive substrates require modification, and post-deposition annealing is often needed to obtain crystalline oxides. For WO_3 , pulse-controlled deposition has been used to regulate porosity and film architecture. Pham et al. [92] employed a triple-pulse sequence to prepare porous WO_3 films on indium tin oxide (ITO) and compared them with compact films produced under continuous cathodic polarization. Alternating cathodic and anodic pulses, separated by a relaxation period, modified interfacial nucleation and mass transport, showing that waveform design can be more influential than deposited charge alone. The resulting porosity is potentially favorable for gas access, although the use of 2000 cycles considerably extends the process and increases the need for stable waveform control and batch-to-batch reproducibility. Morankar et al. [93]

electrodeposited nanogranular WO_3 films from a peroxotungstic acid precursor, as shown in Figure 3e, and found that the number of potential cycles controlled film loading and morphology. An intermediate condition of 20 cycles formed the most uniform structure, indicating that excessive deposition does not necessarily improve film quality. Patil et al. [94] similarly adjusted potentiostatic deposition time to regulate WO_3 deposition on FTO. The relatively mild post-treatment at 100°C was advantageous compared with conventional high-temperature crystallization. However, deposition times of up to 60 min, strongly acidic electrolytes, and hydrofluoric-acid-based substrate pretreatment reduce procedural simplicity and introduce safety concerns. Demir [95] illustrate an important distinction between mild deposition conditions and the overall energy demand of the fabrication process. WO_3 was deposited on ITO at a fixed potential, but the initially poorly crystalline film required annealing at 450°C for 2 h. This illustrates a recurring limitation of aqueous electrodeposition: low-temperature film deposition does not guarantee low-temperature processing when crystallization is deferred to a subsequent thermal step. For chemiresistive sensing, this distinction is important because annealing can simultaneously alter crystallinity, oxygen deficiency, grain size, adhesion, and baseline conductivity, making it difficult to assign the final response solely to the deposition conditions. Electrodeposition also allows compositional modification of MoO_3 -based films. Gao et al. [96] incorporated 0–5 at% W into MoO_3 layers using pulsed-current deposition, as illustrated in Figure 3f. Intermittent current can reduce concentration polarization and support more uniform incorporation of the secondary metal. Nevertheless, the precursor required 10 h of conditioning, and the as-deposited films remained amorphous. Compositional control was therefore achieved without simultaneous control of crystallinity. Changes attributed to W substitution should also be distinguished from variations in defect concentration, local coordination, and deposition kinetics. Adewinbi et al. [97] deposited nanocrystalline $\alpha\text{-MoO}_3$ films within 5–10 min using a simple two-electrode cell. However, the lack of independent working-electrode potential control makes the process sensitive to solution resistance, counter-electrode polarization, and changes in film impedance, potentially limiting reproducibility. A three-electrode cell offers better control but adds complexity and may hinder scale-up. Ijeh et al. [98] prepared pristine and Cu-modified MoO_3 films by potentiostatic deposition, demonstrating that low concentrations of a secondary metal can be introduced directly from the electrolyte. Subsequent thermal treatment was still needed to obtain polycrystalline orthorhombic $\alpha\text{-MoO}_3$, confirming that dopant incorporation and phase formation do not necessarily occur in the same step. Careful structural analysis is required when metal salts are introduced into strongly alkaline baths because Cu-rich secondary phases, surface segregation, or non-uniform incorporation may otherwise be misinterpreted as homogeneous substitution. More complex architectures have also been produced electrochemically. In this regard, Budak et al. [99] constructed binder-free $\text{Mo}_x\text{W}_{1-x}\text{O}_3@\text{CoPc-rGO}$ electrodes on Ni foam (NF) through sequential cyclic electrodeposition and electro-polymerization. This strategy combined tunable Mo/W composition, direct deposition on a 3D current collector, cobalt–phthalocyanine polymerization, and electrochemical reduction of GO. The binder-free architecture provides intimate contact, but the multistep deposition, wide potential windows, repeated annealing, and several variable components obscure the sensing mechanism. Matched controls are needed to isolate the effects of the Mo/W ratio, rGO conductivity, cobalt–phthalocyanine activity, NF structure, and interfacial coupling instead of broadly invoking synergy. Electrodeposition remains attractive for low-cost film deposition and integration with miniaturized TEA sensors. However, most cited studies concern electrochromic or energy-storage devices and demonstrate fabrication feasibility rather than TEA-sensing performance. Future studies should report deposition efficiency, film thickness and loading, adhesion, crystallinity,

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times of 3 and 5 s, respectively, and a detection limit of 200 ppb. The material contained interesting pores, small crystallites, numerous grain boundaries, reduced exposure of the (002) facet, and a comparatively high work function of 4.17 eV. The strength of this study lies in linking TEA response to measurable structural and electronic properties rather than surface area alone. A higher work function can deepen electron depletion in air, while small crystallites and abundant grain boundaries increase the fraction of material affected by surface reactions. However, because facet exposure, porosity, and work function changed together, their individual roles remain unresolved. Operation at 325 °C also limits practical use in low-power, continuous freshness-monitoring devices. Chen et al. [101] prepared bitter-melon-like γ -WO₃ architectures assembled from NRs and nanosheets. The optimized WO₃-RS-5 sensor, calcined at 500 °C, showed a response of 281 to 100 ppm TEA at 217 °C with a 5 s response time; selectivity, reversibility, humidity tolerance, and seafood-freshness monitoring were also evaluated. The enhanced gas-sensing response was attributed to mesoporosity, oxygen-deficient sites, adsorbed oxygen species, and Lewis-acidic W⁶⁺ centers. The morphology and proposed TEA sensing mechanism of the bio-templated WO₃ sensor reported by Liu et al. [102] are shown in Figure 4a–f and Figure 4g, respectively.

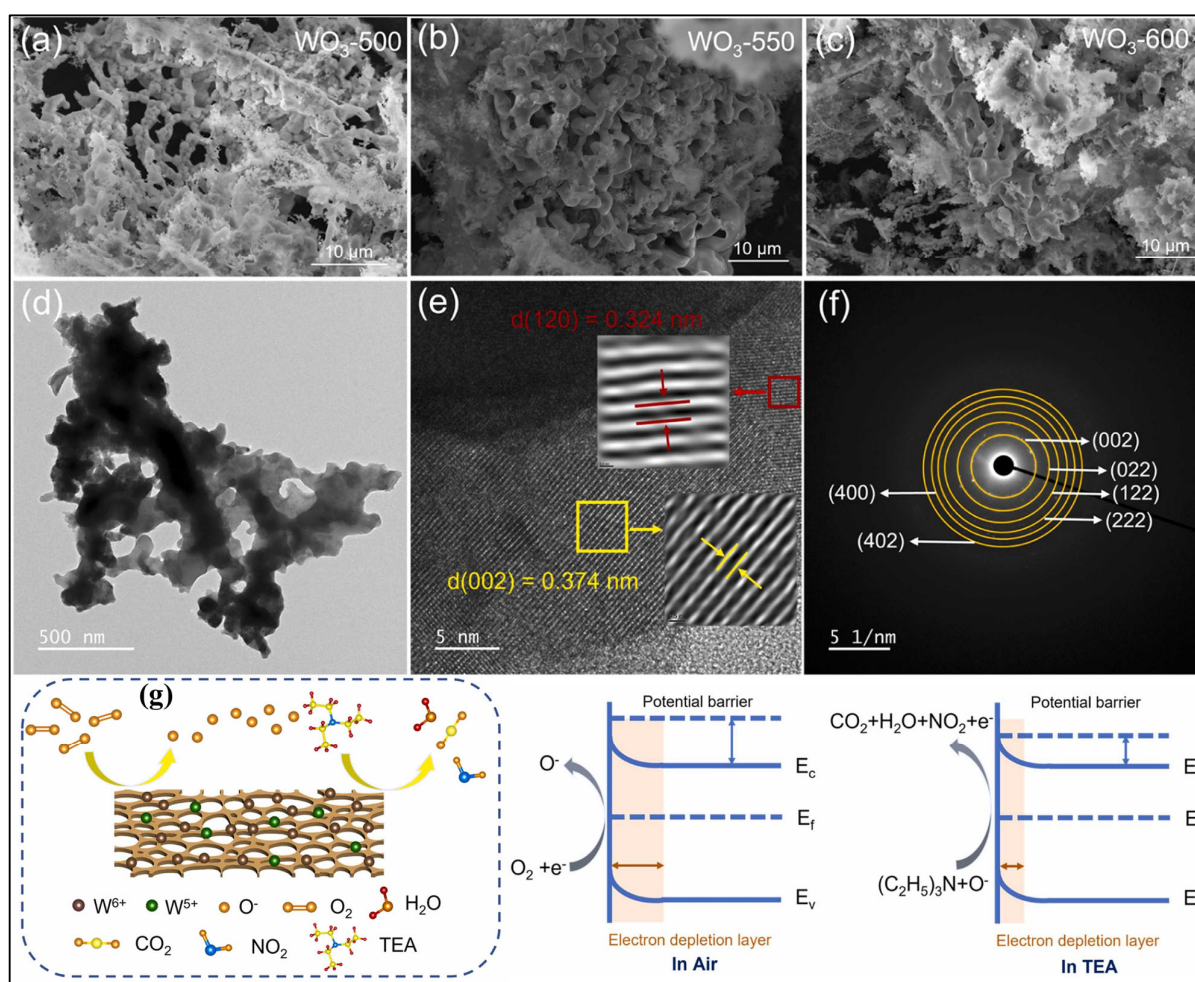
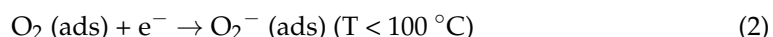
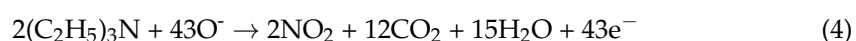


Figure 4. Morphological characterization of bio-templated WO₃ prepared at different calcination temperatures: SEM images of (a) WO₃-500, (b) WO₃-550, and (c) WO₃-600; (d) TEM, (e) HRTEM, and (f) SAED images of WO₃-550, highlighting its porous architecture, resolved lattice fringes, and polycrystalline structure. (g) Mechanism and energy band diagram of WO₃-based TEA sensor. Reproduced with permission [102].

For compositionally pristine WO₃, the sensing pathway can be summarized as follows:



At typical TEA-sensing temperatures, O[−] is widely regarded as a key reactive oxygen species. TEA adsorbs through its electron-donating nitrogen atom at Lewis-acidic W sites. Subsequent oxidation by surface O[−] releases electrons back into WO₃, lowering the sensor resistance:



The equation describes the electron-transfer process which changes resistance, not a fixed product distribution. Complete oxidation of TEA to NO₂, CO₂, and H₂O should not be assumed without identifying and quantifying all products under the actual sensing conditions, as partially oxidized amines or carbonyl intermediates may also form. Humidity is a major challenge for pristine WO₃ because water competes for adsorption sites, alters surface hydroxylation, and affects both baseline resistance and reaction kinetics. Although WO₃-RS-5 showed humidity tolerance, its practical suitability requires quantitative testing across a broad, dynamic humidity range. Long-term measurements should also distinguish repeatability from baseline drift, irreversible hydroxylation, grain growth, and changes in oxygen-vacancy concentration. The *Astragalus membranaceus*-templated WO₃ reported by Liu et al. [102] is better classified as a template-derived material than as conventionally prepared pristine WO₃. Although calcination removes the organic template and may leave chemically undoped WO₃, the fibrous, porous architecture originates from the biological scaffold. This distinction separates compositional purity from synthetic simplicity. Samples calcined at 500–600 °C retained the fibrous structure, whereas WO₃-550 showed interconnected meso- and micropores, a surface area of 20.6 m².g^{−1}, polycrystallinity, and exposed (002) and (120) planes. The sensor responded by 65.56 to 50 ppm TEA at 220 °C, with a detection limit of 0.399 ppm, good repeatability, and stability of 1 month. The response was attributed to accessible porosity, gas diffusion, surface oxygen species, and W⁵⁺-related defects. Because calcination changes template removal, crystallinity, porosity, grain size, surface area, and oxygen stoichiometry together, the specific role of oxygen vacancies remains uncertain. Morphologically and thermally equivalent controls are needed to isolate vacancy effects. Scale-up will also depend on consistent template composition, minimal inorganic residues and combustion shrinkage, reproducible batches, and stable performance under humidity.

4.2. Doped WO₃-Based Materials

It is understood that dopants can modify the carrier density, defect chemistry, surface acidity, phase composition, and oxygen adsorption of WO₃. Their effects depend on both identity and concentration and cannot be explained simply by assuming that more oxygen vacancies are formed. Materials containing segregated oxides or noble metals must also be distinguished from genuinely substitutional systems because secondary phases may introduce catalytic activity, Schottky barriers, or p-n junctions. Zhang et al. [103] examined 3, 5, and 7 wt% indium (In)-doped WO₃. In addition, they changed the irregular WO₃ particles into more uniform cubic nanoblocks, with the 5 wt% sample exhibiting the best response. This sensor showed a response of 11.2 to 50 ppm TEA, response and recovery times of 11 and 40 s, and favorable selectivity. The O 1s component assigned to defects and

chemisorbed oxygen increased from 18.49% to 41.83%, whereas the surface area changed only slightly, from 14.90 to 15.20 m².g⁻¹. This result points to changes in surface oxygen chemistry and electronic structure rather than increased surface area. However, O 1s fitting does not directly measure oxygen-vacancy concentration or charge state, and the simultaneous change in morphology prevents firm mechanistic attribution. Cu modification introduced a more complex sensing mechanism. Zhang et al. [104] prepared electrospun Cu/WO₃ NWs, and the optimized 5% Cu/WO₃-based sensor showed a response of 108 to 100 ppm TEA at 180 °C, with a detection limit of 18.14 ppb. Because Cu₂O and CuO were present as segregated phases, the response likely arose from catalytic activity, altered oxygen chemisorption, and p-n junctions rather than substitutional doping alone. The high response was accompanied by a recovery time exceeding 760 s and a significant loss of response under humid conditions which highlighted the trade-off between strong TEA adsorption and efficient sensor regeneration. Ga modification provides a better balance between operating temperature and low-concentration detection. Wen et al. [105] reported that WGa5, containing 5 wt% Ga, produced a response of 6.7 to 1 ppm TEA at 150 °C and remained responsive at 100 ppb. The improvement was linked to changes in the balance between hexagonal and monoclinic WO₃ and in preferential facet exposure. This explanation extends beyond a vacancy-only mechanism, although the separate effects of phase boundaries and exposed facets were not resolved. WGa5 was also tested using volatiles released from stored yellow croaker, Chi fish, and Youkou fish. As shown in Figure 5a–e, the response increased approximately linearly with storage time under the tested conditions, while the estimated gas concentration rose from about 0.93 to 5.23 ppm over 6 days (Figure 5f). These results suggest potential for spoilage monitoring but do not establish TEA-specific detection because fish headspace contains multiple amines and other volatile compounds. Chromatographic validation, controlled humidity and temperature, and interference tests with trimethylamine, ammonia, ethanol, aldehydes, and sulfur-containing compounds are needed before the signal can be used as a general freshness index.

Zhang et al. [106] prepared electrospun Ni/WO₃ nanofibers, with the optimized 3% Ni sample showing a response of 487 to 100 ppm TEA at 200 °C. The response may be associated with increased chemisorbed oxygen, defect formation, and Ni-assisted surface reactions. Ni²⁺/Ni³⁺ sites may promote electron trapping and redox activity, but carrier-density, work-function, conductivity, and operando valence measurements are needed to separate electronic effects from surface catalysis. A more complex design was reported by Zhou et al. [107], in which authors decorated Fe-modified WO₃ hollow microspheres with PtPd NPs. PtPd@Fe-WO₃ exhibited a response of 409.58 to 20 ppm TEA at 260 °C, with a 1 s response time and a reported detection limit of 69.59 ppb. Density functional theory calculations suggested that Fe altered defect chemistry and adsorption, whereas PtPd likely promoted TEA activation and oxygen dissociation through catalytic and electronic sensitization. Although the response was rapid, the 108 s recovery time, high operating temperature, dual noble-metal content, and multistep fabrication limit its appeal for low-cost devices. Rare-earth ions may act through lattice strain, Lewis acidity, and carrier trapping. In Pr³⁺-doped WO₃ NRs [108], the 1% Pr³⁺ sample showed a faster response, higher sensitivity, and lower detection limit than pristine WO₃. These improvements were attributed to electronic changes and increased chemisorbed oxygen. The role of Pr³⁺ is therefore better interpreted in terms of adsorption-site acidity and electron trapping rather than vacancy formation alone. Fish-freshness tests were encouraging, although humidity dependence, mixed-gas interference, and long-term calibration were not addressed in depth. Pd-modified WO₃ presents a similar mechanistic complexity. Zhang et al. [109] identified both lattice-incorporated Pd²⁺ and surface PdO_x in WO₃ NWs. The optimized 2.5 at%

Pd-based sensor showed a response of 1100 to 20 ppm TEA at 160 °C and a detection limit of 5 ppb. The response cannot be assigned to lattice doping alone because PdO_x can also activate TEA and oxygen and alter interfacial charge transfer. Wu et al. [110] combined Cr-modified WO_3 microspheres with machine-learning-based signal analysis. The 1 wt% Cr- WO_3 -based sensor delivered a response of 330.1 to 50 ppm TEA at 240 °C, with response and recovery times of 15 and 44 s and a detection limit of 42 ppb. Cr addition changed the bandgap, porosity, and defect-related oxygen chemistry at the same time, preventing their individual effects from being isolated. Machine learning may improve discrimination among overlapping signals, but it cannot correct humidity sensitivity, drift, or poor reproducibility. Reliable use therefore requires independently validated datasets collected from multiple sensor batches under changing environmental conditions. In, Ga, Ni, Cr, and Pr may alter the WO_3 lattice, surface acidity, carrier trapping, phase balance, and oxygen adsorption. Cu oxides form p-n junctions, whereas PtPd and PdO_x provide catalytic and interfacial sensitization. Ga- and Pd-modified WO_3 operated at 150–160 °C, whereas PtPd@Fe- WO_3 and Cr- WO_3 required 240–260 °C. Future work may consider dopant location and oxidation state and distinguish lattice substitution from surface clusters or secondary phases. Morphologically equivalent controls, work-function and carrier measurements, operando spectroscopy, and O_2 temperature-programmed desorption can identify the dominant mechanism. Practical validation should examine humidity, recovery, baseline drift, batch variability, and long-term calibration under realistic seafood-storage or industrial conditions.

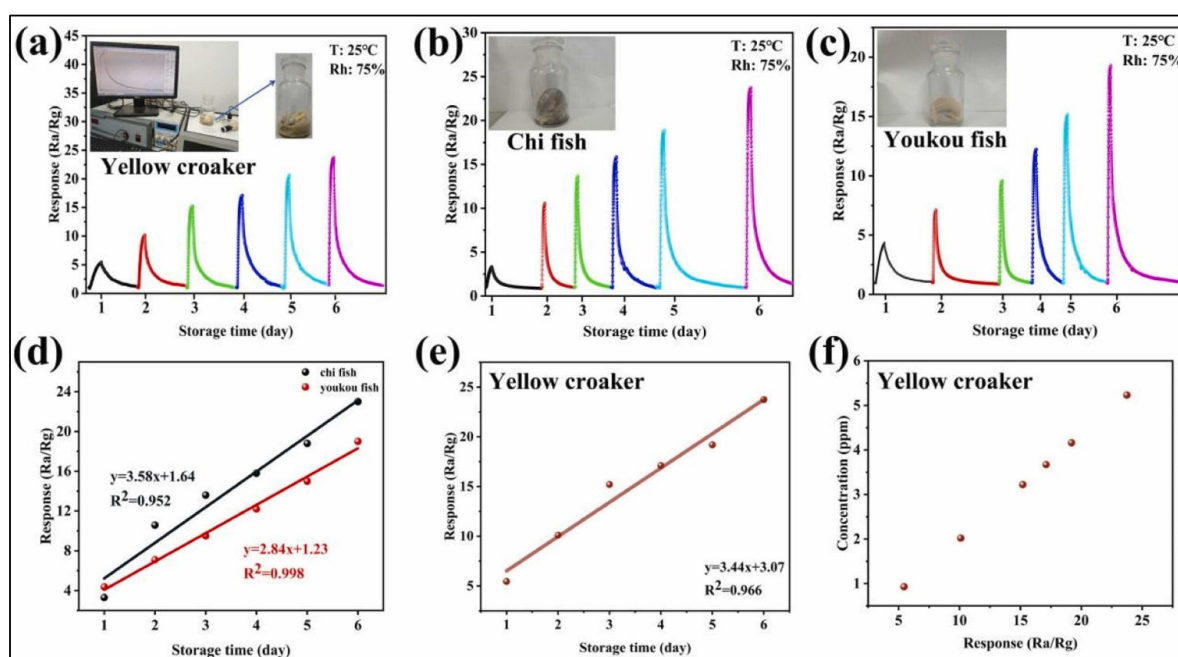


Figure 5. Practical evaluation of the WGa5 sensor for monitoring fish freshness during storage: dynamic sensing responses to volatiles released from (a) yellow croaker, (b) Chi fish, and (c) Youkou fish over six days; (d,e) linear relationships between sensor response and storage duration; and (f) correlation between sensor response and the estimated concentration of released gas. Reproduced with permission [105].

4.3. WO_3 /Metal-Oxide-Based Materials

Integration of WO_3 with another oxide may alter TEA sensing through band alignment, interfacial depletion, catalysis, and morphology. Fermi-level equilibration in n-n junctions changes the interfacial resistance barrier, whereas p-n junctions may create a wider depletion region and stronger resistance modulation. Because junction formation

also changes composition and structure, component controls with comparable morphology, porosity, particle size, and defect density are needed to isolate the interfacial contribution. Yan et al. [111] developed an n-n α - MoO_3 @ WO_3 heterojunction for TEA detection. The optimized MW2-based sensor showed a response of 80.4 to 100 ppm TEA at 200 °C, response and recovery times of 9 and 26 s, a detection limit of 127 ppb, and stable operation over 50 days. Interfacial electron redistribution was proposed to amplify the resistance change, although direct work-function or band-alignment measurements would strengthen this interpretation. The response declined sharply above 83% relative humidity, indicating that the interface did not prevent water interference. Gao et al. [82] deposited ZrO_2 - CeO_2 / WO_3 films by radio-frequency magnetron sputtering. The optimized film responded by 18.56 to 20 ppm TEA, with 1 and 5 s response and recovery times and a working range of 0.5–500 ppm under humid conditions. Its humidity tolerance was ascribed to $\text{Ce}^{3+}/\text{Ce}^{4+}$ redox cycling, which may preserve reactive oxygen species and suppress excessive hydroxylation. Sputtering provides good thickness control and direct device integration, but its equipment and compositional-control requirements increase fabrication complexity. Tian et al. [112] showed that layer sequence can matter as much as composition by comparing NiO-on- WO_3 and WO_3 -on-NiO bilayers. NiO-on- WO_3 performed better, exhibiting a response of 3.62 to 1 ppm TEA at 300 °C, with response and recovery times of 11 and 38 s. The different behavior of the two p-n architectures reflects changes in interfacial depletion and in the oxide exposed directly to TEA. Headspace gas chromatography–mass spectrometry identified acetaldehyde and diethylamine, providing valuable evidence of partial TEA conversion. However, the 300 °C operating temperature limits low-power use. Zhang et al. [113] prepared electrospun WO_3 / SnO_2 nanotubes and nanofibers as a simpler n-n system. The optimized sensor showed a response of 26.8 to 100 ppm TEA at 260 °C, with response and recovery times of 9 and 22 s. Hollow structures improved gas access, whereas oxygen-related defects and the WO_3 / SnO_2 interface may have increased resistance modulation. Its practical appeal lies in the relatively inexpensive composition, although the roles of morphology and interfacial charge transfer were not separated. Yuan et al. [114] fabricated WO_3 sheets on α - Fe_2O_3 to form hollow, lychee-like heterostructures. The W/Fe-0.5 sensor demonstrated a response of 32.41 to 50 ppm TEA at 220 °C, with response and recovery times of 8 and 29 s and a detection limit of 43 ppb. Its lower operating temperature than either parent oxide suggests that the interface altered the energetics of adsorption and surface reaction. The response was ascribed to interfacial band bending, oxygen-related defects, and the hollow architecture, but these effects changed together and could not be quantified separately. Figure 6 summarizes the proposed mechanism. In air, adsorbed oxygen withdraws electrons and forms a depletion layer, whereas Fermi-level equilibration between WO_3 and α - Fe_2O_3 causes interfacial band bending. TEA oxidation returns electrons to the oxides and narrows the depletion region, lowering the resistance. The response therefore reflects both interfacial charge modulation and gas-accessible architecture rather than an undefined “synergistic effect.”

Amorphous–crystalline interfaces can provide reactive sites while retaining a stable conduction pathway. Fan et al. [115] deposited amorphous RuO_x clusters on crystalline WO_3 nanosheets. The optimized A/C-2-based sensor showed a response of about 36 to 10 ppm TEA at 140 °C, a 1 s response time, and a detection limit of 0.5 ppm. Unsaturated RuO_x sites and defect-rich interfaces were proposed to activate TEA and oxygen, whereas crystalline WO_3 supported charge transport and structural stability. Localizing the amorphous phase may therefore avoid the baseline instability often associated with fully amorphous sensing layers. Zhang et al. [116] prepared cauliflower-like In_2O_3 / WO_3 heterostructures. The WI-3-based sensor displayed a response of 16.44 to 10 ppm TEA at 250 °C and showed good linearity over 0.05–100 ppm. XPS and density functional theory

calculations suggested changes in defect-related oxygen species and interfacial charge transfer. As both oxides are n-type, their contact can alter band bending and electron transport. However, the required temperature of 250 °C limits the benefit for low-power sensing. The largest numerical response in this group was reported by Zhu et al. [117] for a δ -MnO₂/WO₃ p-n heterojunction. The WMn1-based sensor achieved a response of 515.37 to 20 ppm TEA at 140 °C and a detection limit of 15.91 ppb. A wide interfacial depletion region and layered morphology were proposed to amplify resistance changes and improve gas access. These gains came with a 196 s recovery time, approximately 33.8% response variation over 30 days, and significant signal loss above 60% relative humidity. Each modification addresses a different limitation. ZrO₂-CeO₂/WO₃ emphasized rapid kinetics and humid operation, δ -MnO₂/WO₃ delivered a high response but poor recovery and stability, and RuO_x/WO₃ combined 140 °C operation with a 1 s response. NiO/WO₃ and In₂O₃/WO₃ still required 250–300 °C. For real-world use, TEA sensors must operate at low temperatures, recover quickly, tolerate humidity, and be fabricated reproducibly.

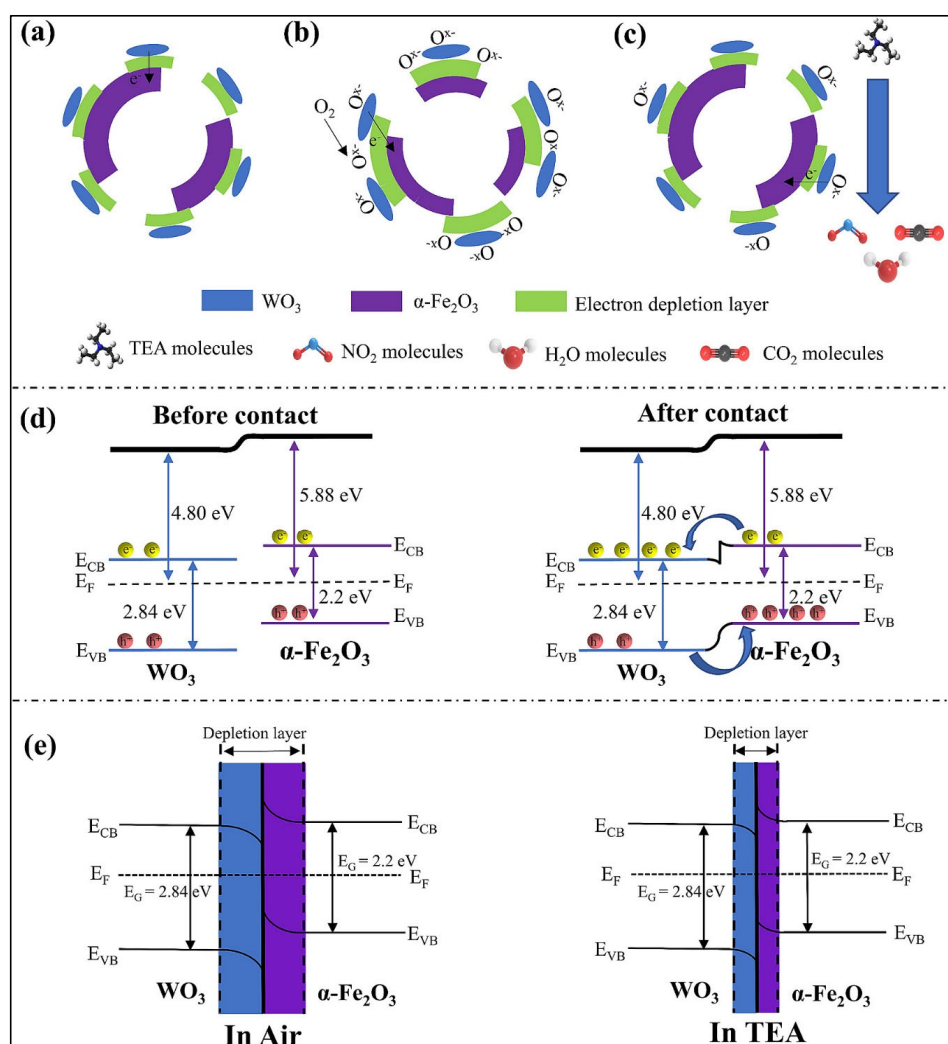


Figure 6. Proposed TEA sensing mechanism of the WO₃/α-Fe₂O₃ hollow lychee-like heterojunction: (a–c) surface oxygen adsorption, electron depletion, and TEA-induced redox reaction; (d) energy-band alignment of WO₃ and α-Fe₂O₃ before and after interfacial contact; and (e) depletion-layer modulation of the heterostructure in air and under TEA exposure. Reproduced with permission [114].

4.4. WO₃/Vanadate- and Tungstate-Based Materials

The modification of WO₃ with tungstates and vanadates was also found to be a promising approach to enhance the performance of the gas sensors. Because heterojunction

formation can simultaneously alter morphology, porosity, and defect density, appropriately matched controls are essential to attribute performance changes specifically to the interface. Multivalent V centers may promote surface redox reactions, whereas tungstates provide W-O frameworks with different band positions and adsorption properties. Wu et al. [118] prepared $\text{ZnWO}_4/\text{WO}_3$ nanorod heterojunctions by a two-step hydrothermal method. The optimized 0.1- $\text{ZnWO}_4/\text{WO}_3$ -based sensor showed a response of 103.1 to 50 ppm TEA at 210 °C, a 10 s response time, and a detection limit of 0.5 ppm. Compared to pristine WO_3 , it responded more strongly and rapidly and operated at a temperature 30 °C lower. These improvements were attributed to interfacial charge redistribution and ZnWO_4 assisted TEA oxidation. However, simultaneous changes in composition and NRs morphology make it difficult to determine their respective contributions to sensing performance. For the vanadate/ WO_3 composite shown in Figure 7, humidity remained a major limitation. The response to 40 ppm TEA and the baseline resistance both decreased as relative humidity increased from 10% to 95% (Figure 7a,b), consistent with competitive water adsorption, surface hydroxylation, and humidity-dependent charge transport. Humidity compensation is therefore still required. The sensor response remained stable over nine weeks (Figure 7c), with good selectivity across the tested gases (Figure 7d). However, baseline drift, calibration stability, repeated humidity cycling, and interference from trimethylamine, diethylamine, and ammonia require further in-depth investigation. The radar plot indicates that 5BVO/WO exhibits the most favorable overall performance (Figure 7e).

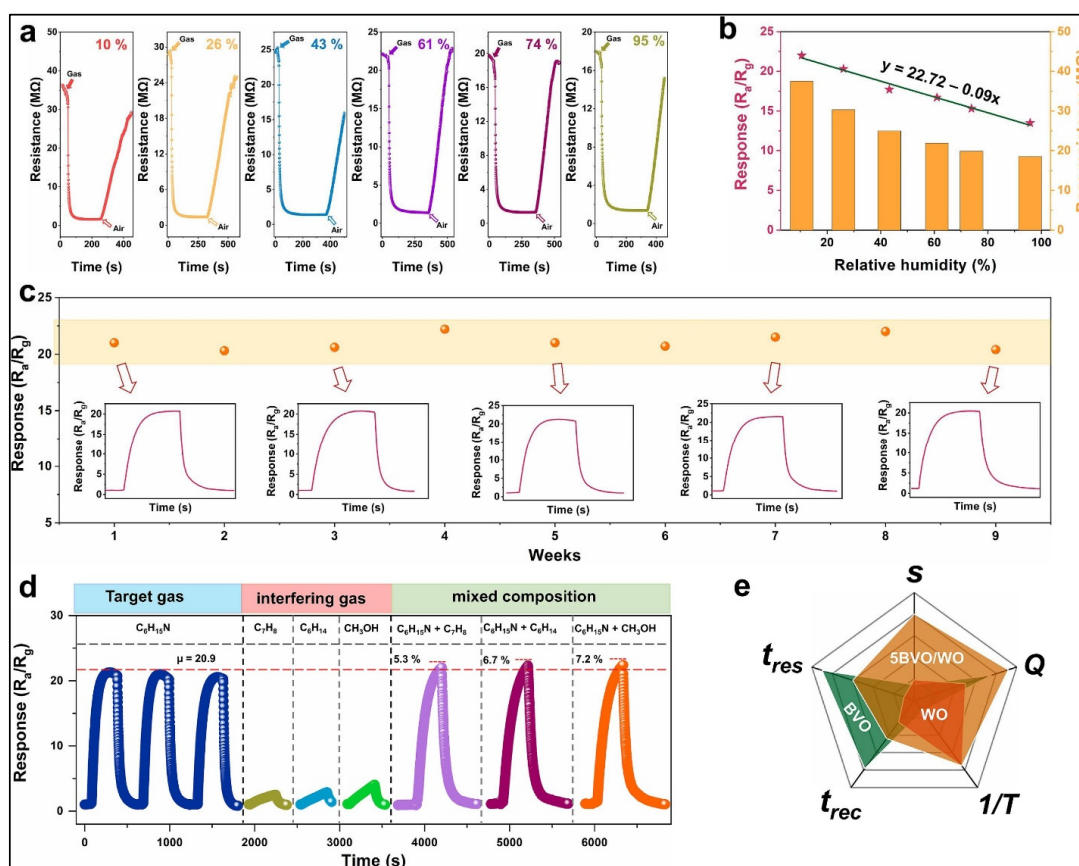


Figure 7. Practical sensing performance of the 5BVO/WO heterostructure toward TEA: (a) dynamic resistance profiles under different relative humidities; (b) dependence of baseline resistance and sensing response on humidity; (c) long-term response stability over nine weeks; (d) resistance to interference from individual and mixed gases; and (e) comparative radar analysis of key sensing parameters for WO, BVO, and 5BVO/WO sensors. Reproduced with permission [119].

In another study, Ma et al. [120] prepared hydrothermal $\text{WO}_3/\text{SnWO}_4$ NRs. The sensor showed a response of 121.39 to 200 ppm TEA, about 5.4 times that of pristine SnWO_4 , and retained a response of 6.6 at 5 ppm. Its 50 s response and 7 s recovery suggest relatively slow analyte activation but efficient surface regeneration. A morphology- and loading-matched WO_3 control is still needed to separate the effects of the WO_3 phase, nanorod structure, and heterointerface. Zhao et al. [121] combined electrospun WO_3 nanofibers with ErVO_4 . The composite produced a response of 15.79 to 100 ppm TEA and recovered faster than pure ErVO_4 , suggesting that its main benefit may be improved regeneration rather than strong response amplification. Measurements of band alignment, work function, and carrier density are needed to determine whether this behavior arises from surface acidity, electron trapping, or interfacial charge transfer. Shi et al. [122] introduced rGO into a $\text{WO}_3/\text{BiVO}_4$ heterojunction, obtaining a response of 15.4 to 5 ppm TEA. The response was attributed to type-II band alignment and rGO-assisted transport and adsorption. However, rGO can also change conductivity, aggregation, adsorption capacity, and junction resistance. Controls containing $\text{WO}_3/\text{BiVO}_4$, rGO/ WO_3 , rGO/ BiVO_4 , and physical mixtures are therefore required to identify the dominant contribution. Ding et al. [119] combined hierarchical $\text{BiVO}_4/\text{WO}_3$ with machine-learning-based signal analysis. The optimized sensor showed a response of 21 at 190 °C with a detection limit of 57 ppb. It could be summarized that above mentioned gas sensors address some limitations. In particular, $\text{ZnWO}_4/\text{WO}_3$ lowered the operating temperature, $\text{WO}_3/\text{SnWO}_4$ improved response at a higher TEA concentration, $\text{ErVO}_4/\text{WO}_3$ favored recovery, and BiVO_4 -based composites extended low-concentration detection. However, future studies may further measure band positions, work functions, and interfacial area and use morphology-matched component controls. In addition, repeated dry–humid cycling should assess both response retention and baseline drift, whereas long-term stability tests should probe interfacial diffusion, elemental segregation, grain growth, and calibration drift. For seafood monitoring, reliable discrimination of TEA from related amines under variable humidity is more important than achieving a high response under dry, single-gas conditions.

4.5. WO_3 /Polymers, Ag, Pd, Pt NPs and h-BN-Based Materials

Polymers, noble metals, photosensitizers, and 2D materials may modify WO_3 through different pathways. Conducting polymers combine amine affinity with interfacial charge modulation, whereas Ag, Pd, and Pt provide catalytic and electronic sensitization. Dyes generate photoexcited carriers, whereas h-BN can alter adsorption and interfacial transport without noble metals. Zhu et al. [123] prepared PEDOT@ WO_3 core–shell structures with a response of 1.58 to 20 ppm TEA and reported good selectivity, stability, and humidity tolerance. The response may involve both amine–polymer interactions and p–n interfacial modulation between p-type PEDOT and n-type WO_3 . Humidity-dependent ionic transport may also contribute. Because these effects were not independently resolved, the influence of PEDOT thickness, thermal stability, and baseline drift requires systematic investigation. A thick PEDOT coating may inhibit gas diffusion and dominate the sensor’s electrical response. Bi et al. [124] decorated hierarchical WO_3 nanoflowers with Ag. The optimized Ag/ WO_3 -2-based gas sensor showed a response of 837 to 100 ppm TEA at 250 °C, responded within 1 s, and remained sensitive at 100 ppb. Ag may promote oxygen activation and TEA oxidation through spillover while altering the WO_3 depletion layer. The nanoflower structure also improves gas access, so the effects of Ag loading, morphology, and interfacial charge transfer require separate evaluation. Moreover, operation at 250 °C may limit its value for low-power monitoring. Zhang et al. [125] reported an optimized 2 wt% Pd/ WO_3 nanosheet sensor with a response of 69.08 to 100 ppm TEA at 255 °C and response and recovery times of 4 and 36 s, respectively, with a detection limit of 37 ppb.

Pd can catalyze oxygen and TEA activation, whereas PdO/WO₃ interfaces can modify the resistance barrier. These chemical and electronic contributions are distinct and require operando identification of the Pd oxidation state. Figure 8 summarizes the TEA-sensing performance of Pd-modified WO₃ at 255 °C. The 2 wt% Pd/WO₃-based sensor showed a linear response from 1 to 100 ppm ($R^2 = 0.998$; Figure 8a), good repeatability over five cycles (Figure 8b), and reasonable stability over 25 days (Figure 8c). At 100 ppm, the response and recovery times were approximately 4 and 36 s, respectively (Figure 8d). However, humidity reduced the response (Figure 8e). Although the sensor was selective toward TEA among the tested gases (Figure 8f), its response to trimethylamine and diethylamine under humid conditions may be examined before use in seafood monitoring.

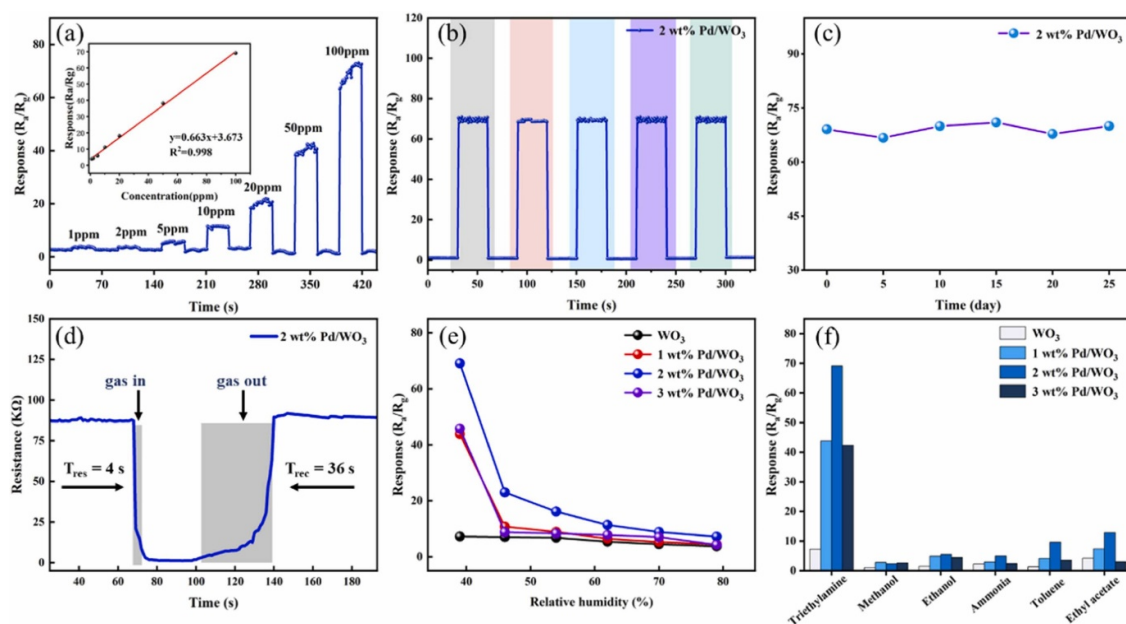


Figure 8. TEA sensing characteristics of Pd-modified WO₃-based sensors at 255 °C: (a) concentration-dependent response of the 2 wt% Pd/WO₃ sensor from 1 to 100 ppm TEA with the corresponding linear fit; (b) repeatability over successive response–recovery cycles; (c) long-term response stability; (d) response and recovery kinetics toward 100 ppm TEA; (e) influence of relative humidity on TEA response; and (f) comparative selectivity of pristine and Pd-modified WO₃-based sensors towards different gases. Reproduced with permission [125].

Bimetallic NPs can enhance catalytic activity, but their individual contributions to the sensing response remain difficult to distinguish. Ma et al. [126] deposited PdRh NPs on flake-like WO₃ and examined the material using density functional theory, diffuse-reflectance infrared spectroscopy, gas chromatography–mass spectrometry, machine learning, and device testing. The optimized 0.6 wt% PdRh@WO₃-based sensor showed a response of 240 to 100 ppm TEA at 240 °C, with response and recovery times of 20 and 27 s, respectively. The results suggest improved TEA adsorption, oxygen activation, and charge transfer. However, the presence of Pd and Rh may affect its scalability and cost-effectiveness for practical applications. In addition, high operating temperature and the dependence of machine-learning models on data quality remain practical limitations. Cao et al. [127] lowered the operating temperature by combining Pd/WO₃ with the N719 photosensitizer. Under white light, the sensor produced a response of 1410 to 50 ppm TEA at 100 °C and a detection limit of 27 ppb. Photoexcited carriers likely supplement thermal activation, while Pd promotes oxygen and TEA conversion. Dark measurements and matched N719/WO₃ and Pd/WO₃ controls are required to separate these effects. Light intensity, spectral distribution, dye stability, and photo-bleaching must also be investigated

for reproducible operations. Dong et al. [128] reported a response of 3323.5 to 50 ppm TEA at 137.5 °C using 1 at% Pt/PtO₂-loaded WO₃. Pt may catalyze oxygen dissociation and TEA oxidation, while PtO₂/WO₃ interfaces modify the resistance barrier. The high response was accompanied by a 262 s recovery time and noticeable H₂ sensitivity, indicating that strong adsorption and catalytic activity may compromise regeneration and selectivity. Zhu et al. [129] developed a noble-metal-free h-BN/WO₃ interface which showed a response of 281.45 to 20 ppm TEA at 100 °C with a detection limit of 20.33 ppb. The response does not arise from bulk conduction through the wide-bandgap h-BN; instead, edge and vacancy defects create localized electronic states and adsorption sites that promote oxygen and TEA adsorption. Charge redistribution at the h-BN/WO₃ interface can also produce an interfacial dipole and potential barrier, allowing surface reactions to modulate the WO₃ conduction pathway and amplify the resistance response rather than increasing the bulk conductivity of h-BN. Response and recovery kinetics, humidity effects, and long-term stability are also required before its practical application. The performance of the WO₃-based gas sensors is summarized in Table 2.

Table 2. Representative performance characteristics of WO₃-based gas sensors for TEA detection.

Optimized Sensing Material	T _{opt} (°C)	TEA (ppm)	Response	Response Time (s)	Recovery Time (s)	LOD	Ref.
C2-400 WO ₃	325	1	2.2	3	5	200 ppb	[100]
WO ₃ -RS-5 (γ-WO ₃)	217	100	281	5	-	-	[101]
WO ₃ -550	220	50	65.56	-	-	399 ppb	[102]
5 wt% In-WO ₃	115	50	11.2	11	40	1 ppm	[103]
5% Cu/WO ₃	180	100	108	33	>760	18.14 ppb	[104]
WGa5 (5 wt% Ga-WO ₃)	150	1	6.7	81	-	100 ppb	[105]
3% Ni-WO ₃	200	100	487	-	-	-	[106]
PtPd@Fe-WO ₃	260	20	409.58	1	108	69.59 ppb	[107]
2.5 at% Pd-WO ₃	160	20	1100	-	-	5 ppb	[109]
1 wt% Cr-WO ₃	240	50	330.1	15	44	42 ppb	[110]
MW2 α-MoO ₃ @WO ₃	200	100	80.4	9	26	127 ppb	[111]
ZrO ₂ -CeO ₂ /WO ₃	-	20	18.56	1	5	0.5 ppm	[82]
NiO-on-WO ₃	300	1	3.62	11	38	1 ppm	[112]
WO ₃ /SnO ₂	260	100	26.8	9	22	-	[113]
W/Fe-0.5 WO ₃ /α-Fe ₂ O ₃	220	50	32.41	8	29	43 ppb	[114]
A/C-2 RuO _x /WO ₃	140	10	36	1	-	0.5 ppm	[115]
WI-3 In ₂ O ₃ /WO ₃	250	10	16.44	-	-	50 ppb	[116]
WMn1 δ-MnO ₂ /WO ₃	140	20	515.37	-	196	15.91 ppb	[117]
5BVO/WO BiVO ₄ /WO ₃	190	-	21	-	-	57 ppb	[119]
rGO-WO ₃ /BiVO ₄	142	5	15.4	-	-	-	[122]
WO ₃ /ErVO ₄	200	100	15.79	-	-	-	[121]
WO ₃ /SnWO ₄	130	200	121.39	50	7	5 ppm	[120]
0.1-ZnWO ₄ /WO ₃	210	50	103.1	10	-	0.5 ppm	[118]
PEDOT:PSS@WO ₃	RT	20	1.58	-	-	-	[123]
Ag/WO ₃ -2	250	100	837	1	-	100 ppb	[124]
2 wt% Pd/WO ₃	255	100	69.08	4	36	37 ppb	[125]
0.6 wt% PdRh@WO ₃	240	100	240	20	27	-	[126]
0.3 wt% N719-Pd/WO ₃	100	50	1410	-	-	27 ppb	[127]
1 at% Pt/PtO ₂ -WO ₃	137.5	50	3323.5	16	262	100 ppb	[128]
h-BN/WO ₃	100	20	281.45	-	-	20.33 ppb	[129]

5. MoO₃-Based Gas Sensors

5.1. Pristine and Doped MoO₃

Li et al. [130] used in situ method to prepare hierarchical α -MoO₃ flower-like films. The proposed gas sensor produced a response of 243.5 for 10 ppm TEA. Its nanosheet assembly and mesoporosity provided accessible diffusion pathways without the structural damage associated with powder coating. These studies show that morphology and device integration may significantly influence TEA sensing even without compositional modification. Doped materials should therefore be evaluated against pristine controls with comparable morphology, loading, film thickness, and substrate contact. Ag-decorated MoO₃ may be treated as a surface-sensitized material rather than a substitutionally doped oxide. Ma et al. [131] deposited Ag on MoO₃ microspheres, which lowered the operating temperature by 20 °C. The proposed gas sensor operated at 220 °C, responded and recovered within 4 and 14 s. It also retained a response of 9.1 at 1 ppm TEA. Ag may promote oxygen activation and TEA oxidation while changing interfacial charge transfer. Its catalytic, defect-related, and electronic contributions remain difficult to separate, and the operating temperature is still relatively high. Rare-earth modification can alter lattice strain, electron trapping, surface acidity, and oxygen adsorption. Zhao et al. [132] compared several rare-earth-modified MoO₃ quasi-core-shell NRs and identified 1% Pr³⁺ as optimal. Its response was 12.3 times that of pristine MoO₃, with improved selectivity, kinetics, moisture tolerance, and performance in seafood-spoilage tests. The comparison also showed that rare-earth modification is strongly dependent on ion identity and loading. Excess dopant may distort the host lattice, form segregated phases, or block accessible surfaces. Chen et al. [133] prepared W-modified MoO₃ flakes for TEA sensing. The 1 mol% W sample showed a response of 340 to 100 ppm TEA at 170 °C and retained a response of 4.4 at 1 ppm. Smaller structural features and oxygen-deficient sites were proposed to explain the improvement, but W substitution, crystallite size, defect concentration, and carrier density changed together. The location and oxidation state of W should therefore be confirmed before attributing the enhanced response primarily to lattice doping. Tan et al. [134] reported Cu-modified MoO₃ nanobelts with a response of 135 to 10 ppm TEA at 240 °C and a detection limit of 12.5 ppb at a Cu/Mo ratio of 7.5%. The low calculated detection limit is promising, but the operating temperature remains high. At this Cu content, Cu-rich clusters or secondary copper oxides should be excluded because they could introduce catalysis or p-n junctions rather than homogeneous lattice modification. The La-MoO₃ series reported by Tian et al. [135] clearly shows the link between dopant loading and morphology. Pristine MoO₃ formed rough microspheres about 900 nm in diameter (Figure 9a–d). The structure remained largely intact at 0.5 and 1 wt% La but became distorted at 2 wt% and collapsed markedly at 4 wt% (Figure 9e–h). This trend suggests a narrow useful loading range in which La modifies the material without destroying its gas-accessible architecture. Elemental maps confirmed a broad distribution of Mo, O, and La (Figure 9i–l), but they do not prove that La entered the MoO₃ lattice. Diffraction, local coordination, and oxidation-state measurements are needed to distinguish lattice substitution from dispersed La-containing surface species. The 1 wt% La sample showed the best sensing performance, with a response of about 30 to 20 ppm TEA at 240 °C (Figure 9m). Its baseline resistance remained higher than that of pristine MoO₃ (Figure 9n), which may increase relative resistance modulation but does not demonstrate a higher active-site density. Carrier concentration, mobility, grain-boundary barriers, and contact resistance may also contribute. The sensor responded more significantly to TEA than to trimethylamine, ammonia, formaldehyde, acetone, and ethanol (Figure 9o), while the response and recovery times improved from 13/34 to 10/29 s (Figure 9p). The sensor showed a consistent response over 2–100 ppm and

good repeatability, but the 14-day test was insufficient to establish long-term calibration stability or humidity tolerance (Figure 9q,r).

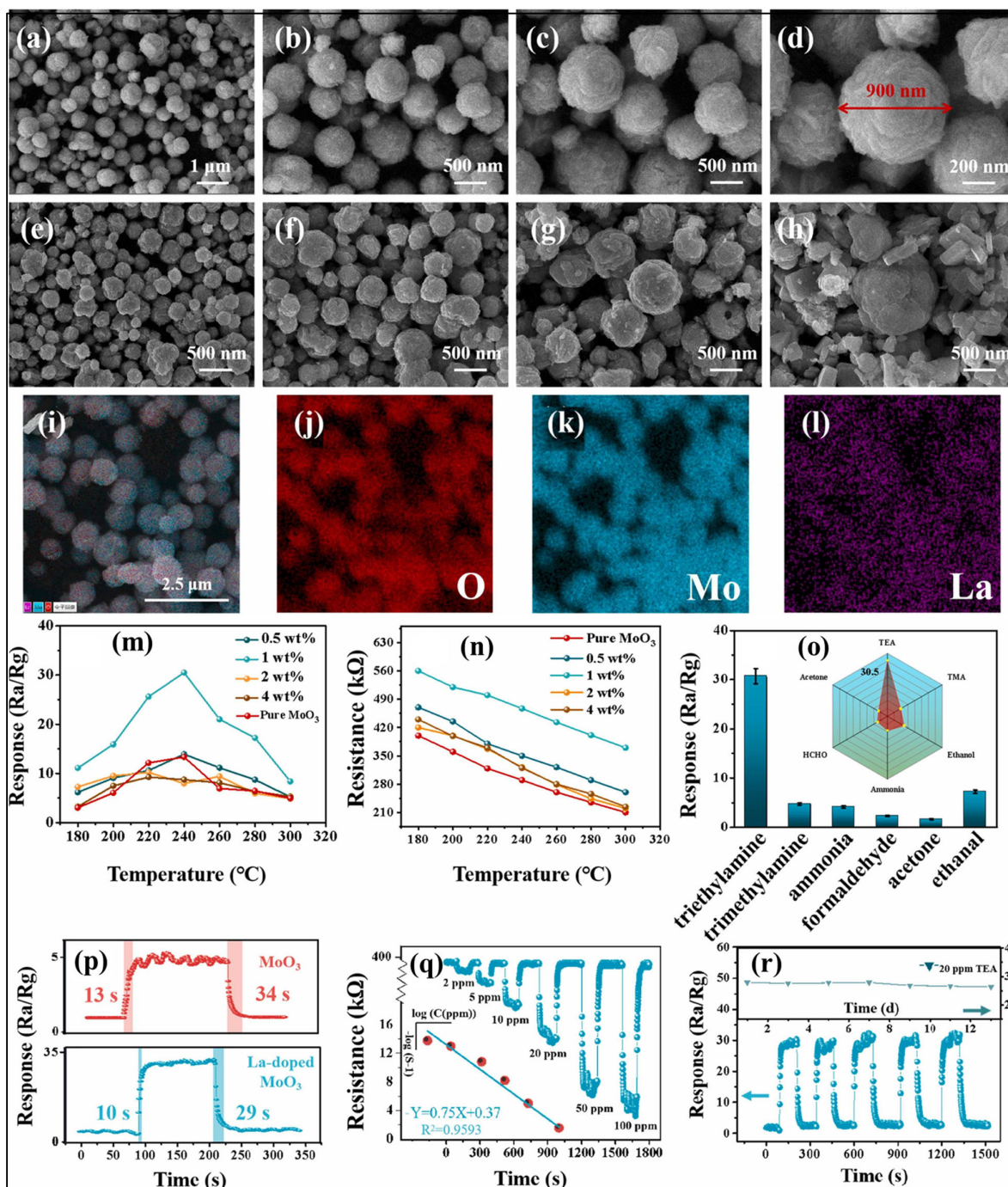


Figure 9. Morphological evolution of MoO₃ microspheres upon La doping: (a–d) SEM images of pristine MoO₃ at different magnifications; (e–h) SEM images of MoO₃ containing 0.5, 1, 2, and 4 wt% La, respectively; and (i–l) elemental mapping of the selected La-doped MoO₃ region, showing the distribution of Mo, O, and La. TEA sensing performance of pristine and La-doped MoO₃ microspheres: (m) temperature-dependent responses to 20 ppm TEA; (n) variation in baseline resistance with operating temperature; (o) selectivity of 1 wt% La-doped MoO₃ towards different gases; (p) comparison of response and recovery kinetics for pristine and 1 wt% La-doped MoO₃; (q) concentration-dependent sensing behavior from 2 to 100 ppm TEA with the corresponding calibration relationship; and (r) repeatability over successive sensing cycles and long-term stability over 14 days. Reproduced with permission [135].

Lv et al. [136] reported a response of 1148 to 50 ppm TEA for La-modified MoO₃ NRs. This response may be attributed to defect formation, oxygen-vacancy-related chemistry, and increased baseline resistance. Jia et al. [137] combined surface characterization and density functional theory to examine Gd-modified MoO₃ NRs. The optimized 3 mol% Gd sample showed a response of about 65.8 to 10 ppm TEA at 150 °C and responded within 16 s. The experimental and computational results provide a stronger basis for linking Gd to changes in adsorption and electronic structure, although systematic humidity testing is still needed for seafood applications. Yue et al. [138] prepared mesoporous Co-MoO₃ nanoribbons through a metal–organic-framework-derived route. MCo-MoO₃-450 delivered a response of 361.62 to 50 ppm TEA at 110 °C with a detection limit of 137 ppb. MOF-derived porosity may improve gas access, whereas Co may alter surface redox chemistry and carrier trapping. Directly deposited pristine films benefit from preserved morphology and improved electrical contact; Ag provides surface catalysis; W, Cu, and rare-earth ions alter lattice, defect, and adsorption properties; and MOF-derived Co-MoO₃ combines doping with engineered porosity. Future studies may establish whether the added metal occupies lattice sites, remains at the surface, or forms a secondary phase. Morphology-matched controls, work-function and carrier-density measurements, temperature-dependent conductivity, and operando spectroscopy would help to separate electronic regulation from catalytic activity. Humidity cycling, recovery completeness, baseline drift, structural aging, and device variability should be assessed alongside sensitivity to determine whether modified MoO₃ provides a reproducible advantage over well-designed pristine films.

5.2. MoO₃/Metal-Oxide-Based Materials

Integration of MoO₃ with another oxide may alter band bending, oxygen activation, charge transport, and gas diffusion. In n-n heterojunctions, carrier redistribution modifies interfacial resistance, whereas p-n junctions form wider depletion regions. Because junction formation changes morphology, porosity, surface composition, and defect density, matched controls are essential. Zhang et al. [139] reported that ZnO/MoO₃ demonstrated a response of 519 to 100 ppm TEA at 180 °C, retained a response of 9.3 at 500 ppb, and had a detection limit of 10 ppb. DFT calculations supported favorable interfacial TEA adsorption, although the effects of morphology, oxygen deficiency, and oxide loading were not fully resolved. Humidity cycling and interference tests with related amines are still needed for seafood applications. CeO₂-modified MoO₃ NRs showed a response of 73.1 to 20 ppm TEA at 240 °C and a detection limit of 20.9 ppb [140]. Ce³⁺/Ce⁴⁺ may contribute to maintain reactive surface oxygen, in addition to modifying charge transfer at the n-n interface. Seafood tests were promising but the high operating temperature and complex composition of fish headspace remain important limitations. Chromatographic analysis is needed to confirm that the signal originates mainly from TEA. The WO₃-MoO₃ heterostructure reported by Zhu et al. [141] exhibited a response of 627.28 to 20 ppm TEA at 100 °C. The response was ascribed to the charge redistribution and oxygen-deficient interfacial regions. Measurements over 1–20 ppm delivered a calculated detection limit of 43.7 ppb (Figure 10a,b). At 20 ppm, the sensor responded within 22 s but required 227 s to recover (Figure 10c), indicating slow desorption or product removal. It also showed decent selectivity for TEA compared to the tested volatile compounds and ammonia, but related amines were not explored (Figure 10d). The response remained above 500 after 30 days but declined by about 16% (Figure 10e). This may be described as measurable drift rather than complete stability. The response decreased as humidity increased (Figure 10f), indicating persistent water interference and the need for humidity compensation in quantitative measurements.

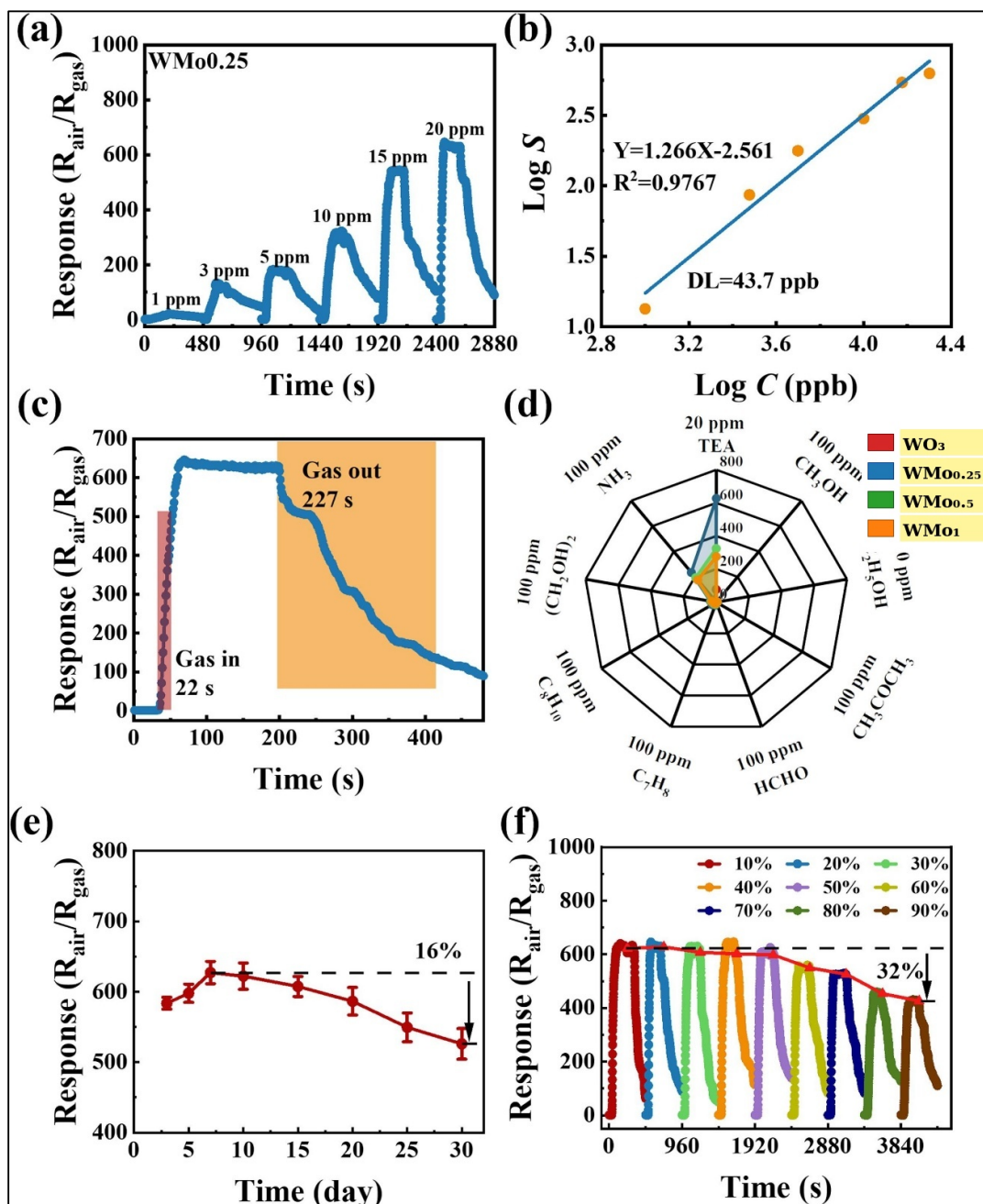


Figure 10. TEA sensing performance of the optimized WMo_{0.25} (WO₃-MoO₃) heterojunction sensor at 100 °C: (a) dynamic responses toward 1–20 ppm TEA; (b) log(response)-log(concentration) calibration plot with the estimated detection limit; (c) response and recovery behavior towards 20 ppm TEA; (d) selectivity toward TEA relative to other volatile organic compounds and ammonia; (e) long-term response stability over 30 days; and (f) influence of relative humidity on the transient TEA response. Adapted with permission [141].

Liu et al. [142] prepared porous α -MoO₃/TiO₂ heterostructures from Mo-MOF/Ti₃C₂T_x derived precursors. The sensor showed a response of 566.7 to 100 ppm TEA, along with rapid kinetics, selectivity, and stable operation. Thermal conversion produced porosity and defect-rich surfaces. The α -MoO₃/TiO₂ interface provided n-n charge regulation. Because these changes occurred simultaneously, controls prepared from the individual components under identical conditions are needed to determine the specific contribution of the junction. In another study, a p-n system-based gas sensor was also developed by depositing NiO/ α -MoO₃ hollow microspheres on ceramic tubes [143]. It

exhibited a response of 188.88 to 50 ppm TEA at 217 °C, a detection limit of 0.1 ppm, and good humidity tolerance. The p-n interface may amplify resistance changes through depletion-layer modulation, whereas the hollow structure and direct deposition improve gas diffusion and electrical contact. Its performance therefore reflects both junction behavior and device architecture. As a non-chemiresistive comparator, Dong et al. [144] reported a mixed-potential GDC sensor using a SmCoO₃/MoO₃ composite sensing electrode, with good selectivity, humidity tolerance, and six-week stability. Because its transduction principle differs from chemiresistive sensing, it was not included in Table 3 and should not be compared directly using resistance-response metrics.

Table 3. Representative performance characteristics of MoO₃-based sensors for TEA detection.

Optimized Sensing Material	T _{opt} (°C)	TEA (ppm)	Response (As Reported)	Response Time (s)	Recovery Time (s)	LOD	Ref.
Hierarchical α-MoO ₃ flowers	133	10	243.5	-	-	0.001 ppm	[130]
Ag/MoO ₃ microspheres	220	1	9.1	4	14	-	[131]
1 mol% W-MoO ₃	170	100	340	-	-	1 ppm	[133]
7.5% Cu/Mo-MoO ₃	240	10	135	-	-	12.5 ppb	[134]
1 wt% La-MoO ₃	240	20	30	10	29	2 ppm	[135]
5 mol% La-MoO ₃	235	50	1148	-	-	1 ppm	[136]
3 mol% Gd-MoO ₃	150	10	65.8	16	-	-	[137]
MCo-MoO ₃ -450	110	50	361.62	-	-	137 ppb	[138]
ZnO/MoO ₃	180	100	519	9	16	10 ppb	[139]
MoO ₃ /CeO ₂	240	20	73.1	13	9	20.9 ppb	[140]
WMo _{0.25} WO ₃ -MoO ₃	100	20	627.28	22	227	43.7 ppb	[141]
MMT-2 α-MoO ₃ /TiO ₂	297	100	566.7	-	-	1 ppm	[142]
2.98 NiO/α-MoO ₃	217	50	188.88	-	-	0.1 ppm	[143]
MoO ₃ -CsPbBr ₃ QDs	240	50	41.06	4	7	-	[145]
MoO ₃ /CsPbBr ₃ QDs	260	50	25.00	-	-	-	[146]
ZnSe/Cr-MoO ₃ -2	200	10/50	371/1301	-	-	1.7 ppb	[147]
2 wt% h-BN/MoO ₃	260	500	8616	-	-	-	[148]
5% Pd-Fe/MoO ₃	180	100	415	20	79	-	[149]
0.5 wt% Y ₂ O ₃ /MoO ₃	160	100	385.65	-	-	50 ppb	[150]

5.3. MoO₃/Perovskite-Based Materials

CsPbBr₃/MoO₃ is a promising hybrid heterostructure for gas-sensing applications. Its sensing response likely arises from interfacial charge transfer, defect-mediated adsorption, and altered surface coverage. Photo-induced charge carriers contribute under controlled illumination. Feng et al. [145] decorated 1D MoO₃ nanobelts with CsPbBr₃ quantum dots as shown in Figure 11. The optimized composite showed a response of 41.06 to 50 ppm TEA at 240 °C, with response and recovery times of 4 and 7 s. The rapid kinetics may reflect faster interfacial charge exchange or surface reactions, but quantum-dot deposition also changes morphology, defect density, and accessible surface area. Pristine MoO₃, physically mixed MoO₃/CsPbBr₃, and controlled dark–light measurements are needed to confirm the proposed interface mechanism. Operation at 240 °C also requires evidence of quantum-dot and ligand stability, humidity tolerance, long-term phase integrity, and safe Pb containment.

Tian et al. [146] also investigated MoO₃ microspheres modified with CsPbBr₃ under photo-assisted conditions. The composite showed a response of 25 to 50 ppm TEA at 260 °C, and DFT calculations indicated favorable adsorption and interfacial electronic redistribution. The proposed low-power benefit also remains unproven because both systems operated at 240–260 °C, with illumination adding another energy input. Humidity effects,

signal drift, light-cycling stability, and interface durability require further investigation before $\text{MoO}_3/\text{CsPbBr}_3$ can be considered a practical photo-assisted TEA sensor.



Figure 11. Schematic illustration of the preparation of MoO_3 and $\text{MoO}_3\text{-CsPbBr}_3$ composite. Reproduced with permission [145].

5.4. Other MoO_3 -Based Scenarios

Multicomponent MoO_3 -based sensors combine doping, semiconductor interfaces, noble metals, or two-dimensional materials. Although these designs can enhance response, they also change defect chemistry, morphology, charge transport, and baseline resistance at the same time. Single-component and binary controls are therefore essential. Tan et al. [147] combined Cr-modified MoO_3 with ZnSe . The fabricated n-n heterostructure showed responses of 371 and 1301 to 10 and 50 ppm TEA, respectively, with a detection limit of 1.7 ppb. Cr may alter defect chemistry, the nest-like structure improves gas access, and ZnSe can modify interfacial resistance. Because all three effects were introduced together, the response cannot be assigned to the junction. Chang et al. [148] wrapped MoO_3 NWs with h-BN and obtained a response of 8616 to 500 ppm TEA. This large value was attributed to increased adsorption and altered charge transport. However, h-BN is a wide-bandgap material and may act through edge defects, interfacial dipoles, or changes in the MoO_3 conduction pathway rather than a conventional depletion-layer mechanism. Li et al. [149] prepared porous Fe-modified MoO_3 nanobelts decorated with Pd. The optimized 5% Pd-Fe/ MoO_3 -based sensor showed a response of 415 to 100 ppm TEA at $180\text{ }^\circ\text{C}$, with response and recovery times of 20 and 79 s, respectively. Fe may regulate lattice defects and carrier trapping, whereas Pd promotes oxygen dissociation, TEA oxidation, and Schottky-barrier modulation. The response likely results from both electronic and catalytic effects, while the slow recovery indicates incomplete surface regeneration. Rahmatullah et al. [150] reported a noble-metal-free $\text{Y}_2\text{O}_3/\text{MoO}_3$ -based sensor. At 0.5 wt% Y_2O_3 , the composite produced a response of 385.65 to 100 ppm TEA at $160\text{ }^\circ\text{C}$ and a detection limit of 50 ppb. The improvement was ascribed to larger pores, oxygen-related defects, and interfacial charge modulation. Given the wide bandgap and strong metal–oxygen bonding of Y_2O_3 , changes in surface acid–base character and adsorption-site distribution may be more important than direct catalysis. The gas-sensing performance of the MoO_3 -based sensors is summarized in Table 3.

6. Challenges and Perspectives

Previous studies on WO_3 - and MoO_3 -based TEA sensors have led to marked improvements in sensitivity, but exploring these advances in practical devices remains challenging.

Many reported sensors still require high operating temperatures and are influenced by humidity, poor recovery, and signal drift. Further progress will therefore depend not only on reporting higher responses under controlled laboratory conditions, but also on clarifying the sensing mechanisms, controlling the material properties that affect performance, and demonstrating stable and reproducible operation under realistic conditions.

6.1. Fundamental Mechanisms

Interpreting the sensing mechanism remains difficult because doping and composite formation sometimes alter several material properties at the same time, including morphology, electrical resistance, porosity, defect density, and surface composition. Performance improvements are generally attributed to oxygen vacancies or improved charge transfer, although the main contributing factor is rarely identified experimentally. More rigorous mechanistic studies therefore require suitable controls with comparable morphology, active-material loading, film thickness, and electrode contacts. Multicomponent sensors should also be evaluated alongside their individual constituents and corresponding physical mixtures prepared and tested under identical conditions. A clearer understanding of the sensing mechanism requires direct observation of the sensor surface under operating conditions. Operando infrared and Raman spectroscopy, ambient-pressure X-ray photoelectron spectroscopy, electron paramagnetic resonance, and gas chromatography–mass spectrometry could reveal adsorbed intermediates, changes in metal oxidation state, and the products formed during TEA oxidation. Together with work-function measurements, impedance spectroscopy, temperature-dependent conductivity, and oxygen temperature-programmed desorption, these analyses could suggest whether the sensor response arises mainly from surface catalysis, electronic sensitization, or changes in the depletion layer. Isotope-labeling experiments using oxygen or TEA could further trace the origin and fate of the reacting species, providing more direct evidence for the roles of lattice oxygen, adsorbed oxygen, and reaction intermediates. Water vapor should be considered part of the sensing mechanism rather than merely an external source of interference. Controlled-humidity measurements can clarify whether water competes with TEA for adsorption sites, changes the degree of surface hydroxylation, influences charge transport, or hinders TEA oxidation. Photo-assisted sensors require similarly rigorous controls such as dark measurements, illuminated tests of the pristine oxide, and complete reporting of the light source and irradiation conditions. These comparisons are essential for establishing whether the improved response originates from photo-activation, interfacial sensitization, or unintended heating.

6.2. Rational Material Design

It is notable that efficient sensor design requires a careful balance among TEA adsorption, surface reaction, charge modulation, and sensor regeneration. Strong adsorption may increase the response, but it can also delay recovery and favor the accumulation of reaction intermediates. A moderate yet reversible response, accompanied by rapid recovery and minimal drift, may therefore be more valuable in practice than a much larger but unstable signal. WO_3 and $\alpha\text{-MoO}_3$ provides distinct opportunities for optimization. In WO_3 , sensing behavior is significantly influenced by phase composition, exposed facets, grain boundaries, work function, and $\text{W}^{5+}/\text{W}^{6+}$ redox chemistry. For $\alpha\text{-MoO}_3$, the layered framework, chemically distinct oxygen sites, $\text{Mo}^{6+}/\text{Mo}^{5+}$ redox couple, and Lewis-acidic surface centers provide additional means of tuning amine adsorption and surface reactivity. Computational models may account for these structural differences by examining TEA adsorption on experimentally relevant facets and defect sites rather than relying solely on ideal surfaces. They should also consider competitive adsorption involving water and chemically related interferents such as trimethylamine, diethylamine, and ammonia. The

position and oxidation state of an added element are as important as its identity. Lattice substitution, surface segregation, and secondary-phase formation can influence sensing in very different ways, even when the same precursor is used. Defect concentration should likewise be carefully controlled rather than simply increased, because excessive disorder can disrupt charge transport and contribute to signal drift. Noble-metal-free heterojunctions, stable 2D materials, and well-defined amorphous–crystalline interfaces may offer more affordable alternatives, but their benefits must be established through comparison with closely matched pristine materials. Practical development should also consider scalability, thermal stability, reproducibility, and environmental safety alongside sensing performance.

6.3. MEMS, Flexible Platforms, and Practical Device Engineering

Conventional ceramic-tube sensors remain valuable for laboratory studies, but their high thermal mass and power consumption restrict their use in portable devices. MEMS microhotplates may address these limitations by confining heat to a suspended membrane, allowing rapid temperature modulation at much lower power. For example, the optimized microheater developed by Zhou et al. reached 300 °C with a continuous power consumption of approximately 2 mW and a thermal response time of only 33 μ s [151]. The suitability of WO_3 for this system was demonstrated by a CuO/WO_3 MEMS sensor which exhibited faster response and recovery and decent long-term stability towards xylene [152]. Similarly, MoO_3 -loaded SnO_2 was integrated into a MEMS microheater and operated using a double-pulse heating mode, enabling the adsorption and partial oxidation steps involved in ethanol sensing to be regulated through the temperature sequence [153]. Although these studies focused on analytes other than TEA, they provide a practical basis for integrating WO_3 and MoO_3 containing sensing layers into low-power, temperature-modulated TEA sensors. Flexible TEA sensing has been more clearly demonstrated using conducting-polymer/metal-oxide hybrids. Sun et al. [154] deposited PPy/WO_3 on flexible PET substrates and achieved room-temperature detection across a TEA concentration range of 10–80 ppm. PANI/WO_3 films fabricated on PET exhibited a response of 81 at 100 ppm TEA, approximately 13 times higher than that of pristine PANI [19]. Similarly, an $\alpha\text{-MoO}_3/\text{PANI}$ network deposited on PET showed an approximately linear response to 10–100 ppm TEA at room temperature [155]. These studies demonstrate the feasibility of flexible, heater-free TEA sensors. The response of these hybrid sensors may reflect several concurrent processes such as polymer protonation, humidity-induced swelling, interfacial charge transfer, and changes across the p-n junction. Their practical reliability should therefore be examined through repeated bending, baseline measurements under different strains, adhesion tests, humidity cycling, and long-term drift monitoring. For MEMS-based sensors, heater power, temperature uniformity, operating duty cycle, and device-to-device reproducibility should also be studied. Humidity remains a major concern for the reliable quantification of TEA. Water interference may be reduced or corrected using hydrophobic gas-permeable membranes, molecular sieves, catalytic filters, reference channels, or integrated humidity sensors. However, any protective layer must allow efficient TEA transport without introducing substantial delays in response and recovery. Sensor performance should be evaluated over repeated dry–humid cycles by monitoring both response retention and baseline drift. Calibration models may also treat temperature and humidity as explicit variables rather than fixed experimental conditions. In addition, long-term stability cannot be established from a few response–recovery cycles. Prolonged operation and repeated analyte exposure may lead to grain growth, irreversible surface hydroxylation, noble-metal agglomeration, interfacial diffusion, elemental segregation, polymer degradation, or changes in contact resistance. Accelerated aging tests, combined with post-test structural and chemical analyses, can reveal the mechanisms responsible for

performance degradation. Reproducibility should be confirmed across multiple devices fabricated from independent material batches. Essential parameters such as film thickness, material loading, power consumption, electrode geometry, baseline resistance, and calibration drift should also be reported consistently.

6.4. System-Level Application

Seafood headspace contains a complex and continuously changing mixture of amines, alcohols, aldehydes, ketones, and sulfur-containing compounds. Under these conditions, a single sensing material may not reliably distinguish TEA from related amines such as trimethylamine, diethylamine, and ammonia. Sensor arrays combining materials with different adsorption affinities and catalytic activities could generate more distinctive response patterns. Selectivity may be further improved by extracting additional information from temperature-modulated signals, impedance spectra, and response–recovery kinetics. Machine learning can support gas classification, concentration estimation, and drift correction, but it cannot overcome limitations in experimental design. Training datasets should include multiple devices and independent synthesis batches evaluated across realistic ranges of analyte concentration, humidity, temperature, and background composition. Randomly assigning measurements from the same device to both training and test sets can lead to data leakage and unrealistically high accuracy. Evaluating models with independently fabricated sensor batches and data collected over extended periods provides a more reliable indication of real-world performance, particularly when aging and signal drift are expected. For seafood applications, sensor performance must be validated against established measures of freshness and spoilage. Sensor outputs should be correlated with gas chromatography–mass spectrometry, total volatile basic nitrogen, microbial counts, sensory evaluation, and documented storage conditions. Validation should cover different seafood species, storage temperatures, packaging environments, and stages of spoilage, since TEA is only one of several volatile freshness indicators. A practical monitoring platform will therefore likely require a sensor array integrated with temperature and humidity measurements, together with drift correction and product-specific calibration. The key challenge is to translate highly responsive laboratory materials into reproducible, energy-efficient sensors which maintain their selectivity and calibration in humid and chemically complex environments.

7. Conclusions

WO_3 and $\alpha\text{-MoO}_3$ could not be treated as interchangeable n-type sensing oxides. WO_3 is most effectively tuned through control of phase, exposed facets, work function, grain boundaries, and catalytic interfaces, whereas $\alpha\text{-MoO}_3$ derives its distinctive TEA response from its layered anisotropy, chemically inequivalent oxygen sites, variable Mo valence, and Lewis-acidic surface centers. Across the surveyed studies, material modification frequently improved sensitivity and lowered operating temperature, but the largest reported response did not necessarily correspond to the most practical sensor because slow recovery, humidity interference, signal drift, high power demand, and inconsistent response definitions often limited comparison and use. Future progress should therefore prioritize balanced performance: low-power operation, rapid and complete recovery, humidity tolerance, stable calibration, reproducible fabrication, and selectivity against related volatile amines. Mechanistic claims require matched controls and operando or interfacial measurements capable of distinguishing catalytic activation, defect-mediated adsorption, and junction-induced charge modulation. For seafood monitoring, validation should extend from single-gas tests to humid, mixed-vapor headspace and correlate sensor output with established freshness indicators. MEMS platforms and sensor arrays may reduce power consumption and improve discrimination, provided that long-term drift and device-to-device variability are

reported. These priorities are more important for translation than further increases in peak response under idealized laboratory conditions.

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