

Article

An Ultrasensitive Ethanolamine Sensor Based on MoO₃/BiOI Heterostructure at Room Temperature

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Abstract

Ethanolamine (EA) is a widely used yet toxic volatile organic compound (VOC). However, existing gas sensors for EA detection face persistent challenges in achieving exceptional sensitivity and low detection limits at room temperature (RT). In this study, a novel and high-performance EA sensor based on the MoO₃/BiOI composite was prefabricated using hydrothermal and cyclic impregnation methods. The response value toward 100 ppm EA reached 861.3, which was 3.5-times higher compared to that of pure MoO₃. In addition, the MoO₃/BiOI composite exhibited a low detection limit (0.13 ppm), excellent selectivity, short response/recovery times, exceptional repeatability and long-term stability. The outstanding gas sensing performance of the MoO₃/BiOI is attributed to the formation of a p-n heterojunction, synergistic effects between the two materials, abundant adsorbed oxygen species and superior charge transfer efficiency. The sensor developed in this work effectively addresses the long-standing challenges, demonstrating unprecedented practical application potential for EA gas detection. Simultaneously, this study provides a novel strategy, a new approach and a promising material for the subsequent development of advanced amine sensors.

Keywords: EA; MoO₃/BiOI; p-n heterojunction; gas sensor; room temperature

1. Introduction

Air pollution relevant to toxic volatile organic compounds (VOCs) with the advancement of technology has intensified significantly and has emerged as one of the threats to human health [1,2]. Ethanolamine (EA) is a typical VOC and is widely used in pharmaceuticals, textiles, cosmetics and other industries [3–5]. However, prolonged exposure to a low-concentration EA environment can damage the nervous system and cause significant irritation to the eyes, skin and respiratory systems [5,6]. According to the National Occupational Health Standard of China and the American Conference of Government Industrial Hygienists, the short-term exposure limit (STEL) for EA is regulated at 15 mg/m³ (5.9 ppm). Therefore, the development of a low-detection-limit and high-sensitivity EA sensor at room temperature (RT) is essential.

Metal oxide semiconductor (MOS) gas sensors have attracted extensive attention due to their structural simplicity, high sensitivity, low cost and robust stability in the gas sensing field [7,8]. Molybdenum trioxide (MoO₃) is an n-type metal oxide semiconductor with a relatively wide band gap. The orthorhombic phase of MoO₃ (α-MoO₃) is popularly



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employed in electrochromic and photochromic devices, lithium-ion secondary batteries, selective catalytic reduction (SCR) systems and gas sensing applications due to its layered structure and superior thermodynamic stability [8,9]. Kwak et al. reported that α - MoO_3 nanobelts acquired using the hydrothermal method exhibited a response value of 2.9 toward 10 ppm NH_3 at 450 °C [8]. Huang et al. obtained MoO_3 nanosheets via the hydrothermal method, demonstrating a sensitivity of 280 toward 50 ppm triethylamine at 235 °C [10]. Li et al. prepared MoO_3 using a simple solvothermal approach with a response of 212 to 50 ppm n-pentylamine at 150 °C [11]. The high operating temperature and low sensitivity of the pure MoO_3 sensor limit its application.

Bismuth oxyhalides (BiOX , $\text{X}=\text{F}$, Cl , Br , and I) have attracted considerable research interest in recent years owing to their distinctive layered structure and the internal static electric field perpendicular to each layer, which contribute to their remarkable catalytic activity [12]. Among them, BiOI is a class of ternary oxide semiconductors (V-VI-VII) with an orthorhombic structure and intrinsic p-type doping [3,13]. It typically consists of $[\text{I-Bi-O-Bi-I}]$ slabs, and the superior optical and electronic characteristics of BiOI arise from the synergistic effect between intralayer covalent bonds and interlayer van der Waals interactions in its tetragonal matlockite structure [3,14]. Moreover, BiOI exhibits a distinctly narrower band gap and superior quantum efficiency compared to other bismuth compounds [15,16]. These character properties contribute to its promising potential for gas sensing applications [17]. It is well established that constructing p-n heterojunctions can enhance gas sensing performance. Meng et al. fabricated a 7%- $\text{NiMoO}_4/\text{MoO}_3$ p-n heterojunction with a response of 62.79 to 10 ppm triethylamine at 200 °C, about five-times that of the MoO_3 [18]. The response of ZnO/BiOI with p-n heterojunction prepared by Li et al. to 1 ppm NO_2 is 13.9, which is about 3.4-times higher than pure ZnO [19]. The MoO_3/BiOI composite with p-n heterojunction constructed from the MoO_3 and the BiOI demonstrates significant potential for enhancing gas sensing performance.

In this study, we synthesize MoO_3/BiOI material via hydrothermal and cyclic impregnation methods. The sensing sensitivity of the MoO_3/BiOI for EA gas is studied systematically at RT. The factors that boost the gas sensitivity performance of composites and the detailed mechanism of gas sensitivity response are also discussed.

2. Materials and Methods

2.1. Materials

Ammonium heptamolybdate tetrahydrate ($(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\cdot 4\text{H}_2\text{O}$) and ethylene glycol ($\text{C}_2\text{H}_6\text{O}_2$) were purchased from Shanghai McLean Biochemistry Co. Nitric acid (HNO_3 , 65–68%) was purchased from Liaoning Quanrui Reagent Co. Bismuth nitrate pentahydrate ($\text{Bi}(\text{NO}_3)_3\cdot 5\text{H}_2\text{O}$), potassium iodide (KI), and ethanol were purchased from Tianjin Komiou Chemical Reagent Co., Ltd (Tianjin, China).

2.2. Preparation of MoO_3 Nanoribbons and BiOI

First, 0.618 g $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\cdot 4\text{H}_2\text{O}$ was added to 50 mL deionized water and stirred for 20 min to obtain a uniform solution. Subsequently, 2.5 mL HNO_3 was added to the solution and stirred for 20 min. The mixed solution was transferred into a 50 mL autoclave and heated at 160 °C for 20 h. After the autoclave was reduced to RT, the product underwent multiple ethanol washes and dried at 70 °C for 10 h to acquire MoO_3 nanoribbons. Then, 6.5 g $\text{Bi}(\text{NO}_3)_3\cdot 5\text{H}_2\text{O}$ was added to 5 mL of ethylene glycol and stirred for 30 min. This was followed by the addition of 2.23 g KI , and the mixture was stirred for an hour. The orange precipitate was collected via centrifugation and dried under vacuum at 70 °C to obtain BiOI .

2.3. Preparation of MoO₃/BiOI Composite

Following this, 0.0607 g of Bi(NO₃)₃·5H₂O (Solution I) and 0.0208 g KI (Solution II) were sonicated in 50 mL ethylene glycol and anhydrous ethanol for 5 min, respectively. Then, 60 mg of MoO₃ nanoribbons was dispersed in Solution I and stirred for 15 min, followed by centrifugation to collect the precipitate. The precipitate was dispersed in Solution II under magnetic stirring for 15 min, followed by centrifugation to harvest the product. The above procedure constitutes one complete impregnation, which is cycled 15 times in this way. Finally, the product underwent multiple ethanol washes followed by drying at 70 °C, yielding the MoO₃/BiOI composite. The preparation process of MoO₃/BiOI composite is shown Figure 1.

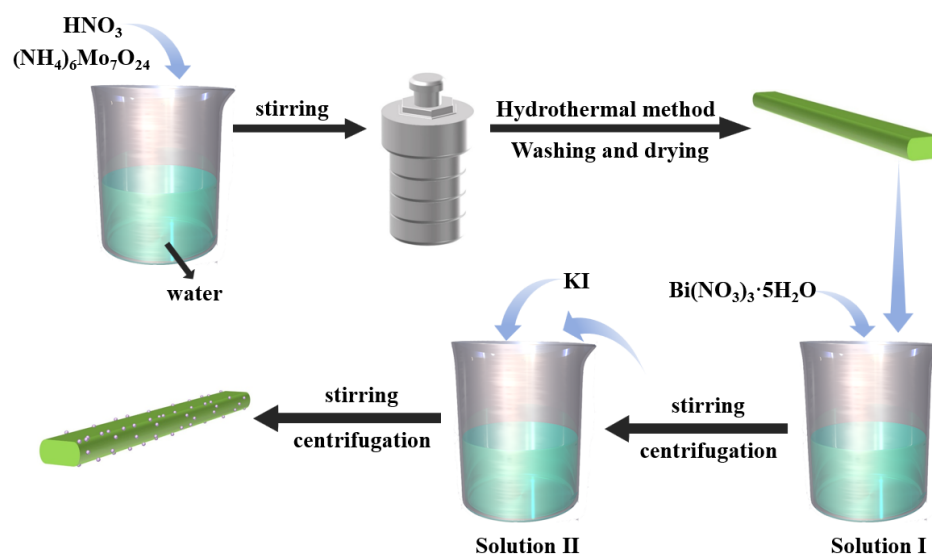


Figure 1. Diagrammatic drawing of the preparation step of MoO₃/BiOI.

2.4. Sensor Fabrication and Measurement

A small amount of the synthesized sample was ground and dispersed in anhydrous ethanol to form a uniform slurry, followed by being coated onto gold interdigitated electrodes (size: 10 × 8 × 0.15 mm). Subsequently, the coated electrodes were placed in an oven and dried at 70 °C for 12 h to ensure complete evaporation of ethanol. Gas sensing tests were conducted using a WS-30B gas detection and analysis system (Zhengzhou Winsen Electronics Technology Co., Ltd., Zhengzhou, China), with the experimental setup illustrated in Figure S1. The sensor performance was evaluated at room temperature (22 °C) and 25% relative humidity (RH). As-prepared sensing elements were connected in series with a reference resistor and placed in an 18 L test chamber. All standard gases used during the measurement were purchased from Harbin Liming Gases Co., Ltd. For NO, H₂S and CO, the desired test concentration is adjusted by using a micro syringe. The test environment is the air, and a part of NO may convert into NO₂. Thus, the actual gas is nitrogen oxides (NO_x). For ethanol, triethylamine, EA, ethylenediamine and ethylene glycol, the volume of liquid analyte calculated using Formula (1) [20] was injected onto the heating board fixed in the glass chamber via a micro syringe, evaporated immediately and mixed with air with the help of a fan. c (ppm) is the target gas concentration; φ , ρ (g/mL), V_1 (μL), and M (g/mol) are expressed as the purity, density, volume, and molecular weight of the liquid (%); and V_2 (L) is the volume of the chamber. The sensor response (R) is defined as $R = R_a/R_g$ for reducing gases or $R = R_g/R_a$ for oxidizing gases, where R_a and R_g represent the electrical

resistance of the sensor in air and target gas, respectively. The response/recovery time is defined as the time required to reach 90% of the total resistance change.

$$c(\text{ppm}) = \frac{22.4 \times \varphi \times \rho \times V_1 \times 1000}{M \times V_2} \quad (1)$$

3. Results and Discussion

3.1. Structural and Morphological Characteristics

The crystal structures of MoO_3 and MoO_3/BiOI are characterized by X-ray diffraction (XRD), and the outcomes are shown in Figure 2. The diffraction peaks at 2θ values of 12.75° , 23.31° , 25.70° , 27.31° , 38.98° and 58.84° correspond to the (0 2 0), (1 1 0), (0 4 0), (0 2 1), (0 6 0) and (0 8 1) crystal planes of orthorhombic MoO_3 (JCPDS NO. 35-0609), respectively. All characteristic peaks of pure MoO_3 are observed in the XRD pattern of the composite. Additionally, a weak peak observed at 31.66° is well attributed to the (1 1 0) crystal plane of BiOI (JCPDS NO. 10-0445). A magnified view of the dashed-box region (in Figure 2I) is presented in Figure 2II for detailed observation. Figure S2 shows the XRD patterns of the samples with 17 cycles (MoO_3/BiOI -17) and the MoO_3/BiOI -15. As shown in Figure S2II, the peak intensity at 31.66° is significantly enhanced when the number of cycles increases (higher loading level). Moreover, a sharp little peak corresponding to the (1 1 4) plane of BiOI at 51.35° is found in the MoO_3/BiOI -17 sample but absent in the MoO_3/BiOI -15 sample (Figure S2III). The absence of distinct BiOI diffraction peaks in the MoO_3/BiOI -15 can be attributed to the low BiOI content in the composite [21] or peak overlap with MoO_3 reflections [22]. No detectable impurity peaks in the XRD pattern confirm the successful fabrication of the MoO_3/BiOI composite. The elemental composition and content of the MoO_3/BiOI composite are detected using energy dispersive spectroscopy (EDS), as shown in Figure S3. The EDS outcomes confirm that the composite consists of Mo, O, Bi and I elements with an atomic ratio of 10.78:88.04:0.61:0.57, and no impurity elements are detected. The successful synthesis of BiOI in the composite is evidenced by the Bi/I atomic ratio of approximately 1:1. These results demonstrate the high purity of the sample and the low BiOI content in the composite.

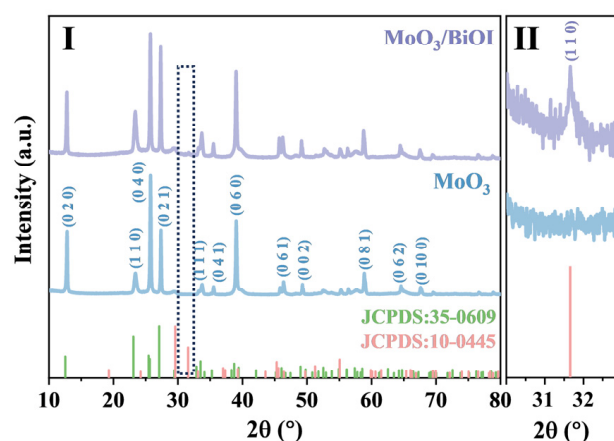


Figure 2. (I) Complete and (II) locally magnified XRD of MoO_3 and MoO_3/BiOI .

The N_2 adsorption/desorption isotherms of MoO_3 and MoO_3/BiOI are shown in Figure 3. Both MoO_3 and MoO_3/BiOI exhibit Type IV isotherms with H3-type hysteresis loops, which confirm mesoporous materials [9]. According to the Brunauer–Emmett–Teller calculation method, the specific surface areas of MoO_3 and MoO_3/BiOI are $8.41 \text{ m}^2 \cdot \text{g}^{-1}$ and $9.43 \text{ m}^2 \cdot \text{g}^{-1}$, respectively. The pore size distribution curves of MoO_3 and MoO_3/BiOI are calculated via the Barrett–Joyner–Halenda method and displayed in the insets of

Figure 3a,b, with average pore diameters determined as 3.43 nm for MoO₃ and 3.12 nm for the composite. This further confirms that both synthesized MoO₃ and MoO₃/BiOI are mesoporous materials. The minimal loading level of BiOI does not result in significant alterations to the microscopic morphology and the specific surface areas. The two samples exhibit only slight differences in specific surface area and pore diameters, indicating that the incorporation of BiOI has minimal influence on the mesostructure of the material.

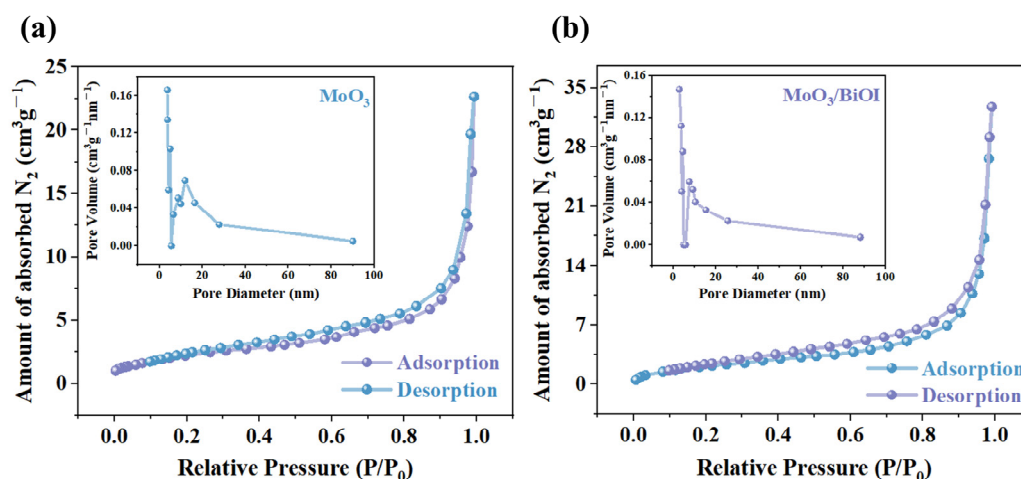


Figure 3. N₂ adsorption–desorption isotherms and pore size distribution of (a) MoO₃ and (b) MoO₃/BiOI.

The morphology and microstructure of the samples are researched using scanning electron microscopy (SEM) and transmission electron microscopy (TEM). Figure 4a and 4d show the SEM images of pure MoO₃ and the MoO₃/BiOI composite, respectively. It can be observed that pure MoO₃ exhibits a nanoribbon structure with smooth surfaces and uniform thickness. The BiOI-modified MoO₃ nanoribbons exhibit surface roughening while preserving the intrinsic morphology and dimensional integrity of pristine MoO₃. The localized nanoribbon TEM images of MoO₃ and the MoO₃/BiOI are displayed in Figure 4b,e. It can be seen that the introduction of BiOI nanoparticles makes the surface of the composite rough without changing its structure, which is in agreement with SEM. The HRTEM image of pure MoO₃ in Figure 4c reveals lattice spacings of 0.195 nm and 0.230 nm, corresponding to the (0 6 1) and (0 6 0) planes of MoO₃, respectively. The lattice spacings of 0.230 nm and 0.301 nm are observed in the HRTEM image of the composite material in Figure 4f, which are assigned to the (0 6 0) plane of MoO₃ and the (1 0 2) plane of BiOI, respectively. Meanwhile, a lattice contact is apparent at the interface between MoO₃ and BiOI, indicating the formation of the MoO₃/BiOI p-n heterojunction (red circle) [23]. To further confirm the successful construction of the heterojunction, Mott–Schottky (M–S) electrochemical analysis is conducted to investigate the conductive behavior of the material. The slopes in M–S plots serve as indicators to distinguish between n-type (positive slope) and p-type (negative slope) semiconductors [23]. The positive slope observed in the M–S plot of MoO₃ (Figure S4) confirms its n-type semiconductor behavior. In contrast, the M–S plot of BiOI displays a negative slope, indicating a p-type semiconductor. The M–S plot of the composite displays a characteristic inverted “V” shape, signifying p-n heterojunction formation at the interface of MoO₃ and BiOI [24]. This is consistent with the HRTEM observation results. The distribution of elements in the MoO₃/BiOI is illustrated in Figure 4g–j. The uniform distribution of Mo, O, Bi and I elements in the composite further proves the successful preparation of MoO₃/BiOI.

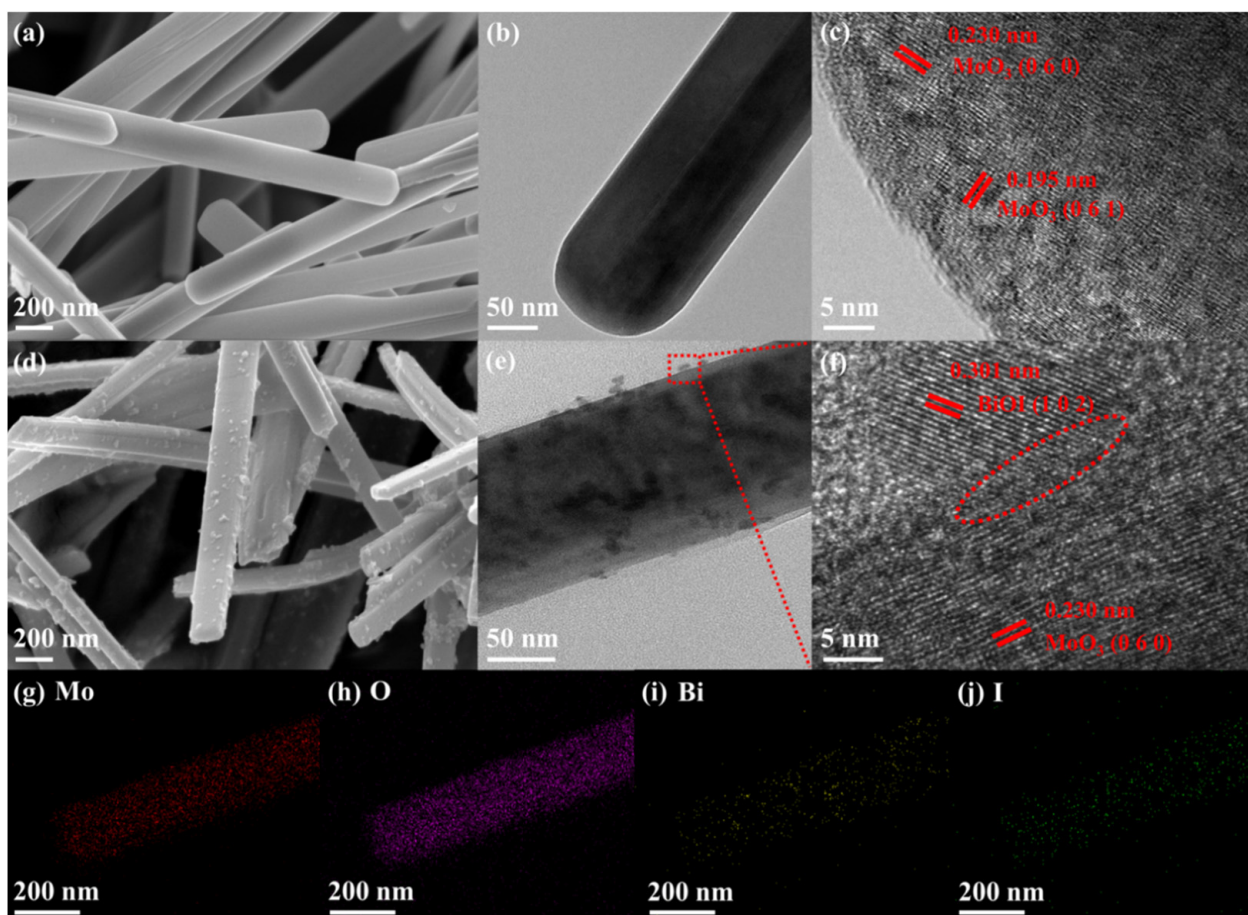


Figure 4. SEM image of MoO_3 (a) and MoO_3/BiOI (d); TEM image of MoO_3 (b) and MoO_3/BiOI (e); HRTEM image of MoO_3 (c) and MoO_3/BiOI (f); elemental mapping of Mo, O, Bi and I in MoO_3/BiOI (g–j).

The elemental composition and chemical valence states of the materials can be investigated using X-ray photoelectron spectroscopy (XPS), and the outcomes are shown in Figure 5. The high-resolution Mo 3d spectra of MoO_3 and MoO_3/BiOI in Figure 5a exhibit peaks of binding energy located at ~ 236.1 eV and ~ 233.0 eV, corresponding to the Mo 3d_{3/2} and Mo 3d_{5/2} of Mo^{6+} , respectively [25]. The peaks observed at ~ 164.05 eV and ~ 158.85 eV in the Bi 4f spectrum of Figure 5b belong to the Bi 4f_{5/2} and Bi 4f_{7/2}, respectively. This indicates that the Bi element in both samples mainly exists as Bi^{3+} [26]. The binding energies of I 3d at ~ 630.2 eV and ~ 618.79 eV in Figure 5c are ascribed to I 3d_{3/2} and I 3d_{5/2}, meaning that I exists in the form of I^- [27]. The peaks of Mo 3d in the composite shift toward lower binding energies compared to the pristine phase, while the peaks of Bi 4f and I 3d move in the opposite direction, demonstrating strong chemical interactions between MoO_3 and BiOI [28]. The O 1s spectra of MoO_3 and MoO_3/BiOI in Figure 5d are further fitted into three peaks located at ~ 532.9 , ~ 531.4 and ~ 530.4 eV, which correspond to chemisorbed oxygen (O_c), vacancy oxygen (O_v) and lattice oxygen (O_L) [29]. The contents of various oxygen species in the two samples are summarized in Table 1. Notably, the MoO_3/BiOI composite exhibits a marked enhancement in the relative concentrations of O_c and O_v compared to pure MoO_3 , suggesting its superior gas sensing performance [22,30]. Furthermore, electronic paramagnetic resonance (EPR) is performed to analyze the O_c of the sample, and the results are presented in Figure S5. The EPR symmetric signal peak at $g = 2.000$ [31] is attributed to the unpaired electrons in the oxygen vacancy, and the peak strength is related to the content of oxygen vacancy [32]. The peak intensity of the

MoO₃/BiOI complex is significantly higher compared to pure MoO₃, indicating a higher content of O_c in the composite material. This agrees with the XPS results.

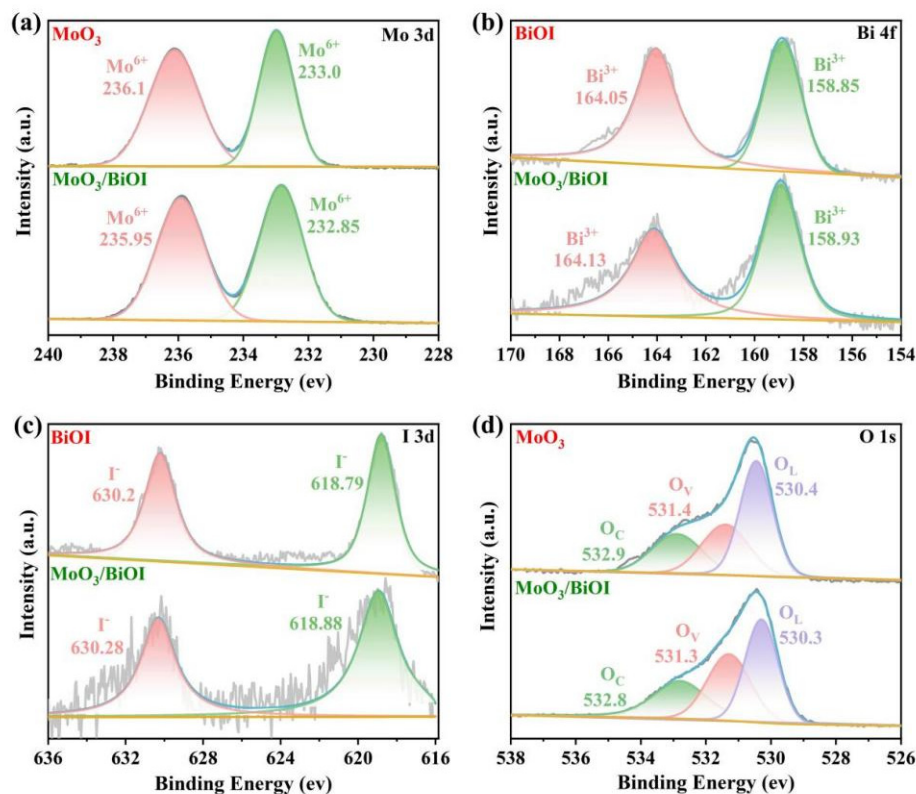


Figure 5. XPS spectra of MoO₃ and MoO₃/BiOI (a) Mo 3d; (b) Bi 4f; (c) I 3d and (d) O 1s.

Table 1. The contents of oxygen species of MoO₃ and MoO₃/BiOI.

Materials	O _c (%)	O _v (%)	O _L (%)
MoO ₃	47.5	28.6	23.9
MoO ₃ /BiOI	37.1	36.7	26.2

3.2. Gas Sensing Performance

The gas sensing response of pure MoO₃, pure BiOI and MoO₃/BiOI composites (13-MoO₃/BiOI, 15-MoO₃/BiOI, and 17-MoO₃/BiOI) with three different loading levels is illustrated in Figure S6, and the composite obtained via 15 cyclic impregnations demonstrates the superior response value among them. Consequently, this composite and pure MoO₃ is selected for more detailed gas sensing investigations. Figure 6a,b depict the response/recovery plots of MoO₃ and MoO₃/BiOI toward various EA concentrations at RT. The response values of MoO₃/BiOI are 861.3–1.6 in the concentration range of 100 to 0.5 ppm EA, while the response values of MoO₃ toward 100–1 ppm EA are only 246.8–1.3. The response value of MoO₃/BiOI toward 100 ppm EA is 3.5-times higher compared to pure MoO₃. Furthermore, the response/recovery times of MoO₃ and MoO₃/BiOI toward 100 ppm EA are 93/368 s and 56/349 s in Figure 6c and 6d, respectively. The results confirm that MoO₃/BiOI exhibits higher sensitivity and faster response/recovery times than pure MoO₃.

The functional relationships between the response values of the two sensors and the EA concentration are shown in Figure 7a,b. The fitting equations for MoO₃ and MoO₃/BiOI are $y = 0.0192x^2 + 0.5185x + 3.6289$ ($R^2 = 0.9986$) and $y = 0.0697x^2 + 1.6502x + 3.9136$ ($R^2 = 0.9992$), respectively. The high R^2 values indicate an intense functional correlation between the response values and the EA concentration. To calculate the detection limit (DL) of the samples, the linear fitting relationships between the response values and the low EA concentration are shown in the insets of Figure 7a,b, and the raw baseline noise trace is shown in Figure S7. The theoretical detection limits of MoO₃ and MoO₃/BiOI for EA are 0.42 ppm and 0.13 ppm based on empirical Formulas (2) and (3), respectively. The fitting equations and calculation results demonstrate that the composite material is more suitable for trace EA gas detection than pure MoO₃.

$$\text{RMS}_{\text{noise}} = \sqrt{\frac{\sum(\bar{y} - y_i)^2}{n - 1}} \quad (2)$$

$$\text{DL} = 3 \frac{\text{RMS}_{\text{noise}}}{k} \quad (3)$$

In the two equations, k is the slope of the linear fit at low gas concentrations, $\text{RMS}_{\text{noise}}$ represents the root mean square noise, $\bar{y}$ denotes the average value of the baseline response, and y_i corresponds to the baseline surveyed value.

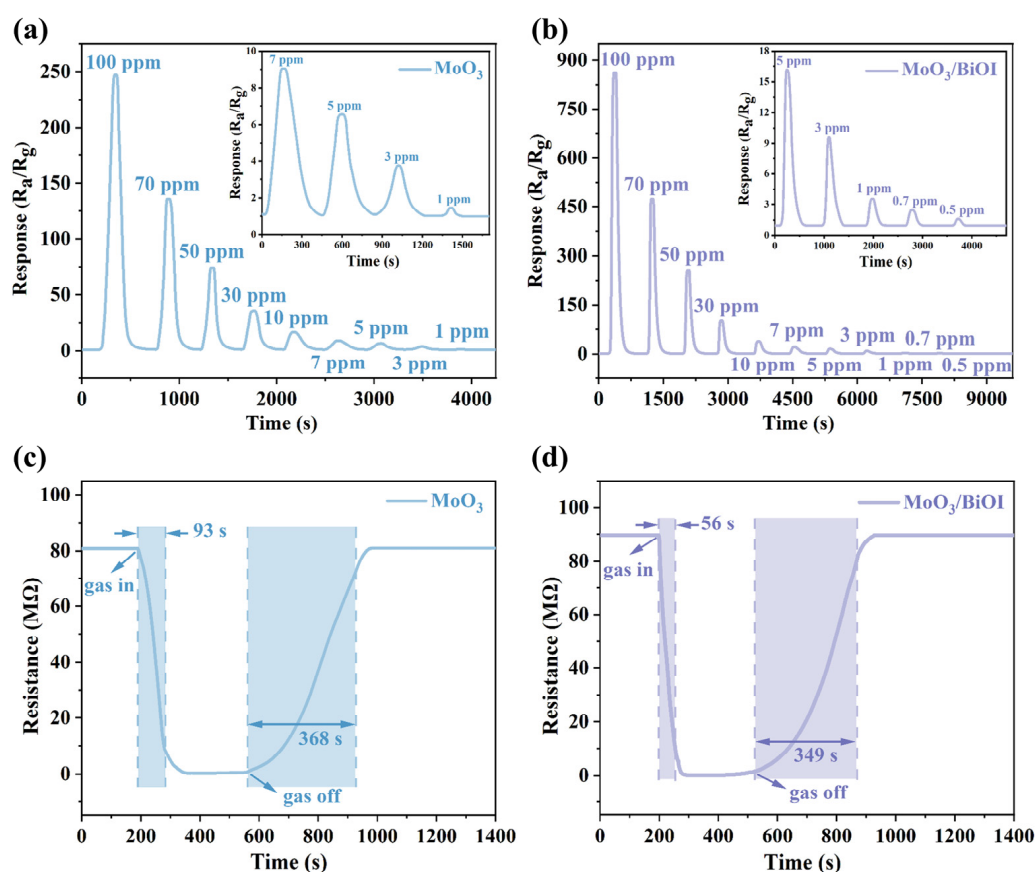


Figure 6. Dynamic response recovery plots to EA of (a) MoO₃ and (b) MoO₃/BiOI; response/recovery time to EA of (c) MoO₃ and (d) MoO₃/BiOI.

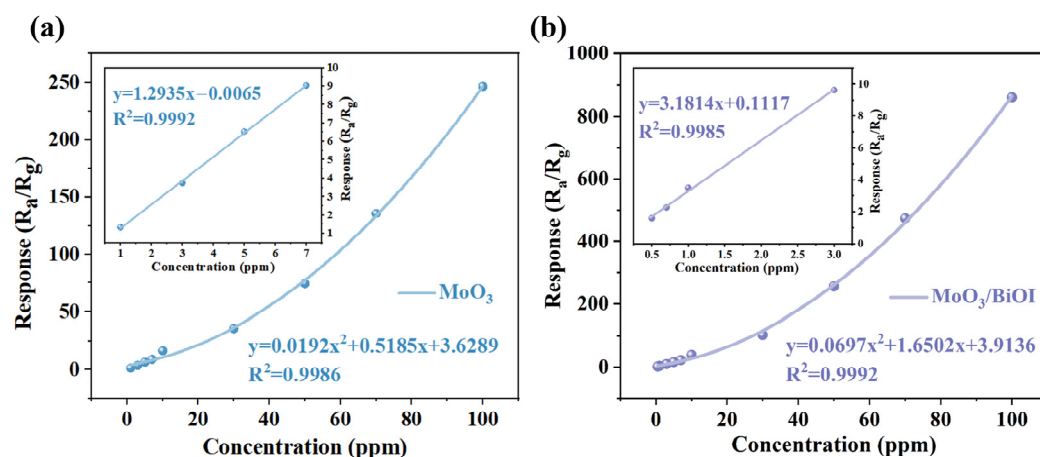


Figure 7. Linear fitting of samples to various concentrations of EA and response values of (a) MoO₃ and (b) MoO₃/BiOI.

The repeatability and long-term stability serve as vital criteria for performance evaluation in practical applications. The five-cycle test curves of MoO₃ and MoO₃/BiOI composites toward 100 ppm EA are shown in Figure 8a. The resistance of samples declines when exposed to the EA atmosphere and recovers to their primal value when returned to air, confirming their typical n-type semiconductor behavior. The sensor response amplitudes vary slightly across five cycles, demonstrating remarkable repeatability in gas detection applications. Otherwise, the response values of sensors fluctuated within 5% during a 60-day testing period in Figure 8b, indicating excellent long-term reliability. The inset of Figure 8b presents the response curves of the MoO₃/BiOI composites measured at intervals of 10, 30, and 50 days. The response/recovery times of MoO₃/BiOI remain relatively stable throughout the testing period. Figure S8 displays the gas sensing responses of different batches of MoO₃/BiOI samples toward 100 ppm EA under identical testing conditions. The standard error of the response values for the five groups is only 3%, demonstrating the excellent reproducibility of the MoO₃/BiOI composite synthesized in this work.

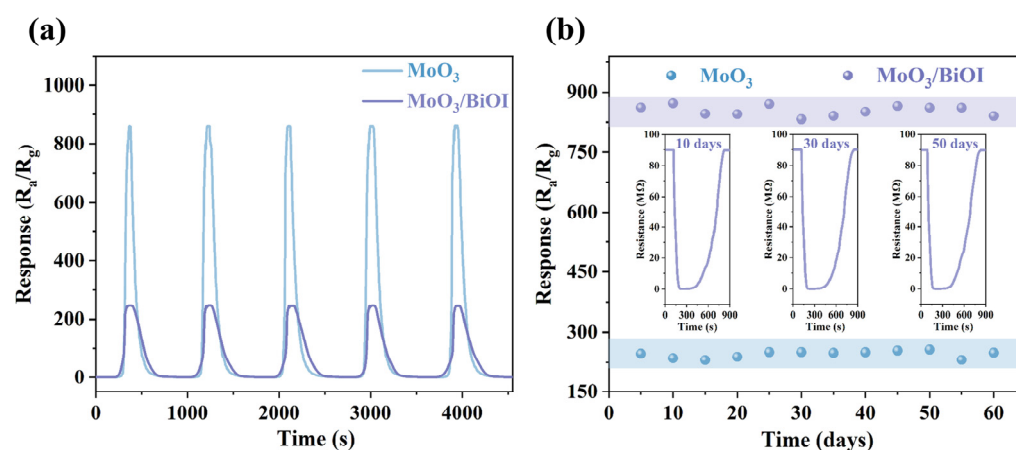


Figure 8. (a) Cyclic curves of MoO₃ and MoO₃/BiOI; (b) long-term stability of MoO₃ and MoO₃/BiOI.

The selectivity is another significant factor in assessing the performance of sensors. To research the selectivity of MoO₃ and MoO₃/BiOI, their response values toward 100 ppm of various gases (NO_x, H₂S, CO, ethanol, triethylamine, EA, ethylenediamine and ethylene glycol) are measured and compared at RT, as shown in Figure 9a. The sensitivity of MoO₃ and MoO₃/BiOI to EA is significantly higher than those of other gases under identical testing conditions. The results indicate that MoO₃ and MoO₃/BiOI exhibit excel-

lent selectivity toward EA. It is well known that selectivity is influenced by a multitude of factors. Firstly, there exists a strong acid/base attractive force between MoO_3 and EA molecules [33]. As a relatively strong organic Lewis base, EA exhibits high reactivity with acidic MoO_3 over other VOCs. Secondly, bond energy significantly influences the stability of compounds. The lower bond energy corresponds to the bond that is more easily broken [34]. The relevant bond energies (C-H: 411 kJ/mol, C-O: 361 kJ/mol, O-H: 458.8 kJ/mol, C-N: 307 kJ/mol) [35] reveal that the C-N bond of EA is the easiest to break. EA molecules have asymmetrical structures and stronger reducing chemical reactivity compared to other volatile gases [35,36]. Finally, the abundance of hydrogen bonds within the EA molecule promotes its adsorption on the surface of the MoO_3/BiOI nanostructure, thereby enabling more efficient interaction with adsorbed oxygen species [5,37]. The influence of relative humidity (RH) on sensor performance is depicted in Figure 9b. The response values of both MoO_3 and MoO_3/BiOI composites to 100 ppm EA gradually decline as RH increases from 22% to 75%. Notably, the MoO_3/BiOI composite consistently demonstrates superior response values compared to pure MoO_3 across this humidity range. The MoO_3/BiOI composite achieves a sensing response of 396 toward 100 ppm EA at 75% RH, which represents a 4.4-times improvement over pure MoO_3 . This pronounced performance shows the feasibility of the MoO_3/BiOI composite as a sensing material for the detection of EA. The decrease in sensor response under high RH can be attributed to the following factors. Firstly, water molecules are adsorbed onto the surface of the sample, and the active sites become occupied, resulting in the chemisorption of oxygen species becoming more difficult [38]. Secondly, the adsorbed water molecules react with chemisorbed oxygen to generate surface hydroxyl groups (OH^-) [39]. A reduced concentration of adsorbed oxygen ions diminishes the thermodynamic driving force required for subsequent reactions. This decreased driving force further hinders both the adsorption and dissociation processes of EA molecules [40].

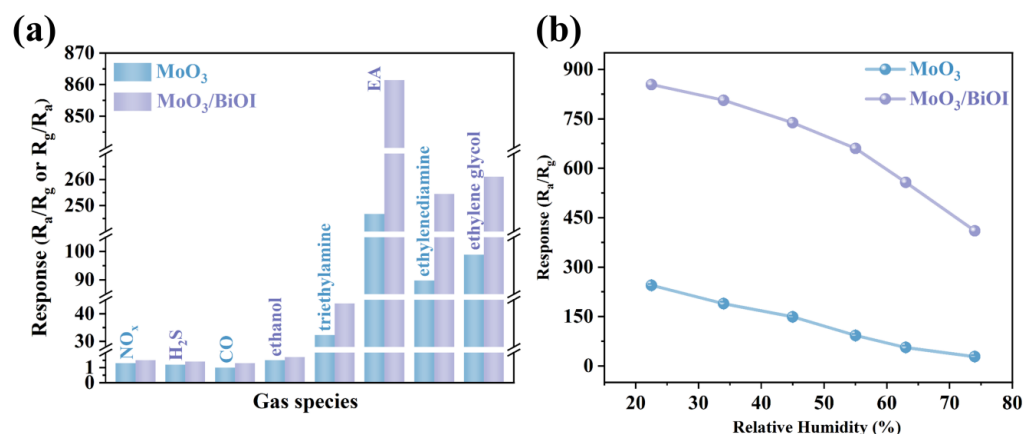


Figure 9. (a) Response values of MoO_3 and MoO_3/BiOI to different gases; (b) response values of MoO_3 and MoO_3/BiOI to EA under different RH.

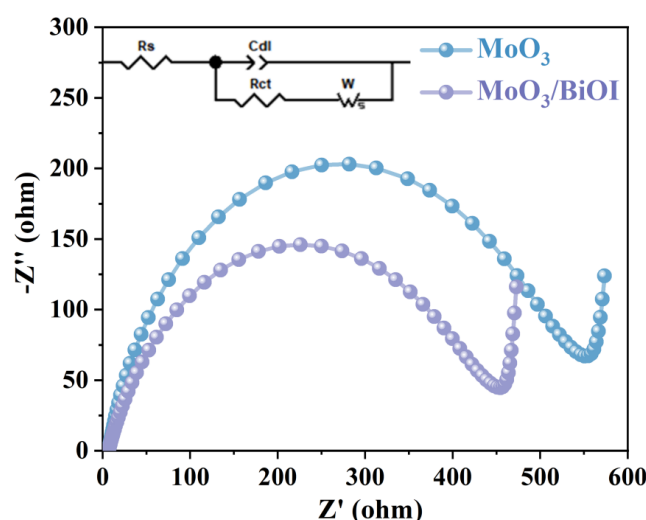
A comparison of the sensor developed in this study with other sensors reported in the literature is summarized in Table 2. It can be concluded that the MoO_3/BiOI composite exhibits a high response to EA, operating conditions of RT and a low detection limit, which indicates that MoO_3/BiOI holds significant potential for practical applications in EA gas detection.

Table 2. Comparison of the EA-sensing properties of the MoO₃/BiOI in this work and the published literature.

Materials	Target Gas	Operating Temperature (°C)	Gas Concentration (ppm)	Response	Lod (ppm)	Refs.
sulfur-doped ZnO	EA	240	100	83	0.089	[2]
2%Au/ZnO	EA	240	300	97.656	0.2	[41]
Ni-LaFeO ₃	EA	260	100	8.3	2.25	[42]
CsPbBr ₃ -SnWO ₄	EA	RT	200	45.16	2	[43]
g-C ₃ N ₄ /SnO ₂	EA	240	100	68	0.294	[36]
KGM-ZnO	EA	300	100	313.49	1	[44]
Pd-SnO ₂	EA	150	100	106	1	[6]
ZnO/In ₂ O ₃	EA	240	50	237.761	1	[45]
MoO ₃ /BiOI	EA	RT	100	861.3	0.13	This work

3.3. Gas Sensing Mechanism

The markedly improved gas sensing performance of the MoO₃/BiOI composite compared to pure MoO₃ arises from the following synergistic mechanisms. Firstly, the MoO₃/BiOI material has more O_c and O_v than pure MoO₃, providing more active sites. These active sites further promote the redox reactions with EA molecules, thereby improving the gas sensing performance [23]. Secondly, the construction of the p-n heterojunctions in the MoO₃/BiOI composite can significantly improve the sensing performance [46]. Lastly, the higher charge transfer efficiency can also enhance gas sensitivity [47]. Nyquist plots of the Electrochemical Impedance Spectroscopy (EIS) data for the pure MoO₃ and MoO₃/BiOI are shown in Figure 10. The corresponding equivalent circuit is shown in the inset of Figure 10. In this circuit, R_{ct} denotes the interfacial charge transfer resistance, R_s represents solution resistance, W refers to Warburg impedance and C_{dl} is the electric double-layer capacitance, listed in Table S1. The composite material exhibits a smaller R_{ct} than pure MoO₃, which partially supports that MoO₃/BiOI possesses a relatively higher charge transfer efficiency. This accelerates the interaction between target gas molecules and adsorbed oxygen species to some extent, which improves the gas sensing performance of the MoO₃/BiOI [48].

**Figure 10.** The Nyquist plots of MoO₃ and MoO₃/BiOI.

The sensing process of MOS originates from the chemical adsorption of oxygen molecules on the material surface and electron transfer between oxygen anions and the tar-

get gas, which alters the carrier concentration and ultimately results in resistance variations. Based on the band structure parameters of MoO₃ and BiOI [49–51], Figure 11 illustrates the gas sensing mechanism and corresponding band structure of the MoO₃/BiOI. The p-n heterojunction forms when contact occurs between n-type and p-type semiconductors due to the difference in work functions and Fermi-level alignment. The difference in work function between MoO₃ (5.18 eV) and BiOI (6.74 eV) drives electron transfer from the higher Fermi level of MoO₃ to the lower Fermi level of BiOI, while holes migrate in the opposite direction, resulting in an equilibrium of the Fermi level and band bending [52,53]. At the same time, a depletion layer is formed at the interface between MoO₃ and BiOI [54]. When MoO₃/BiOI is exposed to air, oxygen molecules (O_{2(gas)}) in the atmosphere adsorb onto the material surface to convert to adsorbed oxygen molecules (O_{2(ads)}). The adsorbed oxygen molecules capture free electrons from the conduction band of the sample to form oxygen anions (O₂[−]), as illustrated in Equations (4) and (5). This process reduces the carrier concentration and extends the depletion layer width, consequently causing a high primal resistance characteristic for the gas sensor in the air. A higher initial resistance can cause a higher response value when detecting reducing gases [55]. Upon exposure to EA gas, the adsorbed EA molecules (EA_(gas)) undergo redox reactions with O₂[−], releasing trapped electrons back into the conduction band. This process increases carrier concentration and reduces depletion layer thickness, as described by Equations (6) and (7) [42,56]. In the meantime, the resistance of the sensor decreases, and the response value increases rapidly. When the material is re-exposed to air, the adsorbed EA species are desorbed and the oxygen molecules are re-adsorbed on the surface of the material, which causes the depletion layer to be reconstructed and the resistance to return to its initial baseline value.

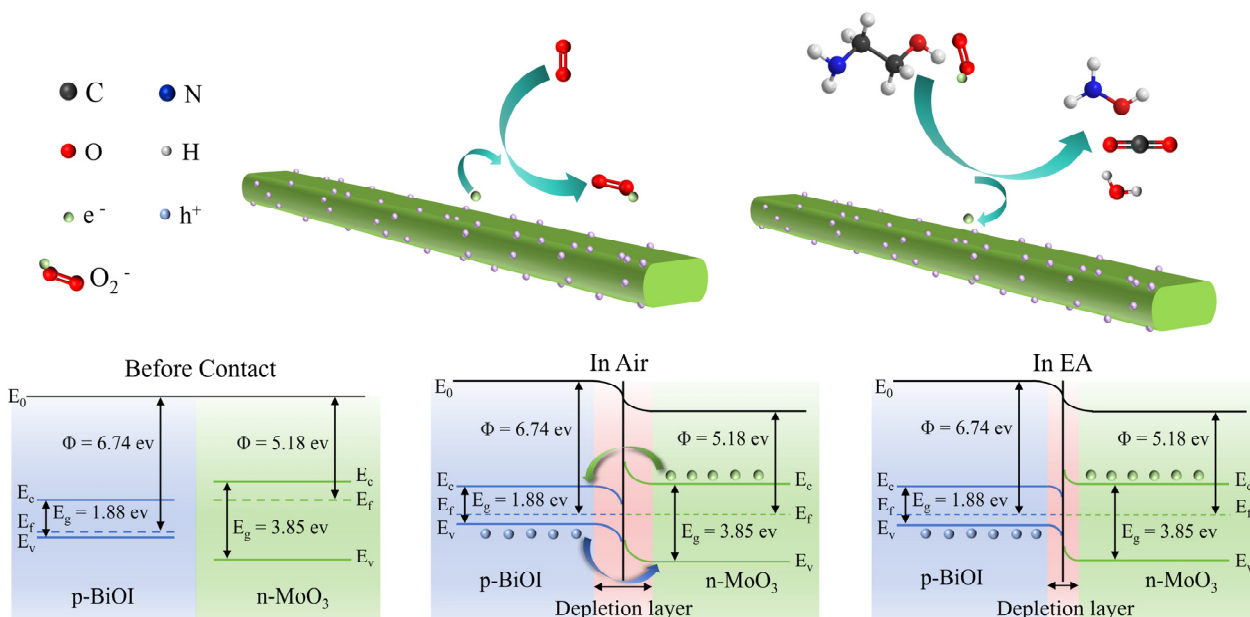
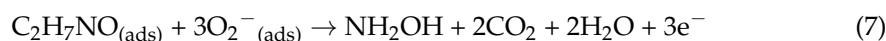
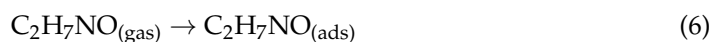


Figure 11. Schematic representation for simulated sensing mechanism and energy bands of the MoO₃/BiOI composite before and after exposure to EA gas.

4. Conclusions

In summary, a high-performance EA sensor based on MoO₃/BiOI composites was prefabricated for the first time using hydrothermal and cyclic impregnation methods at RT. The sensor exhibits exceptional sensitivity (861.3), rapid response/recovery time (56/349 s), a low detection limit (0.13 ppm) and outstanding repeatability and long-term stability toward EA. The composite possesses enhanced gas sensing performance compared to pure MoO₃ due to the higher charge transfer efficiency, more O_v and O_c and the established p-n heterojunction. Moreover, the gas sensing mechanism is systematically explained using the oxygen adsorption model. The outcomes demonstrate that the synthesized MoO₃/BiOI composite holds promise as a prospective gas sensor for EA detection and provides new insights for developing novel gas sensing materials.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/chemosensors14010028/s1>, Figure S1: (a) Ws-30B gas detection and analysis instrument; (b) Weisheng 30 Road test board; (c) schematic diagram of resistance measurement circuit; (d) interfingered electrode; Figure S2: XRD of MoO₃/BiOI-15 and MoO₃/BiOI-17; Figure S3: EDS spectrum of MoO₃/BiOI; Figure S4: Mott–Schottky plots of MoO₃, BiOI and MoO₃/BiOI; Figure S5: EPR spectra of MoO₃ and MoO₃/BiOI; Figure S6: Response values of MoO₃, BiOI and three MoO₃/BiOI to EA of 100 ppm and 50 ppm; Figure S7: The raw baseline noise trace of MoO₃ and MoO₃/BiOI; Figure S8: The reproducibility of the MoO₃/BiOI sensor to EA; Table S1: Fitted impedance parameters of MoO₃ and MoO₃/BiOI.

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