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Two-step hydrothermal synthesis of $\text{Sn}_2\text{Nb}_2\text{O}_7$ nanocrystals with enhanced visible-light-driven H_2 evolution activity

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ABSTRACT

We use a two-step hydrothermal method to successfully synthesize $\text{Sn}_2\text{Nb}_2\text{O}_7$ nanocrystals with an average size of approximately 20 nm. The as-obtained samples are characterized by powder X-ray diffraction, ultraviolet-visible diffuse reflectance spectroscopy, Brunauer-Emmett-Teller analysis, scanning electron microscopy, and transmission electron microscopy. The photocatalytic activity of the $\text{Sn}_2\text{Nb}_2\text{O}_7$ nanocrystals is evaluated by photocatalytic water splitting under visible light irradiation. The $\text{Sn}_2\text{Nb}_2\text{O}_7$ nanocrystals with a large surface area of 52.2 m^2/g show an enhanced visible-light-driven photocatalytic H_2 production activity, approximately 5.5 times higher than that of bulk $\text{Sn}_2\text{Nb}_2\text{O}_7$ powder. The higher photocatalytic activity of $\text{Sn}_2\text{Nb}_2\text{O}_7$ nanocrystals is mainly attributed to its relatively high dispersity of nanosized particles and larger specific surface area when compared with the bulk powder.

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1. Introduction

Photocatalytic water splitting using nanosized semiconductors is an ideal technology for converting the free and abundant solar energy into chemical energy [1–5]. Owing to their unique crystal and energy band structures, Nb-based semiconductors consisting of corner-sharing octahedral NbO_6 , which are beneficial for the transfer of photogenerated electrons and holes, have attracted much attention in various photocatalysis applications [6–10]. Importantly, the conduction band (CB) levels consist of an Nb 4d orbit, which is much more negative than a Ti 3d orbit, giving the photogenerated electrons a strong reducing capability [11–13]. Although many Nb-based semicon-

ductors that function efficiently under UV light have been developed, only a few visible light responsive Nb-based photocatalysts have been reported for photocatalytic water splitting, and further improvement of the photocatalytic water splitting activity is required [5,14–17]. Therefore, it is highly desirable to search for a novel Nb-based semiconductor nanocatalyst with improved visible-light-driven photocatalytic water splitting activity for practical applications.

$\text{Sn}_2\text{Nb}_2\text{O}_7$ with a pyrochlore structure has attracted particular research interest in visible-light-driven photocatalytic water splitting owing to its narrow band gap (approximately 2.4 eV), and proper CB and valence band (VB) potentials [18–20]. This material can absorb visible light because Sn^{II} with a $5s^2$ elec-

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tronic configuration contributes to the formation of a hybrid orbital with O 2p as a VB. However, the reported bulk $\text{Sn}_2\text{Nb}_2\text{O}_7$ powders (bulk SNO) obtained by the high-temperature solid state reaction method have shown unsatisfactory photocatalytic activities owing to its large-sized crystals with low specific surface areas [21,22]. Therefore, it is still a challenge to develop a facile yet effective method for preparing nanosized $\text{Sn}_2\text{Nb}_2\text{O}_7$ with enhanced photocatalytic activity for future applications.

Herein, we have developed a facile two-step hydrothermal method to prepare $\text{Sn}_2\text{Nb}_2\text{O}_7$ nanocrystals (SNO NCs) with an average size of approximately 20 nm. The photocatalytic activity of SNO NCs is evaluated by photocatalytic H_2 production from water using lactic acid as a sacrificial agent under visible light irradiation. SNO NCs show enhanced photocatalytic H_2 production activity when compared with bulk SNO powders owing to its relatively high dispersity of nanosized particles and larger specific surface area.

SNO NCs were synthesized by the facile two-step hydrothermal method shown in Scheme 1. First, a clear solution of soluble potassium niobate $\text{K}_8\text{Nb}_6\text{O}_{19} \cdot 10\text{H}_2\text{O}$ (KNO) was obtained after hydrothermal treatment of Nb_2O_5 powder (0.5 g) in 45 mL KOH solution (3 mol/L) at 200 °C for 12 h. Then, ethanol was added into the above KNO clear solution ($V_{\text{ethanol}}:V_{\text{KNO clear solution}} = 1:2$) to obtain a white precipitant, which reacted with SnCl_2 to produce light yellow SNO precursors by ultrasonication for 5 min under room temperature. Finally, the yellow SNO NCs were obtained by further hydrothermal treatment of the light yellow SNO precursors dispersed into a pure water solution (30 mL) at 200 °C for 12 h.

The morphology of the SNO NCs was characterized by scanning electron microscopy (SEM) and transmission electron microscopy (TEM). As shown in Fig. 1(a) and (b), SNO NCs with an average size of approximately 20 nm in a large scale were observed. From the high resolution transmission electron microscopy (HRTEM) image of a single nanoparticle (Fig. 1(c)), the spacing between the adjacent fringes was measured to be 0.61 nm, which corresponded to the (111) plane of cubic $\text{Sn}_2\text{Nb}_2\text{O}_7$. The successful fabrication of SNO NCs was further confirmed by X-ray diffraction (XRD) analysis. As demonstrated in Fig. 1(d), all of the diffraction peaks could be exactly indexed to the pure phase of $\text{Sn}_2\text{Nb}_2\text{O}_7$ (JCPDS 23-0593). No impurity peaks were detected. The calculated crystal size of SNO NCs was approximately 20 nm, according to the Scherrer Equation based on the peak at $2\theta = 29.2^\circ$, which indicated that pure SNO

Fig. 1. (a) SEM, (b) TEM, (c) HRTEM images and (d) XRD pattern of SNO NCs.

NCs could be successfully fabricated through a facile two-step hydrothermal process.

The band gap is known to play an important role in the efficient capture of solar energy in semiconductors. The band structures of the as-prepared SNO NCs and the bulk SNO powder were examined by UV-Vis diffuse reflectance spectroscopy. As shown in Fig. 2(a), both the SNO NCs and bulk SNO powder exhibited an obvious photoabsorption in the visible light region. Compared with the bulk SNO powder, the absorption edge of the SNO NCs exhibited an obvious blue-shift, which was ascribed to the nanosized effect [23–25]. The band gaps of the SNO NCs and the bulk SNO powder, calculated by a plot of $(ah\nu)^2$ versus photo energy, were 2.52 and 2.38 eV, respectively (Fig. 2(b)). Obviously, SNO NCs have a wider energy band gap compared with that of the bulk SNO powder. To detect the detailed band structures of SNO NCs, valence band X-ray photoelectron spectra (VB XPS) were recorded to determine the relative VB maximum. Compared with the bulk SNO powder, an obvious downshift for the VB edge of SNO NCs was observed, as shown in Fig. 2(c). Based on the VB XPS results and Fig. 2(b), the schematic band structures for SNO NCs and the bulk SNO powder are shown in Fig. 2(d). The CB and VB edges were calculated at -0.27 and 2.25 V for SNO NCs, and -0.73 and 1.65 V for the bulk SNO powder, respectively.

The specific surface area of photocatalysts is also crucial for their photocatalytic performance. N_2 adsorption-desorption measurements were performed to determine the specific surface areas of the as-obtained samples. Fig. 3 shows a typical IV isotherm with an H3-type hysteresis loop, which suggested the co-existence of mesopores and macropores in SNO NCs. The specific surface areas of SNO NCs was measured to be $52.2 \text{ m}^2/\text{g}$, which was much larger than that of the bulk SNO particles (approximately $2.3 \text{ m}^2/\text{g}$). It has been extensively reported that photocatalysts with a high specific surface area beneficial for photocatalysis [26–28]. Therefore, SNO NCs with a large

Scheme 1. Two-step hydrothermal synthesis of SNO NCs.

Fig. 2. (a) UV-Vis diffuse reflectance spectra, (b) plots of $(ah\nu)^2$ vs photon energy, (c) high resolution VB XPS spectra and (d) schematic band structures for SNO NCs and the bulk SNO powder.

surface area could exhibit a high visible-light-driven photocatalytic H_2 production activity.

The photocatalytic activity of SNO NCs was evaluated by photocatalytic H_2 production from lactic acid aqueous solutions under visible light irradiation ($\lambda > 400$ nm). The visible-light-driven photocatalytic H_2 evolution rate of SNO NCs without loading cocatalyst was $2.1 \mu\text{mol}/(\text{h}\cdot\text{g})$, which was approximately three times higher than that of the bulk SNO powder ($0.65 \mu\text{mol}/(\text{h}\cdot\text{g})$). The relatively lower photocatalytic H_2 evolution rates of both the samples were ascribed to the quick recombination of the photogenerated electrons and holes [20]. When Pt nanoparticles (NPs) as cocatalyst were loaded on the

surface of the samples by an *in-situ* photodeposition method, both samples showed an improved photocatalytic H_2 production activity. As shown in Fig. 4(a), the photocatalytic H_2 evolution rate of SNO NCs with 0.3 wt% loading of Pt was $82 \mu\text{mol}/(\text{h}\cdot\text{g})$ under visible light irradiation ($\lambda > 400$ nm), which was 5.5 times higher than that of the bulk SNO powder ($14.8 \mu\text{mol}/(\text{h}\cdot\text{g})$). The higher photocatalytic H_2 production activity of SNO NCs could be ascribed to its high dispersity of nanosized particles, larger specific surface area and more positive VB potential. First, the decrease in the particle size of SNO NCs caused a dramatically decreased migration distance of the photogenerated electrons and holes, which allowed photogenerated charge carriers to move rapidly to the surface to take part in the photocatalytic reaction [29–31]. Second, the specific surface area of SNO NCs was $52.2 \text{ m}^2/\text{g}$, which was much higher than that of bulk SNO particles ($2.3 \text{ m}^2/\text{g}$). It is well-known that a high surface area could increase the surface active sites and charge transfer of a semiconductor photocatalyst, which would result in the improvement of the photocatalytic activity [32,33]. Third, the more positive VB potential of SNO NCs could largely expedite the oxidation capability of holes (Fig. 2(c)), leading to an enhanced separation efficiency of photoinduced electron-hole pairs and thus improved photocatalytic H_2 -production activity [5,23]. All these favorable factors co-contribute to the exceptionally high photocatalytic performance of SNO NCs.

The proposed photocatalytic reaction mechanism of SNO NCs under visible light irradiation can be illustrated as follows. Under visible light irradiation, the VB electrons of SNO NCs are

Fig. 3. N_2 adsorption-desorption isotherms of SNO NCs.

Fig. 4. (a) Photocatalytic H₂ evolution of SNO NCs and bulk SNO powder with and without Pt loading under visible light irradiation ($\lambda > 400$ nm) in lactic acid aqueous solutions. (b) Time course of H₂ evolution over 0.3 wt% Pt loaded SNO NCs from lactic acid aqueous solutions under visible light irradiation ($\lambda > 400$ nm). Catalyst, 50 mg; H₂O, 16 mL; Lactic acid, 4 mL; 300 W Xe lamp with 400 nm UV cut-off filter.

excited to the CB, simultaneously leaving behind positive holes in the VB. Normally, these charge carriers quickly recombine and only a fraction of the electrons and holes participate in the photocatalytic reaction, which results in a poor photocatalytic activity. However, when SNO NCs were modified by Pt NPs, these photogenerated electrons on the CB of SNO NCs tend to transfer to Pt NPs, which leads to electron-hole separation and thus a higher photocatalytic H₂ production activity.

To demonstrate the applicability of SNO NCs in photocatalysis, a 24-h recycling experiment with intermittent evacuation every 6 h was performed under visible light irradiation ($\lambda > 400$ nm). After four consecutive cycles, the SNO NCs maintained an almost constant photocatalytic H₂ production rate without noticeable catalyst deactivation (Fig. 4(b)). The satisfying stability makes SNO NCs a good candidate for photocatalytic water splitting under visible light irradiation.

In conclusion, SNO NCs with an average size of approximately 20 nm were successfully prepared on a large scale through a two-step hydrothermal route. SNO NCs showed an improved visible-light photocatalytic H₂-production activity compared with the bulk SNO powder. The higher photocatalytic activity of SNO NCs was mainly attributed to its relatively high dispersity of nanosized particles and larger specific surface area when compared with the bulk powder. This work provides a novel synthesis method to develop nanosized Nb-based semiconductor photocatalysts with high photoactivity for solar energy conversion.

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Graphical Abstract

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Two-step hydrothermal synthesis of $\text{Sn}_2\text{Nb}_2\text{O}_7$ nanocrystals with enhanced visible-light-driven H_2 evolution activity

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A two-step hydrothermal route is used to synthesize $\text{Sn}_2\text{Nb}_2\text{O}_7$ nanocrystals with an average size of 20 nm. The as-obtained nanocrystals show enhanced visible-light-driven photocatalytic H_2 production activity compared with bulk $\text{Sn}_2\text{Nb}_2\text{O}_7$ powder.

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两步水热法合成 $\text{Sn}_2\text{Nb}_2\text{O}_7$ 纳米晶及其高效可见光分解水制氢性能周超^{a,b}, 施润^{a,b}, 尚露^a, 吴骊珠^a, 佟振合^a, 张铁锐^{a,b,*}^a中国科学院理化技术研究所, 中国科学院光化学转换与功能材料重点实验室, 北京100190^b中国科学院大学, 北京100049

摘要: 铌基半导体光催化材料因其具有独特的晶体结构和能带结构在光催化分解水制氢领域受到科研工作者的广泛关注。然而, 大多数铌基半导体光催化剂仅能够在紫外光驱动下实现光催化分解水制氢, 具有可见光响应的铌基半导体光催化剂不仅数量少而且活性较低, 因此发展新型纳米铌基半导体光催化剂并实现其高效可见光催化分解水产氢具有重要的学术和实用意义。具有烧绿石构型的 $\text{Sn}_2\text{Nb}_2\text{O}_7$ 材料由于具有较窄的禁带宽度(2.4 eV)和合适的导带和价带电势在可见光催化分解水制氢方面引起了科研人员广泛的兴趣。然而, 目前报道的利用高温固相法制备的块体 $\text{Sn}_2\text{Nb}_2\text{O}_7$ 材料由于颗粒尺寸较大和比表面积较小而导致光催化活性较差。因此, 发展一种简便高效的制备方法实现纳米 $\text{Sn}_2\text{Nb}_2\text{O}_7$ 材料的可控制备进而提高其可见光催化活性仍具有一定的挑战性。

我们发展了一种简便的两步水热合成方法实现了 $\text{Sn}_2\text{Nb}_2\text{O}_7$ 纳米晶的可控制备。扫描电镜和透射电镜测试结果表明, 通过两步水热法得到的 $\text{Sn}_2\text{Nb}_2\text{O}_7$ 纳米颗粒具有较好分散度, 其平均颗粒尺寸为20 nm。X射线衍射测试结果也进一步证明, 通过两步水热法可以实现 $\text{Sn}_2\text{Nb}_2\text{O}_7$ 纳米晶的可控制备。比表面积测试结果表明, $\text{Sn}_2\text{Nb}_2\text{O}_7$ 纳米晶的比表面积约为52.2 m²/g, 远远大于固相法制备的块体 $\text{Sn}_2\text{Nb}_2\text{O}_7$ 材料(2.3 m²/g)。大量研究表明, 大的比表面积有利于半导体催化材料催化活性的提升。通过考查所制备的 $\text{Sn}_2\text{Nb}_2\text{O}_7$ 纳米晶的可见光分解水制氢能力, 对其催化性能进行了评价。研究表明, 以乳酸为空穴消耗剂, 负载0.3wt.% Pt纳米颗粒作为助催化剂的 $\text{Sn}_2\text{Nb}_2\text{O}_7$ 纳米晶表现出优异的可见光催化分解水产氢性能, 其产氢速率是块体 $\text{Sn}_2\text{Nb}_2\text{O}_7$ 材料的5.5倍。 $\text{Sn}_2\text{Nb}_2\text{O}_7$ 纳米晶可见光催化分解水产氢性能提高的主要原因是其具有高分散度的纳米颗粒、较大的比表面积和更正的价带电势。首先, 颗粒尺寸的纳米化能够显著减小光生电子和空穴的迁移距离, 实现光生载

流子快速迁移到催化剂表面进而参与催化反应; 其次, 大的比表面积能够提供更多的催化活性位点, 进而有利于催化活性的提高; 最后, X射线光电子能谱测试表明, $\text{Sn}_2\text{Nb}_2\text{O}_7$ 纳米晶具有更正的价带电势, 研究表明, 价带电势越正, 其光生空穴氧化能力越强. 在光催化分解水制氢过程中, 具有较强氧化能力的光生空穴通过与空穴牺牲剂乳酸快速反应而被消耗掉, 抑制了光生电子与空穴的复合, 进而导致其具有较高的光催化产氢活性.

关键词: 水热合成; $\text{Sn}_2\text{Nb}_2\text{O}_7$; 光催化; 分解水; 可见光驱动

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