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Solid-state chemical synthesis of mesoporous α-Fe_2O_3 nanostructures with enhanced xylene-sensing properties

Yizhao Li, Yali Cao, Dianzeng Jia*, Yang Wang, Jing Xie

Key Laboratory of Material and Technology for Clean Energy, Ministry of Education, Key Laboratory of Advanced Functional Materials, Autonomous Region, Institute of Applied Chemistry, Xinjiang University, Urumqi, Xinjiang 830046, China

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ABSTRACT

Uniform mesoporous α-Fe_2O_3 nanostructures have been handily prepared by a solid-state chemical reaction with the features of simple process, mild condition, and high yield. The as-prepared samples with 3-dimensional (3D) honeycomb structures consist of a number of small nanosheets. These mesoporous α-Fe_2O_3 nanostructures have been investigated for application as a sensor to detect various vapors. The experiment results have shown that the mesoporous α-Fe_2O_3 nanostructures exhibited improved performances for xylene-sensing in comparison with the α-Fe_2O_3 nanosheets. The response of mesoporous nanostructures to 1000 ppm xylene was up to 6 times higher than that of nanosheets. The mesoporous α-Fe_2O_3 nanostructures-based sensor had also wide detection range of 1–1000 ppm, good selectivity, and short response time to xylene. The enhancement of properties may be attributed to the large specific surface area and porous nanostructure.

1. Introduction

Gas sensors have been widely used in gas monitoring and alarm in firefighting, industry, and daily life [1–3]. Nanostructured α-Fe_2O_3 has been intensively studied as gas sensing materials owing to its low cost and promising sensing performance associated with nanoscale size [4–9]. To date, various α-Fe_2O_3 structures, such as nanospheres [10], nanorods [11], nanotubes [12], nanowires [13], and nanorings [14], are widely investigated. In particular, mesoporous α-Fe_2O_3 which possesses unique porous structure has attracted much attention because of their enhanced properties [15,16].

Some efforts have been made to prepare mesoporous α-Fe_2O_3 nanostructures for gas sensors. Sun et al. reported the synthesis of mesoporous α-Fe_2O_3 nanostructures through a soft template synthesis method using the triblock copolymer surfactant F127 as the template [17]. The mesoporous α-Fe_2O_3 materials showed high gas sensitivity toward acetic acid and ethanol gas. Hao et al. have fabricated flower-like and urchin-like mesoporous α-Fe_2O_3 nanostructures by annealing the hierarchical α-FeOOH precursors prepared through a solution-based reaction [18]. The obtained mesoporous hierarchical α-Fe_2O_3 architectures exhibited enhanced sensing performances to ethanol. Although the mesoporous α-Fe_2O_3 nanostructures in the above methods revealed good gas sensing performance, the preparations of the samples were complex, expensive, and time-consuming. Therefore, it is still a challenge to develop a facile route to synthesize mesoporous α-Fe_2O_3 nanostructures with improved gas sensing properties.

In this paper, we report a solid-state chemical reaction to synthesize mesoporous α-Fe_2O_3 nanostructures constructed by small nanosheets. The fabrication shows the features of simple process, mild condition, high yield, and low cost. The gas sensing properties of the resulting mesoporous nanostructures and α-Fe_2O_3 nanosheets were investigated. The mesoporous nanostructures-based gas sensor showed improved xylene sensing performances in comparison with the compact nanosheets-based gas sensor. The results are promising for further application of mesoporous α-Fe_2O_3 nanostructures as xylene sensor.

2. Experimental

2.1. Synthesis

All the reagents were analytically pure from commercial sources and used without further purification. The mesoporous α-Fe_2O_3 nanostructures were synthesized through a solid-state chemical reaction method. In a typical experiment, $FeCl_2{\cdot}4H_2O$ (0.60 g, 3 mmol) and $NaBH_4$ (0.23 g, 6 mmol) were mixed with sodium

* Corresponding author. Tel.: +86 991 8583083; fax: +86 991 8580032.
E-mail addresses: jdz0991@gmail.com, jdz@xju.edu.cn (D. Jia).

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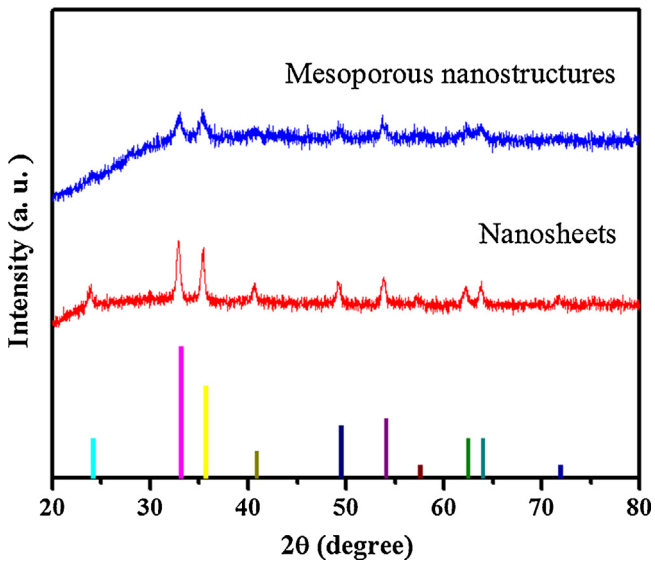

Fig. 1. XRD patterns of the mesoporous nanostructures and the nanosheets.

dodecyl sulfate (SDS) (0.87 g, 3 mmol) by grinding in an agate mortar at room temperature. Subsequently, several drops of water were slowly added into the mixtures. Accompanied with the release of heat and vapor, the color of mixtures changed from green to black. After the mixtures were ground for about 30 min, the resulting solid products were washed with distilled water and absolute ethanol for several times. The products were then dried at room temperature for 24 h followed by annealing at 600 °C for 1 h in air atmosphere. The α-Fe_2O_3 nanosheets were synthesized by a similar process as described above, except for without the addition of SDS.

2.2. Characterization

Powder X-ray diffraction (XRD) patterns were recorded on a Bruker D8 X-ray diffractometer with Cu-Kα radiation (λ = 1.54056 Å) and a scanning speed of 2° min^{-1} ranging from 20° to 80°. Transmission electron microscope (TEM) images were obtained on a Hitachi H-600 transmission electron microscope with accelerating voltage of 100 kV. Scanning electron microscope (SEM) images were obtained on a LEO 1430VP scanning electron microscope with an accelerating voltage of 20 kV. The Brunauer–Emmett–Teller (BET) and Barret–Joyner–Halender (BJH) results were measured on a Micromeritics ASAP 2020 surface area and porosity analyzer. UV–vis spectrum was obtained by a Hitachi U-3900H spectrophotometer.

2.3. Gas sensing test

Gas sensors were made in a conventional way [19,20]. Briefly, the as-prepared products were dispersed in terpineol, which was used as the binder to form pastes. The alumina ceramic tube, assembled with platinum wire electrodes for electrical contacts, was dipped into the paste several times to form the sensing film. Then a Ni–Cr alloy wire as a resistance heater was passed through the ceramic tube. To improve the stability and repeatability, the sensors were aged at 300 °C for 5 days in air prior to use. The test was carried out in a commercial gas sensing measurement system of WS-30A (Zhengzhou Winsen Electronic Technology Co., Ltd.).

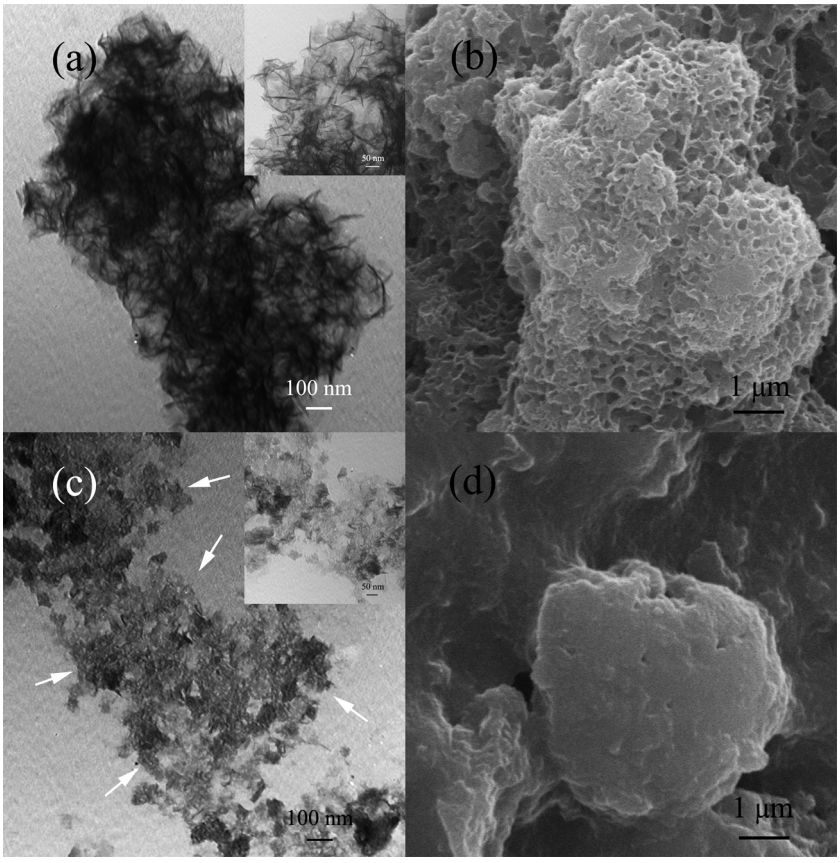

Fig. 2. Typical TEM image (a) and SEM image (b) of the mesoporous α-Fe_2O_3 nanostructures; typical TEM image (c) and SEM image (d) of the α-Fe_2O_3 nanosheets.