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Hematite nanoplates: Controllable synthesis, gas sensing, photocatalytic and magnetic properties

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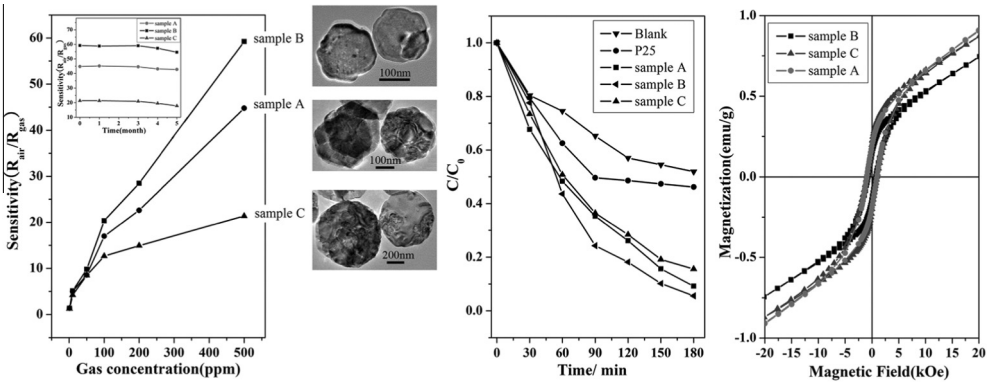
GRAPHICAL ABSTRACT

Single-crystalline α-Fe_2O_3 nanoplates with different size were synthesized, demonstrating remarkable gas sensitivity and stability towards *n*-butanol, good photocatalytic properties towards RhB, and weak ferromagnetic behavior.



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ABSTRACT

Uniform hematite (α-Fe_2O_3) nanoplates exposing {001} plane as basal planes have been prepared by a facile solvothermal method under the assistance of sodium acetate. The morphological evolution of the nanoplates was studied by adjusting the reaction parameters including the solvent and the amount of sodium acetate. The results indicated that both the adequate nucleation/growth rate and selective adsorption of alcohol molecules and acetate anions contribute to the formation of the plate-like morphology. In addition, the size of the nanoplates can be adjusted from ca. 180 nm to 740 nm by changing the reaction parameters. Three nanoplate samples with different size were selected to investigate the gas sensing performance, photocatalytic and magnetic properties. As gas sensing materials, all the α-Fe_2O_3 nanoplates exhibited high gas sensitivity and stability toward *n*-butanol. When applied as photocatalyst, the α-Fe_2O_3 nanoplates show high photodegradation efficiency towards RhB. Both the gas sensing performance and the photocatalytic property of the products exhibit obvious size-dependent effect. Magnetic measurements reveal that the plate-like α-Fe_2O_3 particles possess good room temperature magnetic properties.

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1. Introduction

As a low cost, nontoxic *n*-type semiconductor with band gap of 2.1 eV, hematite (α-Fe_2O_3) is the most stable iron oxide under ambient environment. In recent years, α-Fe_2O_3 has attracted considerable attention due to its wide potential applications in many fields, such as magnetic devices, catalytic/photocatalytic fields, pigments, gas sensing, photo anode, and rechargeable lithium-ion batteries [1–4]. As is well known, shape, size, surface structures, and microstructures are the main factors that influence the chemical and physical properties of nanomaterials. Thus, considerable efforts have been devoted to design and tailor the size and morphology of hematite nanomaterials to enhance and improve their performance in the above applications. To date, a large number of hematite nanomaterials with different structure including nanocrystals [5–7], polyhedral nanoparticles [8–11], nanorods [12], nanoribbons [13], nanotubes [14], nanostructured microspheres [15,16], hollow nanostructures [17,18] and nanoplates [19–29] have been developed as anode materials for lithium ion batteries, gas sensing materials and photocatalysts. In particular, nanostructured hematite has shown dramatically enhanced activity and sensitivity compared with its bulk counterparts in photocatalytic and gas sensing fields due to their novel surface structures and high surface to volume ratio.

Recently, there is an increasing interest in the controlled synthesis and property investigation of 2D hematite nanomaterials such as nanoplates or nanodisks [19–30]. Chen and Zhu et al. reported the top-down etching fabrication of α-Fe_2O_3 nanodisks and studied their lithium storage properties [22]. Lu et al. and Lu et al. have prepared hematite nanoplates and reported their performances in lithium-ion batteries [23,24]. Gas sensing properties based on porous plate-like hematite mesocrystals prepared using an ionic liquid assisted solvent evaporation process [25] and assembled columnar superstructures of α-Fe_2O_3 nanoplates [26] have been reported. Photocatalytic property based on several layered structured α-Fe_2O_3 nanodisks, mesocrystalline hematite nanoplates, 2D hollow hematite microplatelets have been reported by different research groups [27–29]. Although there have been some reports of the gas sensing and photocatalytic property about 2D α-Fe_2O_3 nanostructures, the reports based on single-crystalline hematite nanoplates are still scarce. It is necessary to further explore systematically the effect of particle size and shape on gas sensing and photocatalytic property related to single-crystalline α-Fe_2O_3 nanoplates.

Herein, a surfactant-free solvothermal process for the synthesis of uniform α-Fe_2O_3 nanoplates is reported. The size of the α-Fe_2O_3 nanoplates can be tailored over a certain range simply through adjusting the concentration of the sodium acetate (NaAc) or solvent. Furthermore, it was found that adequate nucleation/growth rate and selective adsorption of capping agent had remarkable effects on the formation of the nanoplates. The gas sensing property, visible-light photocatalytic property and magnetic property of the as-obtained α-Fe_2O_3 nanoplates were investigated. The results showed that these α-Fe_2O_3 nanoplates may have potential applications in gas sensing and photocatalysis fields.

2. Experimental section

2.1. Synthesis

All the reagents were of analytical grade and were used as received without further purification. The α-Fe_2O_3 nanoplates were prepared by a surfactant-free solvothermal process. In a typical synthesis, 0.500 g of $FeCl_3{\cdot}6H_2O$, 1.350 g of sodium acetate (NaAc) was dissolved into the 14.5 mL polyethylene glycol 400 (PEG, MW 400), the mixed solution was then stirred vigorously for 30 min to form a red viscous solution, which was transferred into a Teflon-lined autoclave of 20.0 mL capacity and heated at 200 °C for 12 h in an electronic oven. After completion of the reaction, the autoclave was cooled to room temperature naturally. The red precipitate was collected by centrifugation and washed several times with distilled water and absolute ethanol and dried at ambient temperature. To investigate the effect of the solvent, PEG600, methanol, ethanol, *n*-butanol, *n*-hexanol and distilled water were also used as solvent.

2.2. Characterization

The phase composition and purity of the products were identified by X-ray diffraction (XRD) with a Bruker D8 ADVANCE diffractometer with Cu Kα radiation (λ = 0.15418 nm). The morphologies and microstructures of the samples were characterized by a field-emission scanning electron microscope (SEM, FEI NOVA Nano SEM 230), and a high-resolution transmission electron microscope (HRTEM, FEI, Tecnai G2 F20). Magnetic measurements were carried out at room temperature on a Quantum Design MPMS SQUID VSM DC magnetometer with the field sweeping from −20,000 to 20,000 Oe.

2.3. Gas sensor fabrication and sensing performance measurements

The gas sensing properties of the as-prepared α-Fe_2O_3 nanoplates were tested on a WS-30A sensor measurement system (WeiSheng Electronics Co., Ltd., Henan, China). Certain amount of α-Fe_2O_3 products was mixed with several drops of water and was ground in an agate mortar to form a paste. Then the paste was coated onto a ceramic tube previously mounted with Au electrodes and Pt wires to form a thin sensing film of ca. 10 μm in thickness. A Ni–Cr alloy filament was put through the ceramic tube and used as a heater to control the operating temperature by tuning the heating voltage. The ceramic tube was then welded onto a pedestal with six probes to produce the final sensor unit. A calculated volume of liquid analytes was injected into a glass chamber by a micro-syringe, evaporated immediately and mixed with air. When the response reached a constant value, the sensor was exposed to air again by opening the chamber. The gas sensitivity of the sensor was defined as the ratio of R_{air}/R_{gas}, where R_{air} and R_{gas} are the electrical resistance of the sensor measured in air and in the test gas, respectively. The time taken by the voltage to reach 90% of its saturation after applying or switching off the gas is defined as the response time or recovery time.

2.4. Photocatalytic evaluation

The photocatalytic activity of the α-Fe_2O_3 samples was evaluated by photocatalytic degradation of RhB under the irradiation of the visible light. The degradation reactions were conducted in an XPA-7 type photochemical reactor (Xujiang Machine Factory, Nanjing, China). The experimental procedure was conducted as follows: 12 mg of the as-prepared samples were immersed in a rhodamine B (RhB) solution (2.0×10^{-5} M, 60 mL) and 0.30 mL of a hydrogen peroxide solution (H_2O_2, 30 wt.%) was injected. Then, the mixture was magnetically stirred in the dark for 15 min to reach the adsorption equilibrium and uniform dispersibility. The solution was irradiated with simulated visible light (a 500 W xenon lamp with a 420 nm cutoff filter) at room temperature for 3 h. During the irradiation, the suspension was magnetically stirred using a magnetic stirrer. Every 30 min, 10 mL of solution was sampled and centrifuged immediately to remove the photocatalyst particles. The dye concentration in the supernatant solution was analyzed by measuring the absorption intensity of RhB at 553 nm.