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A facile hydrothermal preparation of O-deficient BiNbO₄ nanorods for effective sonocatalytic decontamination

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Abstract: Niobium oxalate was employed to obtain $BiNbO_4$ for the first time and the $BiNbO_4$ nanorods were successfully synthesized through a hydrothermal method at a low temperature. Oxygen vacancies were introduced into the $BiNbO_4$ nanorods, lowering the $BiNbO_4$ bandgap. The O-deficient $BiNbO_4$ exhibited excellent sonocatalytic performance.

Keywords: Niobium oxalate; BiNbO4; Oxygen vacancies; Sonocatalytic

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1.Introduction

The ultimate goal in materials synthesis remains at the low-cost and effective route to prepare materials with tailored properties for large-scale applications [1-5]. In this regard, proper synthesis of nanometerials is more urgently demanded than their bulk forms because the nano-sized products exhibit better functionalities than the bulk ones [6-8]. A typical example is demonstrated by the preparation of nanostructured BiNbO₄. Due to multifunctionality and potentials in electronics and catalysis, its synthetic methods have been extensively studied [9-12]. However, prior arts suffer from two major difficulties [13-17]. High-temperature sintering or calcination in one-step preparation bars industrial product. Though reaching nanostructured BiNbO₄, the dedicated liquid-phase synthesis (e.g., sol-gel method, hydrothermal method) requires nano-sized particles as precursors, and then careful calcination during product formation stage. The complexity and precursor costs are unsuitable for mass-production of such kind of materials.

In this work, an environment-friendly and easy-handling method was invented to synthesize O-defective BiNbO₄ nanorods with excellent sonocatalytic properties. To our knowledge, this is the first time that niobium oxalate was introduced as the Nb source to synthesize BiNbO₄. Comparing to conventional Nb sources (Nb₂O₅, niobium (V) chloride, etc.) [18-20], it is advanced in ambient stability, aqueous dissolution, and easy dispersion in high temperature and high pressure conditions. The oxygen defective but highly crystallized BiNbO₄ nanorods are endowed with excellent ultrasonic catalytic abilities of degrading model dyes and solid pollutants in the presence of H₂O₂. A degradation rate of 99% has been reached for RhB under 30

minutes ultrasonic irradiation condition, and 99% for DNP after 90 minutes, denoting a superior catalytic ability over most binary composites as previously reported [21-23].

2. Experimental

2.1 Materials

Niobium oxalate was purchased from Ningxia Orient Tantalum Industry Co., Ltd. Bismuth nitrate pentahydrate and Nitric acid were provided by Shantou Xilong Science Co., Ltd. Absolute ethanol was brought from Hunan Jianghong Reagent Co., Ltd. All chemicals were of analytical grade and used without further purification.

2.2 Material synthesis

The hydrothermal synthesis process is performed as follows. 2.69 g Nb(HC₂O₄)₅ was dispersed in 20 mL deionized water. 4 mL of 2 M HNO₃ containing 2.43 g $Bi(NO_3)_3 \cdot 5H_2O$ solution was added dropwise in Nb(HC₂O₄)₅ solution under stirring. After 30 min of vigorous stirring, the solution was placed into an 80 ml autoclave and heated to 210 \Box for 20 h. The powder was obtained by centrifuging, and sequentially washed 6 times with distilled water. Finally, the precipitate was dried at 70 \Box for 48 h.

2.3 Measurement of sonocatalytic properties

The ultrasonic catalytic activity of as-prepared sample was investigated by ultrasonic degradation of RhB (frequency 40 kHz, power 200 W) after the adsorption–desorption equilibrium among the sonocatalyst and dye. In each test, 100 ml aqueous solution containing 10 mg/L of RhB and 50 mg of BiNbO4 were kept in dark under continuous stirring for 30 min. Then the suspension was irradiated under ultrasound for different time in the dark. 3 ml of the suspension was sampled at pre-

set times and centrifuged immediately to remove the sample. Then the supernatant was transferred into a clean tube. RhB was analyzed using a UV-vis spectrophotometer.

3. Results and discussion

The x-ray diffraction (XRD) pattern is shown in Fig. 1a to study microstructures of the as-prepared material. The product owns orthorhombic (JCPDS 82-0348) and triclinic (JCPDS 16-0486) phases. Diffraction peaks at 24.7°, 31.5°, 48.4°, 54.1°, 58.8° and 60.2° are indexed to the orthorhombic phase (a=5.703 Å, b=11.780 Å, c=5.099 Å, $\alpha=\beta=\gamma=90^{\circ}$). Peaks at 11.7°, 45.1° and 46.7° are assigned to (100) (104) and (004) lattice plane of the triclinic BiNbO₄ phase. Peaks at 23.8°, 28.4° and 36.0° can be assigned to the common peaks of the orthorhombic and the triclinic phase [24-26]. No other characteristic peaks were detected from the Nb₂O₅, Bi₂O₃, or Bi₂O₂CO₃. Thus, the synthetic compound is high-purity BiNbO₄ but containing two phases. The appearance of both phases may be due to the selection of niobium oxalate as the source of niobium, which decomposes with nitric acid at high temperature and high pressure. The Bi-O and Nb-O bonds are preferentially formed on the X axis, enabling the cubic and triclinic phases generate at the same time.



Fig. 1. (a) XRD pattern.(b) XPS spectras of O 1s and (c) EPR spectra of the sample. (d) Photoluminescence spectra of as-prepared $BiNbO_4$ sample and no O-deficient $BiNbO_4$.

The elemental chemical states were researched via XPS. The O 1s spectra was shown in Fig. 1b while the full survey in Fig. S1, and other elements in S2-S3. Two peaks were found for O in Fig. 1b, the one at 529.9 eV is the characteristic feature from the oxygen in the BiNbO₄ lattice, with another at 532.3 eV from the over charged O induced by oxygen vacancies (V_0) [27-30]. Two peaks at 206.8 and 209.6 eV in Fig. S2 corresponded to $3d_{5/2}$ and $3d_{3/2}$ of Nb⁵⁺ ions. The binding energies of Bi $4f_{7/2}$ and $4f_{5/2}$ in Fig. S3 are 159.1 eV and 164.4 eV, in accordance to the +3 state of the bismuth [31-32]. The electron paramagnetic resonance (EPR) spectroscopy in Fig. 1c provides direct evidence for the existence of oxygen vacancies. The obvious signal of g value at 2.004 arises from V_0 s, in line with the XPS results. Fig. 1d depicts the photoluminescence (PL) spectrum of the as-prepared sample to study electron deexcitation processes in the sample. The PL intensity of the as-prepared BiNbO₄

sample is much lower than that without V_os as reported in [20]. This is due to the O vacancies trap photoexcited electrons, delay electron-hole pair recombination, and subsequently reduce PL intensities [33-35].

The morphology of the synthetic catalyst was investigated by TEM and the images are shown in Fig. 2a, b. The sample is consisted of nanorods with an average diameter of about 20 nm, and a varied length from about 50 nm to 200 nm. The high-resolution TEM (HRTEM) images clearly show the (121) (200) lattice planes of orthorhombic phase in Fig. 2c and (100) lattice plane of triclinic phase in 3d. The orthorhombic phase and triclinic phase coexist in the as-prepared sample. The microscopic result is in line with XRD analysis, further confirming the coexistence of mixed crystal phases in the as-prepared BiNbO₄. Moreover, the clear lattice fringes in HRTEM images demonstrate high crystallinity of the synthetic nanorods.

Though the as-prepared BiNbO₄ coexists two crystal phases, the morphology of nanorod structure was uniform and evenly distributed. The formation mechanism of BiNbO₄ nanorods was inferred as follows: because of the aquatic insolubility, $Bi(NO_3)_3 \cdot 5H_2O$ was dispersed in the solution under vigorous stirring in the form of tiny particles. Niobium oxalate is water-soluble, which can be definitely dissolved in the solution. Niobium oxalate molecules evenly surrounded at and diffuse into the Bi(NO₃)₃ particles. The reagents were fully contacted. Later the nitrate and oxalate were decomposed under high temperature and high pressure. Bonds of Bi-O and Nb-O were preferable formed in the x-axis direction. The morphology gradually evolved from particles to nanorods, while composition from Bi(NO₃)₃ to BiNbO₄. The formation of tiny BiNbO₄ nanorods is conducive to the exposure of active sites, which can effectively enhancing the catalytic activities in decomposing the organics.



Fig. 2. (a)-(b) TEM images and (c)-(d) HRTEM images of the sample.

Fig. S4 shows the UV-vis spectrum of the synthetic BiNbO₄. The absorption edge of the synthetic BiNbO₄ is around 496 nm corresponding to a band gap of 2.50 eV. It is slightly narrower than that in the previous report (2.81 eV) [36]. This is probably due to modified valance band structures (indirecity shown in PL spectrum at Fig. 1) induced by V_0 s in the BiNbO₄, subsequently causing the band gap narrowing [37-39]. The narrowed band gap is much more favorable for the absorption of light and the separation of electron-hole pair.

The ultrasonic catalytic performance over pure BiNbO₄ was investigated. Fig. 3 presents the UV-vis absorption spectrums of dye after ultrasonic irradiation under different conditions. As shown in Fig. 3a, after achieving the equilibrium of adsorption-desorption, pure BiNbO₄ exhibited low adsorption efficiency (10%) at the end of 30 min of ultrasonic irradiation. While after adding 0.5 ml 30% H_2O_2 into the

aqueous solution, the degradation efficiency of RhB could be significantly improved to 99% in the same time. To compare the effect of H_2O_2 on RhB degradation, the blank group without catalyst was tested under the same condition. The result suggested that the degradation efficiency of RhB is 23%. The degradation efficiency was largely improved when BiNbO₄ combines with H_2O_2 . In addition, the degradation of micro-molecule organic compound (2,4-dinitrophenol) was investigated to evaluate ultrasonic catalytic activity of the product. The results were shown in Fig. 3b. High degradation efficiency (99%) from BiNbO₄ with 0.5 ml 30% H_2O_2 can be achieved after 90 min ultrasonic irradiation. Similarly, the two contrast tests, one without H_2O_2 and the other one without catalytic, were conducted in identical conditions. Only 46% and 27% degradation efficiencies were achieved respectively. These results demonstrate the versatility of the as-prepared sample in decomposing organics. The recycle ultrasonic catalytic tests of RhB were studied to prove the reusability of the as-prepared catalyst. The results (Fig. S5) showed that the degradation efficiency maintain a high level after 5 cycle.

The photocatalytic performance over pure BiNbO₄ was also evaluated to compare with sonocatalytic performance. Experimental condition: 50 mg sample, 100 ml 10 mg/L RhB, 0.5 ml 30% H_2O_2 , 500 W Xe lamp. Test procedures were the same as the ultrasonic irradiation. The results were presented in Fig. S6. The degradation efficiency of RhB after 30 min visible light irradiation (500 W) achieved 85%. While the degradation efficiency reached to 99% under ultrasound (40 kHz, 200 W) irradiation in the same condition.



Fig. 3. UV-vis absorption spectrums of (a) RhB (ultrasound irradiation 30 min) and (b) 2, 4-dinitrophenol (ultrasound irradiation 90 min).

We also investigate the predominant active radicals and the degradation mechanism of as-prepared BiNbO₄ sample under ultrasonic irradiation. The ascorbic acid (AA), isopropyl alcohol and EDTA-2Na were used as $\cdot O_2^-$, $\cdot OH$ and h+ scavengers during the Rhodamine B degradation process [40-41]. As shown in Fig. S7, the addition of IPA has littile impact on the ultrasonic catalysis, suggesting that $\cdot OH$ be not the main active species for oxidizing the organics. On the contrary, the degradation efficiencies (24% and 34%, respectively) are significantly inhibited after the addition of ascorbic acid and EDTA-2Na. This confirms the effective role sof $\cdot O_2^-$ and h⁺ in the degradation process.

The possible degradation mechanism over the BiNbO₄ in the presence of H₂O₂ was proposed in Fig. 4. The light induced by ultrasonic cavitation leads to electron transition within the nanorodes. Oxygen vacancies adsorb O₂ and meanwhile trap electrons subjected to delayed electron-hole pair separation after ultrasonic cavitation excitation of electrons. The V_os provide active sites to host the chemical reaction of $O_2 + e^- \rightarrow \cdot O_2^-$. The superoxide radicals oxidize and decompose the RhB to CO₂ and H₂O as final produts. Meanwhile ultrasound-produced holes also directly react with

RhB and transfer RhB to CO_2 and H_2O . During the sonocatalysis, the H_2O_2 can act as initiator in the oxidative degradation process. It generates O_2 to start the radical reaction in a short time and circularly formed in the ultrasonic solution system [42-43]. The reaction path is described as follows:

$$H_2O \xrightarrow{Sono} \cdot H^+ \cdot OH$$

 $H_2O^+h^+ \longrightarrow \cdot H^+ \cdot OH$
2 ·OH → H_2O_2
 $O_2^+ 2H^{++}2e^- \longrightarrow H_2O_2$
 $2H_2O_2 \longrightarrow O_2^+ 2H_2O$



Fig. 4. Mechanism of ultrasonic catalytic degradation of RhB over the sample.

4. Conclusion

In summary, by using water-soluble niobium oxalate as the Nb source, we realized a facile synthesis of BiNbO₄ nanrods via one-step hydrothermal method. The

formation of oxygen vacancies could improve the efficiency of electron-hole separation due to the narrowed band gap and the trap of electrons. The nanorod structure serves large amounts of active sites on the surface of BiNbO₄. These result in efficient catalytic degradations of organic pollutants under ultrasonic and light irradiation, with the sonic one superior. Besides paving the route for mass-production of one inorganic catalyst, the work is hoped to inspire facile preparations of similar groups of compounds for practical catalysis in industrial scale.

Declaration of Competing Interest

The authors declare no conflict of interest.

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Appendix A. Supplementary data

Supplementary material related to this article can be found online at

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Declaration of interests

 \boxtimes The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

□The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:

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