



Visible-light-driven photocatalytic properties and electronic structures of nickel sulfide nanoflowers



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ABSTRACT

Nickel sulfide (NiS) nanoflowers with the thickness of ca. 5–10 nm and size up to several hundreds of nanometers were synthesized via a facile polyol refluxing process under the open-air condition. The photocatalytic properties of NiS nanoflowers were evaluated by the decomposition ratio of MB was up to nearly 98% after 3 h visible light irradiation, indicating the NiS nanoflowers were good candidates for high performance photocatalysts. Meanwhile, the influencing factor of the photocatalytic reaction had also been studied by calculating the electronic structure of NiS nanoflowers. The band structure indicates that charge transfer upon photoexcitation occurs from the S 3p orbital to the empty Ni orbital.

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1. Introduction

The photocatalytic technique for the degradation of organic contaminants in air or in solution has been widely studied to remedy the effects of environmental pollution because of its simple and exhaustive decomposition process [1–5]. Among all kinds of semiconductors, TiO₂ is one of the most important oxides photocatalyst, which has been widely used [6–9]. However, with a wide band gap of 3.2 eV, TiO₂ is only sensitive under ultraviolet light region and is not active to visible light. It is well known that only 4% of the solar spectra falls in the ultraviolet region, thus much work has been done to synthesize visible light photocatalyst in view of the better utilization of solar energy. Through modification of TiO₂, including doping with metal, such as Fe, Au, Ag [10–12], and nonmetal atoms, such as C, N, and S [13–15], a lot of visible-light-driven photocatalysts have been synthesized. On the other hand, many traditional non-titanium-based visible light photocatalysts have been synthesized, for example, some sulfides have been found to have visible-light-driven catalytic activity, such as SnS [16], Sb₂S₃ [17], CdS [18], and so on [19,20]. All of these studies may provide new insights for the design of visible-light-driven photocatalysts.

Flower-like semiconductor nanomaterials with a large surface area and high photocatalytic activity have been widely investigated due to their important applications in purifying water and air fields. For instance, 3D flower-like Ni(OH)₂ products prepared by Shen's group showed high selective adsorption for cationic/anionic dyes in aqueous solutions under visible light irradiation [21]. From a hydrothermal method, Jing and his coauthors have synthesized flower-like Ni²⁺ doped ZnIn₂S₄ microsphere with greatly enhancing the activity of the photocatalyst [22]. However, few report concerns the yield of nanomaterials via a facile route, thus remains a considerable challenge.

Thus, developing simple and effective methods to fabricate semiconductors with flower-like structures is important for future visible-light-driven photocatalysts applications. The facile polyol refluxing process has several advantages over the conventional hydrothermal method, such as facile, short heating time to the reaction temperature, high output and open-air condition [16,23]. Up to now, to the best of our knowledge, little work on the preparation of nickel sulfide nanoflowers in liquid systems has been reported. Here, the simple synthesis of flower-like NiS from one-step, facile solution chemical method under the open-air condition was realized for the first time. The optical properties, photocatalytic activity and the electronic structures of the NiS nanoflowers were investigated. It was found that the obtained NiS nanoflowers have high photocatalytic activity in degrading

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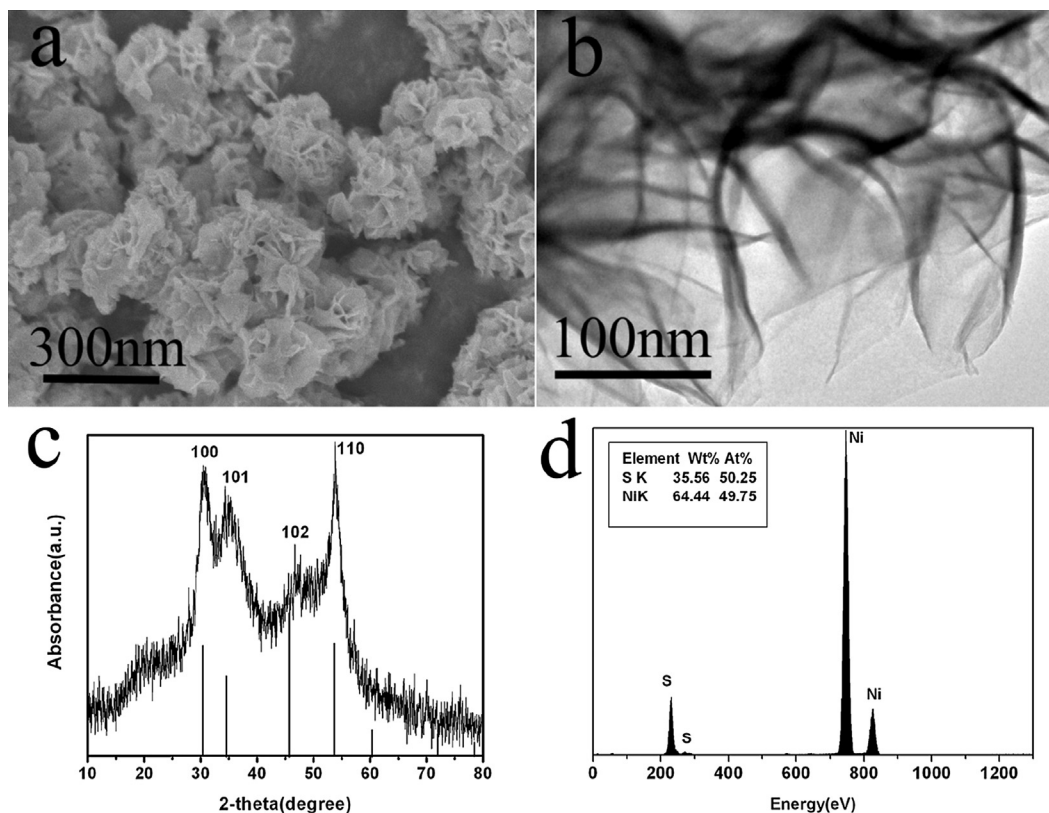


Fig. 1. (a) SEM image, (b) TEM image, (c) XRD pattern and (d) EDS of the as-synthesized flowers-like NiS nanostructure.

organic dyes MB under visible light irradiation.

2. Experimental

2.1. Synthesis of the NiS nanoflowers

In this study, all of the reactants were of analytical grade and were used directly without further purification. NiS nanoflowers were prepared via a facile polyol refluxing process. 1 mmol of four hydrated nickel acetate ($\text{Ni}(\text{CH}_3\text{COO})_2 \cdot 4 \cdot \text{H}_2\text{O}$, 98.5%, AR) and 3 mmol thiourea ($\text{CN}_2\text{H}_4\text{S}$, 99.0%, AR) were put into a three-neck flask (250 mL capacity) to which 50 mL ethylene glycol was added. After stirring for 10 min, all the reagents were dissolved, the reaction system was heated to 135 °C and kept at that temperature for 1 h under vigorous stirring in atmosphere. Finally, black precipitates were collected, washed with distilled water and absolute ethanol and then dried in a vacuum at 70 °C for 3 h.

2.2. Material characterization

The as-prepared sample was characterized by X-ray powder diffractometer (X'Pert PRO, PANalytical B.V., the Netherlands) with radiation of a Cu target ($K\alpha$, $\lambda = 0.15406$ nm) at 25 °C. The energy dispersive spectroscopy was achieved on a micro X-ray fluorescence system (EDX Inc.). The nanoelectrode morphologies were investigated by a JEOL JSM-6700F field emission scanning electron microscopy (FESEM). Transmission electron microscopy (TEM) images (JEOL, JEM-2010) were prepared to analyze the morphologies of the samples. The UV–vis absorption spectra were recorded on a Shimadzu UV-2550 spectrophotometer. Room-temperature photoluminescence (PL) spectra were recorded with an HORIBA Jobin Yvon LabRAM Spectrometer HR 800 UV with the excitation wavelength of 325 nm.

2.3. Photocatalysis

Photocatalytic decomposition of methylene blue (MB) was carried out in a cylindrical reaction vessel containing a suspension of 0.1 g NiS nanoflowers sample in a 100 mL MB or RhB solution (the dye concentration was 8 mg L⁻¹ in both cases). To get the adsorption–desorption equilibrium, the suspension sample was kept in dark for 2 h with continuous stirring. Then the system was irradiated by a 500 W Xe-lamp equipped with a cutoff filter ($\lambda > 420$ nm) and a water filter was used as the light source. At given time intervals, 3 mL of the reaction suspension was collected and filtrated to remove the NiS particles. The absorption spectra of a series of MB solutions were measured on a Shimadzu UV-2550 UV–vis spectrometer at room temperature.

3. Results and discussion

Fig. 1a displays the panoramic SEM image, indicating the formation of a large number of 3D flower-like architectures with the diameter of about 150–200 nm. Fig. 1b clearly exhibits that NiS nanoflowers are built from dozens of nanoflakes with the thickness of 5–10 nm. The formation of porous flowers is caused by the connections and overlaps of the adjacent nanoflakes. The little nanoflowers which were attracted by Van der Waals' force made up some nanoflowers with larger size. X-ray powder diffraction (XRD) was used to study the phase purity of the as-synthesized NiS nanoflowers as shown in Fig. 1c. The main reflection peaks can be indexed to the hexagonal phase of nickel monosulfide with lattice constants of $a = 3.425$ and $c = 5.340$ (JCPDS card No. 65–3419). Four main peaks ((100), (101), (102) and (110) planes) in the XRD pattern show wider full-widths at half-maxima than a bulk NiS [24]. In order to further indicate the lattice formation of the flower-like product, an energy dispersive spectroscopy (EDS) was detected

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