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One-step synthesis of n-CdO/n-SnO₂ heterojunction nanofibers for high-performance 3-hydroxy-2-butanone gas sensing

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Abstract

Listeria monocytogenes (LM) is a pathogenic bacterium which can release 3-hydroxy-2-butanone (3H-2B) as a biological indicator. We report a high-performance 3H-2B gas sensing strategy for the selective detection of LM. This strategy is realized by n-CdO/n-SnO₂ hetero-nanofibers with controllable compositions, synthesized via a facile one-step electrospinning method. The tailored morphologies and microstructures of CdO/SnO₂ nanofibers were systematically characterized by scanning electron microscopy (SEM) and transmission electron microscopy (TEM). With the introduction of CdO into SnO₂ nanofibers, x-ray diffraction (XRD) and x-ray photoelectron spectroscopy (XPS) were performed to investigate the effects of crystal phases and elemental states on the 3H-2B sensing properties. According to the gas sensing results, the variation of Cd/Sn molar ratios has a great influence on the 3H-2B sensing properties of CdO/SnO₂ nanofibers. The maximum response (45) to 5 ppm 3H-2B is found for 5 mol% CdO/SnO₂ nanofibers at 260 °C. Meanwhile, 5 mol% CdO/SnO₂ nanofibers exhibit a short response/recovery time (9 s/5 s), outstanding stability, and discriminative selectivity to 3H-2B. The enhanced sensing performance is mainly attributed to the synergy between the resistance modulation of n-CdO/n-SnO₂ heterojunctions and the doping effect of Cd²⁺ ions.

1. Introduction

Listeria monocytogenes (LM), a widespread pathogenic bacterium in frozen foods such as meats, vegetables, and milk, can cause dangerous diseases to threaten human health [1, 2]. This pathogen can live in various environmental conditions regardless of the moisture and pH degree [3, 4]. The common biological technology and immunoassay for detecting LM need expensive apparatuses and much time. Therefore, it is important and urgent to develop a simple point-of-care test for LM [5–10]. As one of the signature metabolites exhaled by LM, 3-hydroxy-2-butanone (3H-2B) has been recognized as a biological indicator of LM, for the good linear relationship between the level of 3H-2B and the growth time of LM [11, 12]. Nowadays, the detection of the signature metabolites such as the biological 3H-2B indicator of foodborne LM pathogens, has been applied as an effective approach to noninvasively monitor the microbial activity [13–17]. In this area, metal oxide based chemiresistive gas sensors have unique advantages such as the low-cost, easy operation and sensitive response-recovery behavior, making it possible to realize the indirect detection of relevant microbial growth by measuring the level of metabolite indicators [18].

Gas sensors based on metal oxide semiconductors have been explored as potential means for the sensitive inspection of 3H-2B [19, 20]. Deng et al prepared a 3H-2B sensor based on WO₃/Au nanocomposites, which exhibits high sensitivity and selectivity [21]. Owing to the sensitive response and good chemical stability, the typical n-type SnO₂ semiconductor has been utilized to build gas sensors for sensing the 3H-2B indicator [22, 23]. For example, Zhu et al synthesized Pt-doped SnO₂ nanospheres for the selective and rapid sensing of 3H-2B [24]. To improve the gas sensing performance of SnO₂, one of the current trends is constructing SnO₂...
into nanostructures with a considerable surface area and abundant active sites [25, 26], Zhang et al. found that the interfacial oxygen sites contribute to the 3H-2B sensing performance of porous CeO$_2$/SnO$_2$ nanosheets [27]. In particular, one-dimensional (1D) nanostructures have attracted considerable attention due to their fast transport for response signals [28, 29]. Yang et al. reported ZnO@Al$_2$O$_3$ nanocables with a sensitive response and low detection limit to 3H–2B [30]. Another effective strategy to improve the sensitivity and selectivity of semiconductors is doping with secondary metal oxides, which involves two possible factors: the vacancy defects induced by metal ion doping to create more chemisorbed oxygen and the heterojunctions to modulate the electron depletion layer [31, 32]. Zeb et al. fabricated a gas sensor based on Cu-doped Au/PdO/K$_2$WO$_4$O$_{13}$ nanowires, which can selectively detect 3H–2B at a low temperature [33]. Compared with other routes to obtain 1D hetero-nanomaterials, such as the hydrothermal method or electrospinning with post-decoration [34, 35], electrospinning using one-step synthesized precursors can realize the in situ preparation of hetero-nanofibers with tunable compositions and modulated structures [36–39]. Although progress has been achieved based on the above strategies, the sensitivity and selectivity of metal oxide gas sensors to 3H–2B still needs to be improved further. If a 1D nanofiber structure is constructed with two metal oxides such as SnO$_2$ and CdO, the 3H–2B sensing performance might be synergistically by the resistance modulation of n-CdO/n-SnO$_2$ heterojunctions and the doping effect of Cd$^{2+}$ ions, however, which has not been studied.

Herein, we adopt a simple one-step electrospinning approach to synthesize n-CdO/n-SnO$_2$ nanofibers. To structure comparative experiments, n-CdO/n-SnO$_2$ nanofibers with different Cd/Sn molar ratios (0 mol%, 2 mol%, 5 mol%, 8 mol%) were prepared using fixed parameters. The enhanced 3H–2B sensing properties of n-CdO/n-SnO$_2$ nanofibers were systematically studied to establish the synergistic mechanism, which is based on the formation of n–n heterojunctions and the doping effect of Cd$^{2+}$ ions. This work offers a rational perspective to develop highly efficient 3H–2B gas sensing materials.

2. Materials and methods

Tin (II) chloride dihydrate (SnCl$_2$·2H$_2$O), Cadmium nitrate tetrahydrate (Cd(NO$_3$)$_2$·4H$_2$O), anhydrous ethanol (C$_2$H$_5$OH), N, N-dimethylformamide (DMF, C$_6$H$_5$NO) and 3H–2B (C$_4$H$_2$O$_2$·4H$_2$O and proportionate Cd$^{2+}$ ions. This work offers a rational perspective to develop highly efficient 3H–2B gas sensing materials.

The precursors of CdO/SnO$_2$ nanofibers with different Cd concentrations were prepared using the novel sol method [40, 41]. First, 0.4 g SnCl$_2$·2H$_2$O and proportionate Cd(NO$_3$)$_2$·4H$_2$O (the molar ratios of Cd and Sn were 0:100, 2:100, 5:100, 8:100) were dissolved in 8.8 g solution consisting of ethanol and DMF (mass ratio of 1:1). Then, add 0.8 g PVP into the dissolved solution of inorganic precursors. The solution was subsequently stirred for 12 h at room temperature to obtain a colloidal solution. Finally, fix the high voltage at 15 kV to spin the colloidal solution, and adjust the electrospinning distance to 20 cm. After electrospinning, the precursors were calcined at 600°C for 4 h to get CdO/SnO$_2$ nanofibers. The sensors based on nanofibers were fabricated similarly to previous reports, and the gas sensing record was undertaken by a CGS–8 system (Elite Tech) [42, 43]. The response is calculated by the ratio of the sensor resistances in air (R$_a$) and 3H–2B (R$_g$) [44].

SEM (Hitachi SU8010) with energy dispersive spectroscopy (EDS) was used to examine the morphology and elemental composition. The crystallographic structure was determined by XRD (PANalytical B.V. Empyrean, Cu Kα radiation, λ = 1.5406 Å) and TEM (JEOL JEM-2100) with selected area electron diffraction (SAED). The chemical states of surface elements were evaluated by XPS (Thermo Scientific Escalab 250Xi) with an Al Kα radiation source. The ASAP 2460 instrument was used to analyze the Brunauer–Emmett–Teller (BET) surface area and pore-size distribution.

3. Results and discussion

The morphologies of CdO/SnO$_2$ nanofibers with different Cd/Sn molar ratios are displayed in figures 1(a)–(d). After calcination, the precursors are transformed into porous nanofibers with a diameter of approximately 80–180 nm. As shown in figures 1(e)–(h) and figure 2(a), the Sn, Cd, and O elements in CdO/SnO$_2$ nanofibers are uniformly distributed on the surface. For 5 mol% CdO/SnO$_2$ nanofibers, the BET surface area is 20.36 m$^2$ g$^{-1}$, and the main pore size is concentrated at 7.80 nm (figure 2(b)). These mesopores can supply ample surface sites to facilitate the diffusion of target gases [45].

The rough surface and porous structure of CdO/SnO$_2$ nanofibers are approved by the TEM images (figure 3). It can be seen that CdO/SnO$_2$ nanofibers are composed of spherical-like nanoparticles. The particle boundaries can be clearly observed, and numerous pores are located around the junctions of these particles. As shown in the HRTEM image (inset of figure 3(c)), not only the dominant (1 1 0) plane of SnO$_2$ with uniform
lattice fringes, but also the dominant (111) plane of CdO with uniform lattice fringes can be observed. The interplanar spacing of 0.34 nm and 0.27 nm between neighboring fringes matches with the (110) crystal plane of SnO₂ and (111) crystal plane of CdO [46]. A clear interface appears between the parallel fringes of SnO₂ and CdO, indicating the formation of CdO/SnO₂ heterojunction interfaces. In the SAED pattern (inset of figure 3(c)), the polycrystalline structure of CdO/SnO₂ nanofibers agrees with the heterojunction between CdO and SnO₂ in HRTEM analysis.

The XRD patterns in figure 4(a) show that all the diffraction peaks of SnO₂ in CdO/SnO₂ nanofibers correspond with the tetragonal rutile phase (JCPDS No. 41-1445). With the increasing amount of CdO, the peak of CdO (JCPDS No. 75-0594) related to the (222) crystal plane becomes more obvious [47]. As shown in figure 4(b), the (110) and (101) peaks shift to a smaller angle after Cd doping. Considering that the ionic radius of Cd²⁺ is larger than that of Sn⁴⁺, this shift can be explained by the lattice distortion caused by the incorporation of Cd²⁺ into the SnO₂ lattice [48]. The aliovalent substitution can also be supported by the increased lattice parameters in table 1. In addition, as the amount of CdO increases, the intensity of peaks gradually decreases, suggesting that the functionalization of CdO can restrain the growth of SnO₂ grains [49].

The presence of Sn⁴⁺, Cd²⁺, and O²⁻ has been confirmed by the XPS spectrum of 5 mol% CdO/SnO₂ nanofibers (figure 5(a)) [50]. In figure 5(b), a blue shift of 0.8 eV can be seen for the Sn 3d binding energy of CdO/SnO₂ nanofibers compared with that of SnO₂ nanofibers, signifying the replacement of Sn⁴⁺ by Cd⁴⁺ in the SnO₂ lattice [51]. Figure 5(c) shows the Cd 3d binding energies at 406.4 eV and 413.1 eV. The Cd 3d₅/₂ peak and Cd 3d₃/₂ peak suggest the valence state of Cd²⁺ [52]. The asymmetrical O 1s peak with a shoulder in figure 5(d) can be decomposed into three components at 530.8 eV, 532.1 eV, and 533.1 eV, which are assigned to
Figure 3. TEM images of (a) 0 mol%, (b) 2 mol%, (c) 5 mol% and (d) 8 mol% CdO/SnO₂ nanofibers; inset of (c) HRTEM image and SAED pattern of 5 mol% CdO/SnO₂ nanofibers.

Figure 4. (a) XRD patterns of CdO/SnO₂ nanofibers with different Cd/Sn molar ratios; (b) enlarged (110) and (101) diffraction peaks of CdO/SnO₂ nanofibers.

Table 1. Lattice parameters of CdO/SnO₂ nanofibers with different Cd/Sn molar ratios.

| Samples | Lattice constant $a = b$ (Å) | Lattice constant $c$ (Å) | Unit cell volume ($Å^3$) |
|----------|-----------------------------|--------------------------|--------------------------|
| 0 mol%   | 4.738                       | 3.185                    | 71.50                    |
| 2 mol%   | 4.746                       | 3.187                    | 71.79                    |
| 5 mol%   | 4.755                       | 3.194                    | 72.22                    |
| 8 mol%   | 4.757                       | 3.194                    | 72.28                    |
the O$^{2-}$ ions in the SnO$_2$ lattice (O$_{L}$) and oxygen-deficient regions (O$_{V}$), and the chemisorbed oxygen species (O$_{C}$), respectively \cite{53}. The O$_{C}$ in CdO/SnO$_2$ nanofibers implies the important role of Cd doping in inducing chemisorbed oxygen species to improve the response.

Figure 6(a) presents the temperature dependence of the sensing responses to 3H-2B. When the Cd/Sn ratio is 5 mol\%, the maximum response of 45 appears at 260 °C. In figure 6(b), the lowest detection limit of CdO/SnO$_2$ nanofibers to 3H-2B is 20 ppb at the optimum temperature of 260 °C, and the responses are not saturated, even increasing the concentration of 3H-2B up to 100 ppm. The linear calibration curves (figure 6(c)) at sub-ppm levels (20 – 1000 ppb) also demonstrate the latent capacity for monitoring low concentrations of 3H-2B. Figure 6(d) depicts the response-recovery kinetics of CdO/SnO$_2$ nanofibers against 5 ppm 3H-2B at 260 °C. For 5 mol\% CdO/SnO$_2$ nanofibers, the response/recovery time does not exceed 9 s/5 s.

Figure 7(a) reveals the good selectivity of CdO/SnO$_2$ nanofibers to discriminate 3H-2B from interfering gases. The responses of CdO/SnO$_2$ nanofibers toward 3H-2B change little during 60 days (figure 7(b)). As illuminated in figure 8(a), the conspicuous repeatability of 5 mol\% CdO/SnO$_2$ nanofibers guarantees no distinct difference in the response value and response/recovery time. All above-mentioned gas sensing characteristics were measured under 40 RH\%.

Figure 8(b) records the 3H-2B responses of 5 mol\% CdO/SnO$_2$ nanofibers against higher humidity, and the decay is weak. The mechanism for the improved 3H-2B gas sensing characteristics of CdO/SnO$_2$ nanofibers may be discussed from the following two aspects: the resistance modulation of n-CdO/n-SnO$_2$ heterojunctions and the doping effect of Cd$^{2+}$ ions. As illustrated in figure 9, the majority of the Cd$^{2+}$ ions in CdO/SnO$_2$ nanofibers exist in the form of CdO; due to the difference between CdO and SnO$_2$ in work functions, electrons will transfer from CdO to SnO$_2$ until an equilibrium is reached between two Fermi levels \cite{54}. Thus, n-n heterojunctions are formed between the electron depletion layer in CdO and the electron accumulation layer in SnO$_2$. On the other hand, as proved by the characterizations including XRD and XPS, a part of the Sn$^{4+}$ ions in CdO/SnO$_2$ nanofibers are replaced by Cd$^{2+}$ ions through doping, leading to a change in the defect equilibrium and extra oxygen vacancies. The absorbed oxygen molecules by oxygen vacancies will capture the electrons in SnO$_2$ to generate additional chemisorbed oxygen species, resulting in an increased R$_{o2}$ \cite{55–57}. The chemisorbed oxygen species on the surface of nanofibers are closely related to the temperature. The dominant chemisorbed oxygen species include the O$_2^{-}$ at the working temperature lower than 150 °C, the O$^{-}$ at the working temperature of
150°C–400°C, and the O$^{2-}$ at the working temperature higher than 400°C [58, 59]. For the CdO/SnO$_2$ sensor, the absorbed oxygen molecules will change into more reductive O$^{-}$ rather than O$_2$ at the optimal temperature of 260°C. At higher temperatures, the possible aggregation of nanoparticles in the sensing layer may be unfavorable to the gas sensing response. When CdO/SnO$_2$ nanofibers are in contact with 3H-2B, the dominant chemisorbed oxygen species at the temperature range of 150°C–400°C, namely active O$^{-}$ ions on the surface, will oxidize 3H-2B molecules to produce acetic acid and CO$_2$ [60]. More electrons are delivered back to the conduction band of SnO$_2$ in this process, and the width of electron depletion layer is decreased. As a result, the lower $R_g$ and higher $R_a$ contribute to the enhancement of 3H-2B sensing characteristics [31, 61–63]. The 3H-2B gas sensing properties of SnO$_2$ nanomaterials or nanofiber-based materials in recent references are listed in table 2. Compared with other nanomaterials, CdO/SnO$_2$ nanofibers show comprehensive and impressive 3H-2B sensing characteristics.
The selectivity of CdO/SnO$_2$ nanostructures to 3H-2B may be dependent on the fact that different gases need different energies for the adsorption and desorption on the surface of gas sensing materials [65]. The sensor detects a certain gas at different working temperatures with different responses. The main reason for this phenomenon is the specificity of the optimum temperature to a gas, that is to say, a gas sensor based on metal oxides such as SnO$_2$ often tends to show different optimum temperatures towards diverse gases [27]. Apart from the difference in the optimal temperatures of the sensing material to 3H-2B and interference analytes, the more polar functional groups and chemical bonds in the molecular structure of 3H-2B make it more reactive to be absorbed on the surface, so is the oxidation of absorbed 3H-2B molecules by chemisorbed oxygen species [66–68]. The catalytic activity of CdO and porous nanostructure with n-CdO/n-SnO$_2$ heterojunctions enhances the above reactions further, thus endow the sensor with a selective detection for 3H-2B [69]. Since the preparation temperature is much higher than the working temperature, the structure of CdO/SnO$_2$ nanostructures can remain relatively stable during the sensing process. More importantly, the host material of the sensing layer,
SnO$_2$ is hydrophobic. The stable and hydrophobic SnO$_2$-based sensing layer raises the high-temperature operability of sensors against higher humidity, making the response decay at higher humidity is weak.

4. Conclusions

In summary, n-CdO/n-SnO$_2$ heterojunction nanofibers with different Cd/Sn molar ratios have been fabricated via a one-step in situ electrospinning route. The optimal 5 mol% CdO/SnO$_2$ nanofibers possess significantly enhanced 3H-2B gas sensing response, rapid response-recovery behavior, excellent selectivity, and supreme stability at 260 °C, ascribed to the synergy between the resistance modulation of n-CdO/n-SnO$_2$ heterojunctions and the doping effect of Cd$^{2+}$ ions. We expect the combination strategy in this study could be applied to more semiconductor sensing materials for the real-time monitoring of 3H-2B.

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Data availability statement

The data that support the findings of this study are available upon reasonable request from the authors.

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