Facile Design and Hydrothermal Synthesis of In$_2$O$_3$ Nanocube Polycrystals with Superior Triethylamine Sensing Properties

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**ABSTRACT:** Triethylamine (TEA), a typical kind of volatile organic compound, is widely used as an industrial solvent, which is a threat to environment and human health. In this paper, a novel In$_2$O$_3$ nanocube gas sensing material with high sensing performances was synthesized through a simple one-pot hydrothermal method. The gas sensing test results are satisfactory such that the single phase of In$_2$O$_3$ nanocubes even exhibits a quite higher response of 12−10 ppm of TEA at a lower temperature (180 °C) than many conventional In$_2$O$_3$-based complexes and maintains high consistency in morphology and stability after the consecutive tests of 2 months. This work provides a facile and quite effective gas sensing material for TEA gas monitoring with high sensitivity and stability at lower working temperature that can be prepared in batch and further used as templates.

**INTRODUCTION**

With the improvement of health consciousness for people, more and more attention has been paid to the air quality in workplace and living environment. Triethylamine (TEA), a typical volatile organic compound with strong ammonia odor, is widely used as a low polar solvent, curing agents, catalysts, preservatives, and synthetic fuels in industry, which is liable to harm the environment and human health because of its volatility, flammability, and explosive properties. Long-term contact with human body can lead to eye and skin burns, vascular headache, breast cancer, embryo abnormalities, and so forth. According to the recommendations of the National Institute for Occupational Safety and Health Administration, the environmental concentration of TEA should be lower than 10 ppm. Nowadays, a variety of ways have been implemented for the detection of TEA, including gas/liquid/film chromatography, electrochemistry analysis, colorimetric method, and other customary methods. Although these traditional methods are effective for TEA detection, they usually suffer from several challenges such as relative low sensitivity, long response and recovery time, and poor selectivity. Therefore, the development of highly efficient TEA sensors with available sensitivity and favorable selectivity under lower/room working temperature is of great significance.

In the past decades, some classical metal oxide semiconductor (MOS) nanomaterials exhibit low cost, easy fabrication, high sensitivity, fast response, portability, and other benign characteristics, thus having potential applications in TEA measurement. Among those, In$_2$O$_3$, a typical n-type semiconductor with a wide band gap (a direct gap of 3.5−3.7 eV), has excellent conductivity and superior photo-electrochemical stability to pave the way for gas sensors. Besides, the uniformity, morphology, and constitution of materials are known to have great effects on their sensing performances; therefore, various In$_2$O$_3$ products with modified morphology or tunable components have been fabricated, especially, these In$_2$O$_3$ nano/microstructures have manifested good sensing performance to CO, ethanol, formaldehyde, acetone, etc. Comparatively, it is a rare report on the safety detection of TEA by In$_2$O$_3$-based nanocomposites, not to mention the single phase of In$_2$O$_3$ with highly efficient sensing performances and lower working temperatures. Based on these requirements, so far, we have not found many reports. Typically, Yang and the co-workers reported that sensors based on In$_2$O$_3$ microtubes showed a response of 75−100 ppm TEA at 300 °C. Zheng et al. fabricated the sensors based on synergy between Au and In$_2$O$_3$ microspheres which had a response of 648.2−100 ppm TEA at 280 °C. Hence, it may be a good concept of developing a single-phase In$_2$O$_3$ instead...
of its complex to achieve the same or even better detection effect through a facile process.

According to the report, different nanoscaled MOS-based gas sensors exhibit quite different sensing properties, even the same kind of MOS enables different results. This may cause by many factors including surface morphology, grain size, specific surface area, exposed crystal facet, etc. In order to obtain a deeper acquaintance with the effect of structure construction styles on gas sensing performances, we took n-type In$_2$O$_3$ as an example on the premise of controlling the above factors and reported a novel In$_2$O$_3$ nanocube candidate by a simple hydrothermal method with a subsequent calcination process. According to the sensing test results and comparison table, the single-phase In$_2$O$_3$ nanocubes displayed competitive performances in the TEA gas-sensitive material such as high sensitivity, good selectivity, lower working temperature, and so forth, demonstrating that the as-synthesized In$_2$O$_3$ nanocubes could be potentially used for TEA gas pollutant monitoring.

## RESULTS AND DISCUSSION

### Characterization.

The crystal structure and purity of the sample were determined by X-ray diffraction (XRD). As shown obviously in Figure 1a, the diffraction peaks of our product are highly consistent with the standard In$_2$O$_3$ card (JCPDS 06-0416), and no other impurity diffraction peaks could be found. Besides, all the sharp patterns with high intensity indicate that the In$_2$O$_3$ nanocubes made by the facile hydrothermal method are of high crystallinity and purity. Further, X-ray photoelectron spectroscopy (XPS) spectra were analyzed to investigate the surface composition and chemical states of the elements for the In$_2$O$_3$ nanocubes. Figure 1b shows that the In$_2$O$_3$ nanocubes were mainly composed of In and O elements, while the C element comes from the conductive tape used in scanning electron microscopy (SEM). It indicates that no other impurities are observed in the sample.

Figure 1. (a) XRD pattern, (b) survey spectra, (c) In 3d, and (d) O 1s of XPS spectra of In$_2$O$_3$ nanocubes.

![Figure 1](image1.png)

To give a more clear insight into the microstructure, Figure 3 presents the typical transmission electron microscopy (TEM) and high-resolution TEM (HRTEM) images of In$_2$O$_3$ nanocubes. Figure 3a,b shows the TEM and HRTEM images of In$_2$O$_3$ nanocubes, and the In$_2$O$_3$ nanocube side length is about 200 nm, which is consistent with the previous SEM images. From the HRTEM image in Figure 3c, the lattice fringes can be seen clearly. The distance of the fringes at 0.252 nm corresponds to the (400) plane of In$_2$O$_3$ nanocubes and that at 0.292 nm corresponds to the (222) plane. The corresponding selected area electron diffraction (SAED) image displayed in Figure 3d shows the polycrystal planes of (211), (222), (400), (440), and (622) of the cubic phase In$_2$O$_3$, which demonstrates the coincidence of the XRD results.

![Figure 2](image2.png)

![Figure 3](image3.png)

and these nanocubes are uniform and neat. Furthermore, in the higher magnification of Figure 2b, the surface of In$_2$O$_3$ is smooth, and the edges and angles are clear with the surface size about 180–200 nm in width. Figure 2c shows the mapping patterns of the as-prepared In$_2$O$_3$ nanocubes. From the spectrum, it can be seen that the sample contains only three elements, In, O, and C, among which the C element comes from the conductive tape used in scanning electron microscopy (SEM). It indicates that no other impurities are observed in the sample.

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Gas Sensing Test. The In$_2$O$_3$ nanocubes were fabricated as a gas sensor, and the sensing properties were tested. As is known to all, working temperature is one of the important factors to evaluate the performance of MOS-based gas sensors. In order to determine the optimum working temperature of In$_2$O$_3$ nanocubes, 100 ppm of TEA was tested with dry air as the background gas along with the temperature from 60 to 240 °C. As shown in Figure 4a, during the target operating temperature range, first the response of the In$_2$O$_3$ nanocube sensor increases with the rise of temperature until 180 °C and then reaches the maximum response at 180 °C; afterward, the response decreases sharply with the increase of working temperature; thus, the optimum working temperature is determined as 180 °C. Therefore, the subsequent experiments are carried out under the optimal working temperature of 180 °C. Besides, humidity interference is a difficult problem in practical application, so the humidity resistance of the device was tested in different relative humidity (RH) conditions to 100 ppm of TEA, as shown in Figure 4b. As can be seen in Figure 5a, the response increases with the increase of gas concentration and exhibits excellent positive correlation. Besides, the response ($S$) versus gas concentration of MOS gas sensors is usually empirically represented as $S = aC^b + 1$ where $a$ and $b$ are the constants and $C$ is the concentration of the test gas. At a certain operating temperature, the above equation can be rewritten as $\log(S - 1) = b\log(C) + \log a$. As shown in Figure 5b, the sensor based on In$_2$O$_3$ nanocubes exhibits a good linear relationship with the concentration of TEA (10–200 ppm) in logarithmic forms, which is in good agreement with the theory of power laws proposed for semiconductor sensors. The above result shows that TEA detection is feasible in practice.

Selectivity and circularity are also important performance indicators for gas sensors. As shown in Figure 6, in order to exhibits fine humidity resistance to a certain extent but needs further improvement.

In addition, the dynamic response change curves of In$_2$O$_3$ sensor at different concentrations (10, 20, 50, 100, and 200 ppm) of TEA at 180 °C have been provided in Figure 5. As can be seen in Figure 5a, the response increases with the increase of gas concentration and exhibits excellent positive correlation. Besides, the response ($S$) versus gas concentration of MOS gas sensors is usually empirically represented as $S = aC^b + 1$ where $a$ and $b$ are the constants and $C$ is the concentration of the test gas. At a certain operating temperature, the above equation can be rewritten as $\log(S - 1) = b\log(C) + \log a$. As shown in Figure 5b, the sensor based on In$_2$O$_3$ nanocubes exhibits a good linear relationship with the concentration of TEA (10–200 ppm) in logarithmic forms, which is in good agreement with the theory of power laws proposed for semiconductor sensors. The above result shows that TEA detection is feasible in practice.

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Further confirm the selectivity of the In$_2$O$_3$ nanocube sensor, 100 ppm of 14 kinds of industrial gases was chosen to test at 180 °C. Figure 6a shows that the test gas types can be divided in to five categories: amines, alcohols, ketones, aldehydes, and benzene. However, In$_2$O$_3$ nanocubes display highest response to TEA of amines ($S$ of 175), about 3 or 4 times higher than alcohols, ketones, aldehydes, and benzene, which illustrates that the In$_2$O$_3$ nanocube-based gas sensor possesses good selectivity to TEA among various industrial gases that are very helpful for practical applications. Furthermore, in order to verify the stability of the In$_2$O$_3$ nanocube sensor, the gas sensitivity versus 100 ppm of TEA was detected every 6 days for a total of 3 times. As shown in Figure 6b, the sensor was basically stable and the response remained stable at a high level as there is little change in the error. Figure 6c–e shows the FESEM images of the samples after 2 months of continuous testing. As can be seen, the morphology of the same position basically remains unchanged. This result fully proves that the sensor can run for a long time and remain stable and efficient, which also meets the expectation of the design and can be applied to the actual detection.

**Gas Sensing Mechanism.** To assess the sensing capabilities of the In$_2$O$_3$ nanocubes, a comparison between our work and other literature studies including In$_2$O$_3$-type gas sensors is listed in Table 1. It is clear that the sensor fabricated by In$_2$O$_3$ nanocubes displayed superiority in the detection of TEA compared to other reports, especially with a quite higher response of 175 for 100 ppm of TEA at a lower temperature (180 °C), which fully demonstrates that the single phase of In$_2$O$_3$ can also be equal with its complexes to show excellent gas sensitivity and will save a lot of tedious processing. Or it can be further treated as a template or platform with good sensing performances.

As a typical n-type MOS, the change of In$_2$O$_3$-based gas sensitivity is caused by the change of the resistance, the surface of the sensing material and target gas adsorption reaction. As shown in Figure 7, when In$_2$O$_3$ sensors are exposed to air, oxygen molecules from the conduction band can be adsorbed on the surface of In$_2$O$_3$ and capture the free electrons in the form of oxygen ion and then adsorbed on the surface of the sensor. As the reaction temperature increases, different forms of oxygen ions can be formed, as described in eqs 1–4. According to the previous experiment, the optimal operating temperature is 180 °C, and the reaction product is determined as eq 3.

$$\text{O}_2(\text{gas}) \rightarrow \text{O}_2(\text{ads})$$

$$\text{O}_2(\text{ads}) + e^- \rightarrow \text{O}_2^-(\text{ads}) \quad (T < 100 \, ^\circ \text{C})$$

$$\text{O}_2^-(\text{ads}) + e^- \rightarrow 2\text{O}^-(\text{ads}) \quad (100 \, ^\circ \text{C} < T < 300 \, ^\circ \text{C})$$

$$\text{O}^-(\text{ads}) + e^- \rightarrow \text{O}^{2-}(\text{ads}) \quad (T > 300 \, ^\circ \text{C})$$

$$\text{N}((\text{C}_2\text{H}_5)_3(\text{gas}) + 21\text{O}^-(\text{ads}) \rightarrow \text{N}_2 + 6\text{CO}_2 + 9\text{H}_2\text{O} + 21e^-$$

When the oxygen molecules are adsorbed onto the In$_2$O$_3$ surface, O$^-$ ions will be generated and lead to the formation of thick electronic depletion layer, resulting in the decrease of free-charge-carrier (electron) concentration or increase of sensor resistance. When the reducing gas of TEA contacts with the surface of the sensor, it will react with captive O$^-$ ions and release electrons back to the conduction band of In$_2$O$_3$, thus narrowing the electronic depletion layer and reducing the sensor resistance at the same time, such reaction is described in eq 5.

The reason for high selectivity to TEA can be explained as follows. Reduction and adsorption abilities of target gas are important factors that determine material response. First of all, different bond energies have a great influence on the activity of the compound. The higher the bond energy of the compound, the more difficult it is to break. The bond strengths of C–N, O–H, C–C, C=O, and C=H are 307, 462, 345, 748.2, and 411 kJ/mol, respectively. Obviously, the bond energy of C–N in TEA is lowest among all the detected gases, so its activity is highest. Second, CH$_3$CH$_2$– is a kind of electron-donating group, and the electron cloud density around N atoms in TEA is higher than O atoms in ethanol, methanol, acetone, formaldehyde, etc. Additionally, the high attraction between N atoms and In$^{3+}$ makes TEA molecules more easy to stick to the surface, which indicates that the number of TEA molecules involved in surface catalytic oxidation will increase probably such that the response to TEA is higher than other gases. Based on the above two points, the sensor can exhibit a good selectivity to TEA.

**CONCLUSIONS**

In summary, the special In$_2$O$_3$ nanocubes were prepared through a simple hydrothermal synthesis and characterized by XRD, XPS, SEM, and TEM analyses to confirm the polycrystal...
structure, pure composition phase, and uniform morphology of our product. For its practical application, the gas sensitivity of the product was systematically studied. The results are satisfactory such that the single phase of In$_2$O$_3$ nanocubes even exhibits a quite higher response of 175 to 100 ppm of TEA at a lower temperature (180 °C) than many conventional In$_2$O$_3$-based complexes and maintains high consistency in morphology and stability after the consecutive tests of 2 months, especially certain degree of humidity resistance. The work provides a facile and quite effective polycrystallized In$_2$O$_3$ material for TEA gas monitoring and exhibits high sensitivity and stability and good selectivity at lower working temperature that can be prepared in batch or further used as templates for the in-depth processing with other novel multifunctional applications.

## EXPERIMENTAL SECTION

### Synthesis of In$_2$O$_3$ Nanocubes.

All the reagents used in the experiment, including indium nitrate (In(NO$_3$)$_3$·5H$_2$O, AR, 99.9%), and urea (CH$_4$N$_2$O, AR, 99%), were purchased from Aladdin Industrial Corporation (Shanghai, China). Sinopharm Chemical Reagent Co. Ltd. (Shanghai, China) provides ethanol (CH$_3$CH$_2$OH, AR). Besides that, the distilled water was used in the synthesis procedures. In a typical procedure, 0.362 g of In(NO$_3$)$_3$·5H$_2$O and 0.720 g of urea were dissolved in two beakers with 40 mL of distilled water, respectively. Then, after 600 r/min magnetic stirring at room temperature for 15 min, the In(NO$_3$)$_3$ solution was slowly dropped into urea solution and stirred for 15 min. Then, the mixture was transferred into a 100 mL Teflon-lined stainless steel autoclave and heated at 130 °C for 3 h (a heating rate of 2 °C/min) to obtain a pale yellow solid product.

### Characterization.

The samples were characterized by means of powder XRD analysis (Rigaku Ultima IV, Japan; Cu Kα radiation, λ = 1.5418 Å), FESEM (Hitachi SU5000, Japan), TEM and HRTEM with energy-dispersive X-ray spectroscopy (FEI Tecnai G2 F20 s-twin, 200 kV), and XPS (Thermo Scientific ESCALAB 250Xi, Al Kα X-ray monochromator).

### Preparation of Gas Sensor and Sensing Test.

The detailed preparation process of gas sensor has been proposed in our previous research, which is simply described here. A proper amount of sample was ground with adding several drops of deionized water until it became sticky and then coated on the surface of the ceramic tube. A Ni–Cr alloy filament heater was put through the ceramic tube to control the operating temperature by adjusting the heating voltage. The ceramic tube and the Ni–Cr heater were then welded onto a pedestal with six probes to produce the final sensor device. Gas sensing test was carried out through the CGS-8 intelligent test system (Beijing Elite Tech Co. Ltd., China). A calculated volume of liquid analyte was injected into a glass chamber with a microsyringe, evaporated immediately, and mixed with air. When the response was stable, the whole air sensitivity test is completed and continued to sweep with air until the sensor refreshed again. The response value (S) of sensors based on the n-type semiconductor is defined as $S = R_a / R_p$, where $R_a$ and $R_p$ represent the resistance value of sensor in air and in target gas, respectively, while the p type is the opposite. Response time is defined as the time taken by the sensor to reach 90% of the final equilibrium value or the recovery time for gas desorption. Most tests were carried out under dry air condition, except the discussion of RH effect on the response of the sensor.  

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**Notes**

The authors declare no competing financial interest.

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