# Preparation of Bi<sub>2</sub>WO<sub>6</sub> nanoplates modified by Nd doping photocatalyst for rhodamine B dye degradation

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0-3% Nd-doped  $Bi_2WO_6$  nanoplates were synthesized via hydrothermal method. The  $Bi_2WO_6$  without and with Nd doping can be indexed to the single phase of orthorhombic  $Bi_2WO_6$  nanoplates with orientation growth in the along the [001] projection by XRD, SEM and TEM analysis. The 2% Nd-doped  $Bi_2WO_6$  nanoplates shows the maximum photocatalytic activity under visible light irradiation owing formation of oxygen vacancies as recombination centers with decreased rate of recombination carries charge pairs after Nd doping. Furthermore, the radical scavenger of RhB degradation over 2% Nd-doped  $Bi_2WO_6$  nanoplates was analyzed to discuss the mechanism of the photodegradation possible in this research.

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### 1. Introduction

The photocatalytic technology as green technology using semiconductor photocatalysts such as ZnO [1, 2], TiO<sub>2</sub> [3-5], WO<sub>3</sub> [6, 7] and etc. is the effectively promising strategies with utilization of photon energy for environmental pollution treatment and water splitting into H<sub>2</sub> and O<sub>2</sub> [1, 3, 4, 7-9]. Among various semiconductors, TiO<sub>2</sub> (E<sub>g</sub> = 3.2 eV) as a photocatalyst has been the most widely studied because of its excellent photochemical reaction, reusability, inexpensive and non-toxicity [3, 4, 10, 11]. However, its photocatalytic application was limited due to its active only UV light (~5% of solar light) and high rate of photoinduced carriers recombination [3, 5, 10, 11].

 $Bi_2WO_6$  ( $E_g$  of 2.8 eV) is an Aurivillius-phase perovskite layers which shows the excellent visible-light-driven photocatalytic application because of its excellent intrinsic layer crystal structure, electron transmission efficiency and non-toxicity [11-16]. The practical photocatalytic application of  $Bi_2WO_6$  is low performance because of its low quantum efficiency and high photoinduced carriers recombination [12, 13, 15, 17]. The rare earth element doping such as Er [15], La [17, 18], Eu [19], Pr [20] and Dy [21] was selected to increase the photocatalytic

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performance of  $Bi_2WO_6$  because of the unique electronic configuration in un-filled in 4f orbital in rare earth elements which suppress the photoinduced carriers recombination and extend the absorption in visible region of  $Bi_2WO_6$  [15, 18, 22]. The Nd element as lanthanide metal was doped in  $Bi_2WO_6$  because of the ionic radius of Nd<sup>3+</sup> (0.983 Å [23-25]) is closely the ionic radius of  $Bi^{3+}$  (1.03 Å [15, 18, 19, 26, 27]) which is easy substituted  $Bi^{3+}$  ions in  $Bi_2WO_6$  lattices. Moreover, the photoinduced carriers separation of semiconductor increases after Nd doping which suppress the photoinduced carriers recombination, increasing the photocatalyst efficiency of semiconductor [22, 28].

In this paper, visible-driven Nd-doped  $Bi_2WO_6$  photocatalyst for rhodamide B (RhB) degradation was synthesized by hydrothermal method and characterized the phase, atomic vibration, morphologies, surface area and optical properties of products. The 2%Nd-doped  $Bi_2WO_6$  shows an outstanding RhB degradation under visible light illumination.

#### 2. Experiment

The 100 ml of 0.01 mole of Bi(NO<sub>3</sub>)<sub>3</sub>.5H<sub>2</sub>O and 0.005 mole of Na<sub>2</sub>WO<sub>4</sub>.2H<sub>2</sub>O solutions were mixed and stirred together. After that, the 0–3 mol% Nd(NO<sub>3</sub>)<sub>3</sub>.6H<sub>2</sub>O was mixed into the precursor solution and adjusted the pH = 6 using 3 M NaOH under vigorously stirred. It was sealed into a 200 mL hydrothermal autoclave reactor and transferred to the electric oven. After that, it was heated at 200 °C for 20 h. Then, the products were collected, washed and dried.

To investigate the photocatalytic activities, the 0.2 g of Bi<sub>2</sub>WO<sub>6</sub> with different weight of Nd loading was vigorously stirred into 200 mL of 1 x 10<sup>-5</sup> M RhB solution in the dark condition for 30 min and irradiated the visible light ( $\lambda \ge 420$  nm). Then, it was withdraw 5 ml RhB at different time intervals. Then, the RhB suspension was centrifuged and analyzed the absorption of RhB at 554 nm through a UV–vis spectrophotometer.

#### 3. Results and discussion

XRD pattern of pure Bi<sub>2</sub>WO<sub>6</sub> sample without Nd doping (Fig. 1a) presents the diffraction (131), (002), (202), (133), (262) and (400) planes at  $2\theta = 28.38^{\circ}$ ,  $32.89^{\circ}$ ,  $47.19^{\circ}$ ,  $55.95^{\circ}$ ,  $58.62^{\circ}$  and  $68.77^{\circ}$  of the orthorhombic Bi<sub>2</sub>WO<sub>6</sub> structure with according to the JCPDS no. 39-0256 [29] as standard. After Nd doping, the dominant diffraction crystal of (131) plane of Nd-doped Bi<sub>2</sub>WO<sub>6</sub> was slightly shifted to higher 2 $\theta$  as shown in Fig. 1b because the ion radius of Nd<sup>3+</sup> (0.983 Å [23-25]) is smaller ion than radius of Bi<sup>3+</sup>(1.03 Å [15, 18, 19, 26, 27]). It suggests that the Nd ions were substituted Bi ions in Bi<sub>2</sub>WO<sub>6</sub> lattices which lead the distorted in orthorhombic Bi<sub>2</sub>WO<sub>6</sub> structure. XRD pattern of 3% Nd-doped Bi<sub>2</sub>WO<sub>6</sub> shows the diffraction peaks of orthorhombic phase of Bi<sub>2</sub>WO<sub>6</sub> at  $2\theta = 28.43^{\circ}$ ,  $32.96^{\circ}$ ,  $47.24^{\circ}$ ,  $56.09^{\circ}$ ,  $58.69^{\circ}$  and  $69.09^{\circ}$  for (131), (002), (202), (133), (262) and (400) planes, respectively. The phase and structure of Bi<sub>2</sub>WO<sub>6</sub> after Nd doping are still orthorhombic Bi<sub>2</sub>WO<sub>6</sub> phase, indicating that the Nd ions were not change the orthorhombic Bi<sub>2</sub>WO<sub>6</sub> structure.



Fig. 1 XRD patterns at  $2\theta$  of (a)  $20^{\circ}-80^{\circ}$  and (b)  $27^{\circ}-33^{\circ}$  of 0-3% Nd-doped  $Bi_2WO_6$ .



Fig. 2 (a) FTIR spectra at wavenumber of 400-4000 cm<sup>-1</sup> and (b) Raman spectra at wavenumber of 100-1000 cm<sup>-1</sup> of  $Bi_2WO_6$  and Nd-doped  $Bi_2WO_6$ .

FTIR spectra of  $Bi_2WO_6$  with different weight of Nd loading show in Fig. 2a. FTIR spectrum of  $Bi_2WO_6$  without Nd doping at 729 and 818 cm<sup>-1</sup> are corresponding to the W–O stretching and W–O–W bridging vibrations in WO<sub>6</sub> octahedrons [10, 11, 18, 21, 30, 31]. The Bi–O stretching in BiO<sub>6</sub> octahedrons of Bi<sub>2</sub>WO<sub>6</sub> lattices showed at 581 cm<sup>-1</sup> [10, 11, 18, 21, 30, 31]. Comparing to the Bi<sub>2</sub>WO<sub>6</sub>, the wavenumbers of metal-oxygen vibration in Nd-doped Bi<sub>2</sub>WO<sub>6</sub> were slightly shifted due to the changing the metal-oxygen bonding by replacement of Nd ions in Bi<sub>2</sub>WO<sub>6</sub> lattices. However, the vibration of OH stretching from moisture on sample surface in Bi<sub>2</sub>WO<sub>6</sub> and Nd-doped Bi<sub>2</sub>WO<sub>6</sub> was detected at 3200-3400 cm<sup>-1</sup> [10, 11, 18, 21].

Raman spectra of  $Bi_2WO_6$  with different weight of Nd loading show in Fig. 2b. The dominant Raman peak in  $Bi_2WO_6$  at 793 cm<sup>-1</sup> was ascribed to the vibration of antisymmetric bridging mode of O–W–O bridging [11, 21, 32, 33]. The symmetric and anti-symmetric of terminal O–W–O bridging were at 826 and 712 cm<sup>-1</sup> [11, 21, 32, 33]. Moreover, the minor Raman peak at 306 cm<sup>-1</sup> is corresponding to the simultaneous motion of Bi<sup>3+</sup> and WO<sub>6</sub> translation mode [11, 21, 32, 33]. The external vibrations in WO<sub>6</sub> octahedron was detected at 153 cm<sup>-1</sup> [11, 21, 32, 33]. In addition, Raman spectra of Nd-doped Bi<sub>2</sub>WO<sub>6</sub> become broad with increasing the weight of Nd doping.

SEM images of  $Bi_2WO_6$  without Nd loading (Fig. 3a) show the agglomerated dense flower in shape assembled by nanoplates. After Nd doping, the morphologies of Nd-doped  $Bi_2WO_6$  (Fig. 3b-d) were composed of the self-assemble agglomerated loosely nanoplates to building up the flower-like structure with same  $Bi_2WO_6$ . The N<sub>2</sub> adsorption and desorption isotherms of  $Bi_2WO_6$  and 2% doped  $Bi_2WO_6$  samples (Fig. 4) are corresponding to the type IV isotherms, suggesting that the all samples are mesoporous materials [21, 34, 35]. The specific active surface areas of  $Bi_2WO_6$  and 2% doped  $Bi_2WO_6$  samples were 14.52 and 37.12 m<sup>2</sup>.g<sup>-1</sup>, respectively. The 2% Nd-doped  $Bi_2WO_6$  sample shows the highest specific active surface areas which improved the dye molecules adsorption on active surface areas of photocatalyst, enhancing the photocatalytic reaction [8, 21, 32, 33, 35].



Fig. 3 SEM images of (a) Bi<sub>2</sub>WO<sub>6</sub>, (b) 1% Nd-doped Bi<sub>2</sub>WO<sub>6</sub> (c) 2% Nd-doped Bi<sub>2</sub>WO<sub>6</sub> and (d) 3% Nd-doped Bi<sub>2</sub>WO<sub>6</sub>.



Fig. 4. N<sub>2</sub> adsorption and desorption isotherms of Bi<sub>2</sub>WO<sub>6</sub> and 2% Nd-doped Bi<sub>2</sub>WO<sub>6</sub> samples.

TEM images of Bi<sub>2</sub>WO<sub>6</sub> and 2% Nd-doped Bi<sub>2</sub>WO<sub>6</sub> as shown in Fig. 5a and 5c reveal uniform nanoplates in shape with size of 100-500 nm for Bi<sub>2</sub>WO<sub>6</sub> and 100-200 nm for 2% Nd-doped Bi<sub>2</sub>WO<sub>6</sub>, suggesting that the Nd doping was suppressed the nucleation and growth of Bi<sub>2</sub>WO<sub>6</sub> nanoplates. HRTEM images of Bi<sub>2</sub>WO<sub>6</sub> and 2% Nd-doped Bi<sub>2</sub>WO<sub>6</sub> nanoplates were corresponding to the (200) and (020) planes of orthorhombic Bi<sub>2</sub>WO<sub>6</sub> structure, suggesting that they were orientation growth in along the [001] projection due to the building up the alternate layer structure between WO<sub>6</sub> octahedral layer and Bi<sub>2</sub>O<sub>2</sub> layer of crystal orthorhombic Bi<sub>2</sub>WO<sub>6</sub> nanoplates as shown in Fig. 5b and 5d show the bright symmetric square electron diffraction pattern, suggesting that the samples were high crystallinity in nature. They can be indexed to (020), (220) and (200) crystal planes with zone axis of [002] of orthorhombic Bi<sub>2</sub>WO<sub>6</sub> phase.



Fig. 5 TEM images, HRTEM images and SAED patterns of (a and b) Bi<sub>2</sub>WO<sub>6</sub> and (c and d) 2% Nd-doped Bi<sub>2</sub>WO<sub>6</sub>.



Fig. 6 (a) UV-vis DRS spectra in wavelength of 200-800 nm and (b) Tauc's plots for  $E_g$  calculation of 0-3% Nd-doped Bi<sub>2</sub>WO<sub>6</sub> samples.

UV-vis DRS spectrum of pure Bi<sub>2</sub>WO<sub>6</sub> nanoplates (Fig. 6a) shows the strong UV-visible absorption at 320–450 nm which is according to the transition from the hybridized Bi 6s and O 2p states as valance band to W 5d states as conduction band of Bi<sub>2</sub>WO<sub>6</sub> [19, 36, 38]. It found that the Nd-doped Bi<sub>2</sub>WO<sub>6</sub> samples shows the higher strong visible absorption than pure Bi<sub>2</sub>WO<sub>6</sub> sample which may be the transition of impurity energy level and oxygen vacancies in Bi<sub>2</sub>WO<sub>6</sub> after Nd doping [14, 15, 18, 19, 22, 37]. According to the previous reports, the formation of donor level in Bi<sub>2</sub>WO<sub>6</sub> was occurred after Y<sup>3+</sup> doping which increase the visible absorption of Bi<sub>2</sub>WO<sub>6</sub> with different weight of Nd loading can be calculated using Tauc equation [15, 28, 30-32, 40]. According to the linear extrapolation, the E<sub>g</sub> of 0%, 1%, 2% and 3% Nd-doped Bi<sub>2</sub>WO<sub>6</sub> were 3.39, 3.36, 3.33 and 3.31 eV, respectively.



Fig. 7 (a) photocatalytic performances and (b) Langmuir–Hinshelwood kinetic models of RhB degradation over Nd-doped Bi<sub>2</sub>WO<sub>6</sub> under visible light radiation.

Fig. 7a shows the photocatalytic performances of RhB degradation in present of asprepared  $Bi_2WO_6$  with different weight of Nd loading samples under visible light illumination. The all Nd-doped  $Bi_2WO_6$  samples as photocatalyst show the higher photocatalytic performance of RhB degradation than  $Bi_2WO_6$  nanoplates. Among them, the photocatalytic performance of RhB degradation under visible light illumination was increased form 66.91% for  $Bi_2WO_6$  nanoplates to 93.35% for 2% Nd-doped  $Bi_2WO_6$  naoplates within 240 min due to the higher visible light harvesting, formation of oxygen vacancies and more active surface area of Nd-doped  $Bi_2WO_6$ nanoplates [8, 11, 15, 21, 28, 32]. In addition, the Nd doping act as electron scaverger to decreased rate of recombination carries charge pairs [22, 28, 41, 42]. After that, the photocatalytic performance of RhB degradation was decreased to 80.38% for 3% Nd-doped Bi<sub>2</sub>WO<sub>6</sub> within 240 min under visible light illumination due to the excess Nd dopant acts as recombination center trap [43, 44]. Among them, the 2% Nd-doped  $Bi_2WO_6$  shows the highest photocatalytic activity of RhB degradation which is higher than 1.40 time than Bi<sub>2</sub>WO<sub>6</sub> nanoplates under the same condition. The Langmuir-Hinshelwood kinetic model of RhB degradation in present of Bi<sub>2</sub>WO<sub>6</sub> without and with Nd was calculated as shown in Fig. 7b [1, 10, 11, 19, 28. 31, 45, 46]. The photocatalytic kinetic reaction value of samples was calculated from linear slope relation between  $\ln(C_0/C_t)$  and irradiation time (t) which was 4.48 x 10<sup>-3</sup>, 5.27 x 10<sup>-3</sup>, 0.0101 and 6.28 x 10<sup>-3</sup> min<sup>-1</sup> for 0%, 1%, 2% and 3% Nd-doped Bi<sub>2</sub>WO<sub>6</sub>, respectively. Among them, the apparent rate constant of 2% Nd-doped Bi<sub>2</sub>WO<sub>6</sub> is highest because of Nd dopant acts as electron trapping which effectively the separate photoinduced carriers and improve the absorption in visible light region [15, 21, 22, 28, 41, 42]. Moreover, it was confirmed that the photocatalytic performance of RhB degradation was still remains at 89.32% after 5<sup>th</sup> recycling runs as shown in Fig. 8a, indicating that the 2% Nd-doped Bi<sub>2</sub>WO<sub>6</sub> as photocatalyst is a reusable photocatalytic application in long term.



Fig. 8 (a) The photocatalytic stability for recycles photocatalytic reaction for five times and (b) photocatalytic efficiency of RhB degradation over Nd-deped Bi<sub>2</sub>WO<sub>6</sub> under visible light radiation after EDTA-2Na, IPA and BQ adding.

The benzoquinone (BQ) trap the superoxide anions ( $^{\circ}O_2^{-}$ ) trapping, iso-propanol (IPA) for hydroxyl radicals ( $^{\circ}OH$ ) trapping and ethylenediaminetetraacetic acid disodium salt (EDTA-2Na) for holes ( $h^+$ ) trapping were added in during the photocatalytic reaction for the photocatalytic mechanism study [10, 11, 14, 15, 21, 31, 33, 47, 48]. Fig. 8b shows the photocatalytic performance of RhB degradation over 2% Nd-doped Bi<sub>2</sub>WO<sub>6</sub> nanoplates with different scavenger regents adding. It presents that the RhB degradation over 2% Nd-doped Bi<sub>2</sub>WO<sub>6</sub> nanoplates was suppressed after BQ and EDTA-2Na adding to 45.98% and 28.39% within 240 min under visible light illumination while the RhB degradation over 2% Nd-doped Bi<sub>2</sub>WO<sub>6</sub> nanoplates was slightly decreased to 79.32% after IPA adding, implying that  $^{\circ}O_2^-$  and  $h^+$  are the main active species for RhB degradation in present of 2% Nd-doped Bi<sub>2</sub>WO<sub>6</sub> nanoplates under visible light illumination.

## 4. Conclusions

In summary, 2% Nd-doped  $Bi_2WO_6$  nanoplates were the orthorhombic  $Bi_2WO_6$  nanoplates orientation growth in along the [001] projection by hydrothermal synthesis. The 2% Nd-doped  $Bi_2WO_6$  nanoplates show the highest RhB photodegradation under visible light illumination due to higher visible light harvesting, formation of oxygen vacancies and more active surface area of Nd-doped  $Bi_2WO_6$  nanoplates, enhancing the photodegradation efficiency.

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