

Sensitivity Enhancement of Benzene, Toluene, and Xylene Gas Sensors using Nanostructured TiO₂: a Mini-Review

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ABSTRACT: Benzene, toluene, and xylene (BTX) are hazardous volatile organic compounds commonly found in industrial and environmental settings, requiring the development of highly sensitive and selective gas sensors to ensure public safety. Nanostructured titanium dioxide (TiO₂) has emerged as a promising BTX-sensing material due to its high chemical stability, advantageous catalytic properties, and tunable electronic structure. However, material modification is required because of the limited gas sensitivity of pristine TiO₂. This review discusses key strategies for enhancing the BTX-sensing performance of TiO₂-based gas sensors, including nanostructuring, metal doping, metal and metal oxide decoration, thermal treatment, and organic functionalization. These approaches improve gas adsorption, charge-carrier mobility, and reaction kinetics, thus enhancing the response and selectivity of the resulting sensor. The integration of TiO₂ sensors with microheater platforms also enables low-power operation for practical deployment. This review highlights current challenges facing the development of TiO₂-based gas sensors, including sensor stability, cross-sensitivity, and real-time monitoring while proposing future directions for these devices.

KEYWORDS: Nanostructured TiO₂, BTX gas sensing, Sensitivity enhancement, Decoration, Functionalization

1. INTRODUCTION

Benzene, toluene, and xylene (BTX) are petrochemical solvents used in numerous industries, including fuel production, pharmaceuticals, and rubber manufacturing [1]. They are also frequently detected in everyday products and facilities, such as adhesives, paint, vehicles, gas stations, and dry cleaners [2]. Thus, BTX compounds have become significant contributors to outdoor and indoor air pollution and pose a significant risk to human health. Benzene is particularly harmful because it is classified as a first-class carcinogen and is associated with an increased risk of leukemia and other cancers [3,4]. Although toluene and xylene are not classified as carcinogens, long-

term exposure can seriously affect blood production and the nervous system [5]. Because of these risks, the US National Institute for Occupational Safety and Health has set permissible exposure limits of 0.1, 100, and 100 ppm (8 h weighted average) for BTX, respectively [6].

Several BTX detection methods have been developed to protect public health, although many have operational limitations. For example, although gas chromatograph mass spectrometry has been demonstrated to produce accurate results in the detection of BTX, this approach lacks portability and is not cost-effective, hindering the establishment of monitoring systems that deploy sensors in multiple locations [7]. An alternative strategy is to use metal oxides as a chemiresistive material that promotes redox reactions between BTX and oxygen ions on the metal-oxide surface [8], facilitating the development of highly sensitive and inexpensive metal oxide-based BTX sensors [9]. Titanium dioxide (TiO₂), a high-resistance n-type semiconductor with a bandgap of approximately 3 eV, has attracted particular attention as a gas-sensing metal oxide owing to its high chemical stability, strong catalytic properties, and nontoxicity [10]. TiO₂ exists in three common phases

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(rutile, anatase, and brookite) that are composed of TiO_6 octahedrons with various connection patterns. Rutile and anatase are more stable and widely used, with anatase transitioning to rutile at 600–700°C [11]. However, all TiO_2 phases generally exhibit lower sensitivity to target gases than other metal oxides because of their inherent chemical stability. Therefore, several strategies have been proposed to enhance the sensitivity of TiO_2 -based gas sensors while maintaining their practical advantages.

This review summarizes strategies devised to enhance the sensitivity of TiO_2 -based gas sensors for the detection of BTX together or as individual components. First, we introduce the advantages of using nanostructures for gas detection and describe the BTX-sensing mechanisms of TiO_2 . We then review the various methods developed to increase the BTX sensitivity of TiO_2 nanostructures, including doping and/or decorating with metals, metal oxides, or organic materials. Finally, this review outlines the current challenges and future outlook for TiO_2 -based BTX sensors.

2. BTX-sensing mechanisms for TiO_2

Chemiresistive TiO_2 -based BTX sensors function using redox reactions on the surface that alter the electrical resistance of TiO_2 . When ambient air is introduced to TiO_2 , oxygen vacancies become active sites for the chemisorption of oxygen ions (O , O_2^- , and O^{2-}) at higher temperatures [12]. This induces the formation of a depletion layer in the nanostructured TiO_2 (Fig. 1 (a), left), which acts as a potential barrier between the nanostructures (Fig. 1 (b), left) [13], increasing the electrical resistance of TiO_2 . Redox reactions between BTX molecules and chemisorbed oxygen ions then occur [14], resulting in the oxidation of BTX to form oxygen and water molecules and the simultaneous transfer of electrons captured by oxygen ions to

the TiO_2 (Fig. 1 (a), right). This decreases the thickness of the depletion layer and the height of the potential barrier (Fig. 1 (b), right), reducing the electrical resistance of the TiO_2 . Consequently, the BTX concentration can be estimated based on the degree to which the resistance changes.

3. Nanostructured TiO_2 for BTX sensing

Nanostructured materials, such as nanoparticles, nanowires, and nanotubes, are particularly effective for gas-sensing applications because of their unique physical, chemical, and structural properties. These include a high surface-to-volume ratio and the presence of abundant active sites for the adsorption and interaction with gas molecules [15–17]. These nanostructures offer higher sensitivity and faster response times compared to bulk materials [18], while their structure can be tailored to optimize sensor performance by reducing operating temperatures and power consumption and enhancing operational safety [19]. TiO_2 nanotubes can be synthesized using wet-chemical methods [20], which has prompted the development and testing of various TiO_2 nanostructures as BTX-sensing materials. Seo et al. [21] investigated the sensing performance of TiO_2 nanotubes and nanoparticles for toluene detection. TiO_2 nanotubes, synthesized using a hydrothermal method and calcinated at 600°C for 1 h (Fig. 2 (a)), produced the strongest response to toluene. The selective response of TiO_2 nanotubes to toluene in the presence of CO, H_2 , and ethanol was also higher than that of nanoparticles. This was attributed to the porous structure of nanotubes, which selectively promoted the diffusion of toluene molecules containing benzene rings compared to other gases (Fig. 2 (b)). The same research group also investigated the effects of controlling the porosity of TiO_2 nanotubes via ball-milling treatment [22]. This treatment method shortened the TiO_2 nanotubes and increased their packing density, optimizing their response and selectivity to toluene.

In another study, Kida et al. [23] produced a sensor that integrated TiO_2 nanotubes with a Si membrane, microheater, and electrodes using micromechanical systems technology (Fig. 2 (c)). The presence of the microheater significantly reduced the power consumption, indicating that the sensor was suitable for portable, battery-operated applications. However, the poor adherence of the sensing layer to the electrodes resulted in a higher resistance. The response to toluene was lower than that to ethanol because of its larger molecular size and slower diffusion through the thick sensing film. Investigating another morphological form, Dutta et al. [24] reported the BTX-sensing characteristics of p-type nanoporous TiO_2 thin films at relatively low temperatures. The fabricated sensor exhibited fast response and recovery times (<50 and 30 s, respectively) across a range of relative humidity levels (Fig. 2 (d)). Although

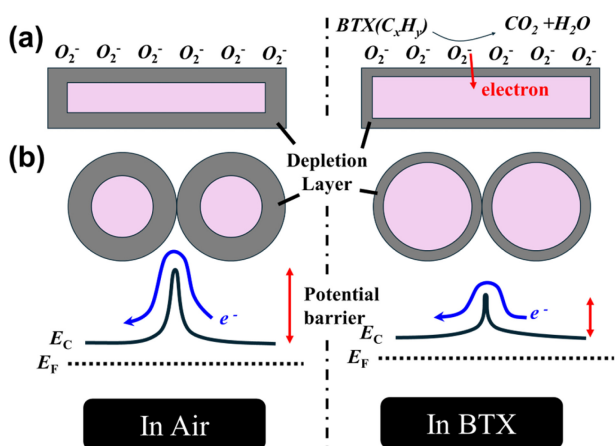


Fig. 1. BTX-sensing mechanisms of nanostructured TiO_2 . Change in (a) thickness of depletion layer and (b) potential barrier when exposed to BTX.

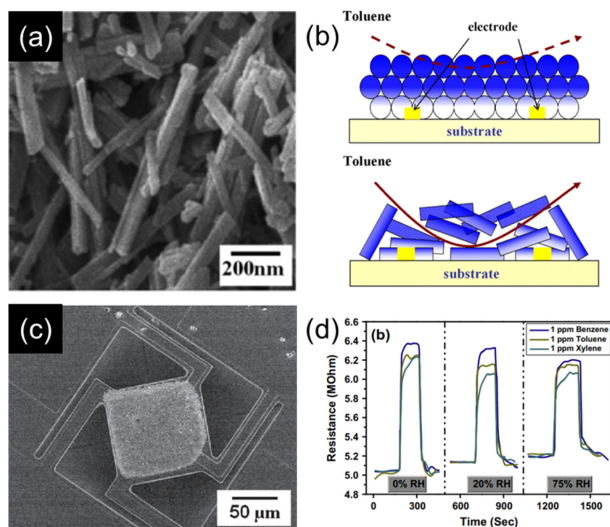


Fig. 2. Nanostructured TiO₂ for BTX sensing. (a) Hydrothermally synthesized and calcinated TiO₂ nanotubes. (b) Schematic model of the diffusion of toluene gas inside nanoparticles and nanotubes. Reprinted with permission from Ref. [21], Copyright (2009) ELSEVIER SCIENCE SA. (c) Scanning electron microscope image of a microheater combined with TiO₂ nanotubes. Reprinted with permission from Ref. [23], Copyright (2013) IOP Publishing Ltd. (d) Transient response of a p-type nanoporous TiO₂ thin film to BTX at a relative humidity of 0, 25, and 75%. Reprinted with permission from Ref. [24], Copyright (2015) PERGAMON-ELSEVIER SCIENCE LTD.

the response to 1 ppm BTX decreased as the relative humidity increased owing to the occupancy of adsorption sites by water molecules, the performance was more robust than that of other metal oxide-based sensors.

4. Nanostructured TiO₂ with metal doping or decoration for BTX sensing

The doping or decoration of semiconductor metal oxides with metals has been widely used to enhance gas-sensing properties such as the sensitivity, selectivity, response time, and stability [25]. Metal doping modifies the microstructure, electronic properties, and surface chemistry of metal oxides, promoting stronger interactions with target gases [26]. In particular, dopants such as novel metals (e.g., Pt and Au) catalyze reactions by lowering the activation energy and creating oxygen spillover effects, which improve gas adsorption and desorption kinetics [27]. Transition metal doping can generate oxygen vacancies [28], which act as reactive sites for gas molecules, significantly enhancing conductivity. Doping also influences the microstructure by inhibiting the grain growth and reducing the particle size [29], which increases the surface area and adsorption capacity. Moreover, dopants alter the electronic

band structure, modulating the carrier concentration and band bending [30], which are required for the sensing of oxidizing and reducing gases. Owing to these benefits, metal doping has also been employed in TiO₂-based sensors to induce redox reactions and increase their sensitivity to BTX. For example, Ni-doped bowl-like TiO₂ particles exhibited 2.4 times stronger response than pure particles to 100 ppm xylene at 302°C (Fig. 3 (a)) [31]. Ni doping also reduced the optimal operating temperatures for xylene detection from 324°C for pure TiO₂ particles to 302°C for 2 mol% Ni-doped TiO₂ particles (Fig. 3 (a)). Fig. 3 (b) presents the response characteristics of pure and 2 mol% Ni-doped TiO₂ particles in the presence of xylene at a concentration of 100 ppm, with fast response and recovery times observed (12 and 9 s for the pure and 2 mol% Ni-doped particles). These rapid response and recovery times were attributed to the bowl-shaped particles, which contained numerous macropores that facilitated molecular diffusion.

Metal decoration enhances the sensitivity to BTX based on chemical and electronic sensitization. In electronic sensitization, differences in work functions promote the transfer of electrons from metal oxides to noble metals, forming Schottky barriers and increasing oxygen chemisorption, thus amplifying changes in resistance following gas exposure [32]. Chemical sensitization, also known as the spillover effect, is based on the noble metal-assisted dissociation of gas molecules into more reactive species, promoting their interaction with surface oxygen [33]. Thus, the chemical and electronic sensitization of TiO₂ nanostructures using metal decoration has been investigated for gas-sensing applications. For example, pecan-kernel-like hierarchical TiO₂ nanostructures were synthesized via the hydrothermal method and then decorated with Au via chemical precipitation to detect toluene gas [34]. Fig. 3 (c) shows the response of these TiO₂ nanostructures to 100 ppm toluene under various operating temperatures and Au loadings. The sensor produced an optimal response at an Au loading of 5 wt%, increasing from 2.3 to 7.3 ($R_{\text{air}}/R_{\text{gas}}$) at 375°C. This improvement originated from the catalytic effect of Au, which promoted the formation of oxygen ions at the interface between the Au and TiO₂. This induced a change in the thickness of the depletion layer when exposed to toluene, enhancing the sensitivity of the proposed sensing material (Fig. 3 (d)).

Maake et al. highlighted the significant role of bimetallic decoration in enhancing xylene sensing based on synergistic catalytic effects and modifications to electronic properties [35]. AgCu nanoparticles were hydrothermally synthesized and loaded onto TiO₂ nanoparticles, with the synergistic interaction between Ag and Cu strongly improving the response to xylene compared to other BTX compounds. This enhancement was attributed to electronic sensitization, with the introduction of Ag and Cu modifying the charge-carrier dynamics, leading to

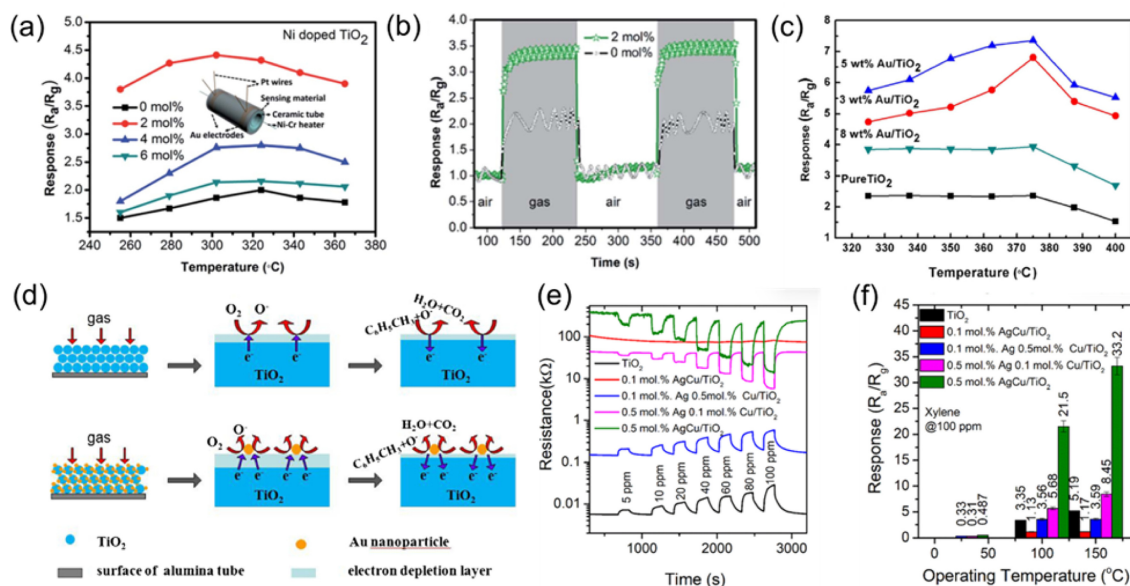


Fig. 3. Nanostructured TiO₂ with doped or decorated metals for BTX sensing. (a) Response of Ni-doped bowl-like TiO₂ particles to 100 ppm xylene at different temperatures. (b) Transient response to 100 ppm xylene with and without Ni doping. Reprinted with permission from Ref. [31], Copyright (2015) ROYAL SOC CHEMISTRY. (c) Response of TiO₂ nanostructures to 100 ppm toluene under various operating temperatures and Au loadings. (d) Schematic illustration of changes in the thickness of the depletion layer with and without Au decoration. Reprinted with permission from Ref. [34], Copyright (2018) ELSEVIER SCIENCE SA. (e) Changes in the transient resistance of pure and AgCu-decorated TiO₂, showing a p- to n-type transition as the AuCu loading increases. (f) Response of TiO₂ nanoparticles at different operating temperatures and various AgCu loadings. Reprinted with permission from Ref. [35], Copyright (2022) ROYAL SOC CHEMISTRY.

a p- to n-type transition at higher metal loadings (Fig. 3 (e)). This transition resulted from electron transfer between metal nanoparticles and TiO₂, affecting the depletion layer and facilitating greater resistance following gas exposure. Fig. 3 (f) presents the sensing performance of TiO₂ loaded with 0.5 mol% AgCu, which exhibited an excellent response of $R_{gas}/R_{air} \approx 33.2$ to 100 ppm xylene at 150°C.

5. Nanostructured TiO₂ decorated with metal oxides for BTX sensing

The decoration of metal oxide semiconductor materials with other metal oxides can be used to significantly enhance the gas-sensing performance via heterojunction effects, which influence the charge transfer dynamics, surface reactivity, and gas adsorption behavior [36]. When the two materials come into contact, they form a heterojunction, with the difference in their Fermi levels inducing charge transfer across the interface. This results in the formation of an electron depletion layer that sensitively modulates the electrical resistance following gas exposure. Tungsten oxides have been frequently used as decoration materials on nanostructured TiO₂ to enhance its sensitivity. One of the primary mechanisms contributing to this enhancement is the formation of n-n heterojunctions between tungsten trioxide (WO₃) and TiO₂, which facilitates charge

transfer, increases the electron depletion layer, and amplifies the response to gas molecules. For example, Chen et al. [37] reported WO₃-decorated TiO₂ nanoparticles with an exceptional xylene sensing performance, achieving a gas response ($R_{air}/R_{gas} \approx 92.53$) to 10 ppm xylene at 160°C that was considerably superior to that of pristine TiO₂ (Fig. 4 (a)). This improvement was attributed to the increased surface reactivity owing to the higher concentration of chemisorbed oxygen species and the introduction of oxygen vacancies. These vacancies acted as active adsorption sites, promoting gas molecule interactions and leading to an enhanced sensor response.

Further advancements in WO₃-decorated TiO₂ gas sensors have been demonstrated by Zhang et al. [38], who investigated the performance of WO₃ nanocube-decorated TiO₂ nanofibers synthesized by combining electrospinning and hydrothermal techniques (Fig. 4 (b)). The addition of monodispersed WO₃ nanocubes to the TiO₂ nanofibers resulted in a strong response ($R_{air}/R_{gas} \approx 52.4$) at 255°C. A WO₃ nanocube-based sensor had the lowest operating temperature with a response of 24.9, while the WO₃-TiO₂ heterostructural nanofibers (HNFs) achieved a significantly higher response of 52.4, surpassing both the TiO₂ nanofibers and WO₃ nanocubes alone (Fig. 4 (c)). However, the optimal operating temperature of WO₃-TiO₂ HNFs was slightly higher than that of pure WO₃, likely due to the additional energy required to overcome the interfacial

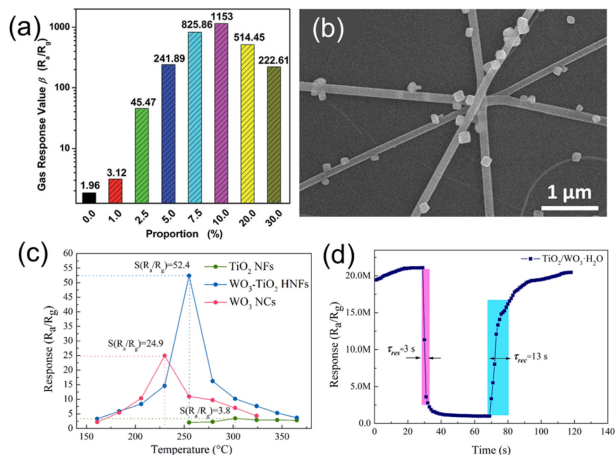


Fig. 4. Nanostructured TiO₂ decorated with metal oxides for BTX sensing. (a) Responses to 200 ppm xylene gas of sensors based on pure and WO₃-decorated TiO₂ nanoparticles at different operating temperatures and molar ratios of WO₃. Reprinted with permission from Ref. [37], Copyright (2016) ROYAL SOC CHEMISTRY. (b) Scanning electron microscope image of WO₃-TiO₂ HNFs. (c) Response of bare TiO₂ nanofibers, WO₃ nanocubes, and WO₃-TiO₂ HNFs to 100 ppm xylene. (d) Dynamic response–recovery times for a WO₃-TiO₂ HNFs-based sensor to 20 ppm of xylene. Reprinted with permission from Ref. [38], Copyright (2021) ELSEVIER.

potential barrier between TiO₂ and WO₃. Nevertheless, the WO₃-TiO₂ HNFs exhibited rapid response and recovery times following exposure to xylene. The higher oxygen vacancy den-

sity enhanced gas adsorption and accelerated charge transfer, while the formation of n-n heterojunctions between WO₃ and TiO₂ facilitated rapid electron movement, leading to a more rapid change in the resistance. The high surface area and catalytic activity of WO₃ also promoted efficient gas–surface interactions, enabling a more rapid response (3 s) and recovery (13 s) compared to pristine TiO₂ sensors (Fig. 4 (d)).

6. Other approaches to enhancing the BTX sensitivity of nanostructured TiO₂

One effective method to enhance BTX sensitivity is the thermal treatment of TiO₂ nanostructures, which modifies their crystal phase composition, defect density, and surface reactivity. In particular, high-energy {001} facets formed through annealing considerably improved the gas adsorption and charge transfer efficiency. Tshabalala et al. [39] reported that hierarchical TiO₂ spheres annealed at 700°C (T7) achieved an optimal anatase (80%)–rutile (20%) phase ratio, which enhanced charge transport and surface reactivity. Fig. 5 (a) shows the hierarchical TiO₂ structure formed via hydrothermal synthesis and thermal treatment, showing the higher surface area and porosity. The T7 sensor, which was annealed at 700°C, exhibited a high response of 13.6 to 100 ppm toluene at an operating temperature of 150°C, outperforming sensors annealed at lower temperatures (Fig. 5 (b)). These findings demonstrate that thermal treatment effectively enhances BTX

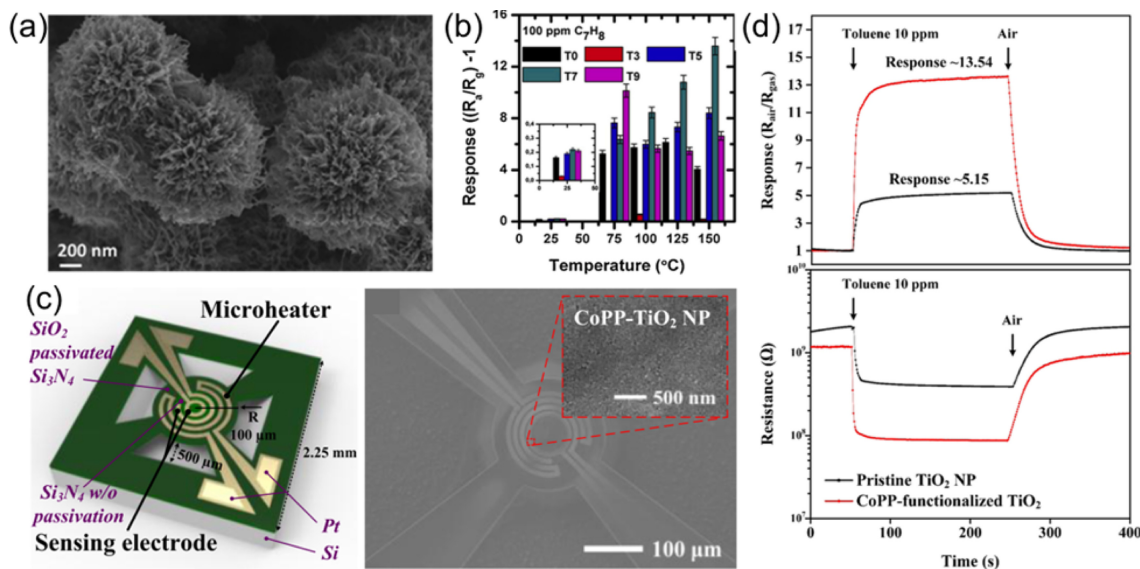


Fig. 5. Other approaches for enhancing the BTX sensitivity of nanostructured TiO₂. (a) Scanning electron microscope image of hierarchical TiO₂ spheres thermally treated at 700°C. (b) Response of thermally-treated TiO₂ to 100 ppm toluene at various operating temperatures. T3, T5, T7, and T9 correspond to a treatment temperature of 300, 500, 700, and 900°C, respectively, while T0 denotes that no thermal treatment was conducted. Reprinted with permission from Ref. [39], Copyright (2021) ELSEVIER SCIENCE SA. (c) Schematics of the proposed sensor platform and a scanning electron microscope image of the CoPP-functionalized TiO₂-integrated sensor platform. (d) Response of pristine and CoPP-functionalized TiO₂ nanoparticles to 10 ppm toluene. Reprinted with permission from Ref. [40], Copyright (2020) AMER CHEMICAL SOC.

sensing by exposing high-energy facets and increasing the oxygen vacancy density.

Another reported approach for BTX detection involves the use of metalloporphyrin as a functionalization material. Kang et al. [40] found that cobalt porphyrin (CoPP)-functionalized TiO₂ nanoparticles exhibited significantly enhanced sensitivity to BTX gas. Metalloporphyrins are organic macrocyclic compounds with a central metal ion surrounded by a nitrogen-coordinated porphyrin ring that strongly interact with aromatic volatile organic compounds (VOCs) through π - π stacking and hydrogen bonding. These interactions improve the gas adsorption efficiency and facilitate charge transfer, leading to a stronger sensing response. To fabricate the CoPP-functionalized TiO₂ sensor, TiO₂ nanoparticles were drop-coated onto a microheater-integrated sensing platform, followed by the deposition of a thin CoPP layer via e-beam evaporation. Fig. 5 (c) presents a schematic diagram of the sensor structure, with CoPP serving as a functionalization layer that enhances the gas-sensing capabilities of TiO₂. This functionalization significantly increased the response of the sensor, achieving a 245% enhancement in sensitivity to 10 ppm toluene compared to pristine TiO₂ nanoparticles (Fig. 5 (d)). The CoPP-TiO₂ sensor also demonstrated an ultrasensitive limit of detection (5 ppb for BTX), confirming its exceptional sensing capability for the detection of extremely low concentrations of BTX compounds.

7. Outlook and Conclusion

Recent advances in nanostructured TiO₂-based gas sensors have significantly enhanced the sensitivity and selectivity of BTX detection via material engineering strategies. The studies reviewed in this paper have demonstrated that metal doping, metallic and metal oxide decoration, thermal treatment, and organic functionalization are effective in enhancing the sensitivity of TiO₂-based BTX sensors. However, despite these advancements, several challenges remain, and further research is required to improve sensor performance for practical applications. The stability and reproducibility of sensing materials over extended periods remain a concern, particularly with regard to fluctuations in the humidity and temperature. Moreover, producing sensors with a high selectivity for individual BTX components without sensitivity to other VOCs remains a challenge. Finally, the integration of these sensing materials into miniaturized, low-power, and wireless sensor platforms for real-time monitoring is required for practical implementation. Future studies should focus on overcoming these challenges. Among them, the selectivity of the sensor to each BTX would be highly important as it has different effects to the human body. One approach to achieve selectivity is the com-

bination of pulsed heating [41] and machine learning-assisted gas classification [42]. Alternatively, the newly proposed combination of nanostructured TiO₂ with plasmonic materials, two-dimensional materials (e.g., MXenes and graphene), and bio-inspired sensing layers may provide new pathways for the commercialization of next-generation gas sensors with superior sensitivity, selectivity, and environmental robustness.

CRedit Authorship Contribution Statement

Kyubin Bae: Investigation, Methodology, Writing - original draft. **Eunhwan Jo:** Writing - review & editing, Supervision, Funding acquisition. **Yunsung Kang:** Writing - review & editing, Supervision, Funding acquisition.

Declaration of Competing Interest

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

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