Temperature-dependent preparation of bismuth pyrostannate $\text{Bi}_2\text{Sn}_2\text{O}_7$ and its photocatalytic characterization

Nguyen Dang Phu$^a$, Pham Khac Vu$^a$, Dang Duc Dung$^b$, Do Danh Bich$^a$, Le Mai Oanh$^a$, Luc Huy Hoang$^a$, Nguyen Van Hung$^a$, Pham Van Hai$^{a,*}$

$^a$Faculty of Physics, Hanoi National University of Education, 136 Xuan thy, Cau giay, Hanoi, 10000, Viet Nam
$^b$School of Engineering Physics, Hanoi University of Science and Technology, 01 Dai Co Viet Road, Hanoi, 10000, Viet Nam

HIGHLIGHTS

- $\text{Bi}_2\text{Sn}_2\text{O}_7$ nanoparticles were synthesized via a fast microwave-assisted method.
- The optimal treatment temperature for enhance photocatalytic activity is at 500°C.
- The RhB degradation process is dominated by the contribution from N-deethylation.
- A better separation of charge carriers leads to enhanced photocatalytic activity.

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ABSTRACT

$\text{Bi}_2\text{Sn}_2\text{O}_7$ nanoparticles were successfully synthesized via a fast microwave-assisted method and high temperature treatment. The obtained nanoparticles were characterized using X-ray diffraction (XRD), thermogravimetric analysis (TGA), differential scanning calorimetry (DSC), scanning electron microscopy (SEM), UV-Vis diffuse reflectance spectroscopy (DRS), photoluminescence spectroscopy (PL), total organic carbon (TOC) measurement and Raman spectroscopy. We found that there is an optimal thermal treatment temperature for $\text{Bi}_2\text{Sn}_2\text{O}_7$ for the highest photocatalytic activity in the decomposition of rhodamine B (RhB) under visible light irradiation. We observed an improvement in activity when the thermal treatment temperature increases from room temperature to 500°C, but the activity decreases when the sintering temperature is increased further. The reason behind the enhanced photocatalytic efficiency is a better separation of the charge carriers which is competitive with the specific surface area, crystallinity and crystal size.

1. Introduction

Semiconductor-based photocatalysis is an active field of research due to its high-efficiency low-cost and non-toxic application in organic pollutants degradation [1,2]. Among a great number of photocatalytic materials, bismuth-based semiconductors, for example, $\text{Bi}_2\text{O}_3$, $\text{Bi}_2\text{WO}_6$, $\text{BiVO}_4$ and $\text{Bi}_2\text{Sn}_2\text{O}_7$, are promising new candidates for visible-light responsive photocatalysts [3]. Wu et al. [4], investigated for the first time the photocatalytic activity of $\text{Bi}_2\text{Sn}_2\text{O}_7$ nanomaterials in the decomposition of methyl orange (MO) under both UV and visible-light irradiation. The authors indicated that a highly-dispersed band structure ($s$ and $p$ orbitals) leads to a high mobility of the photoinduced charges which therefore enhances the photocatalytic performance. In addition, the origin of the improved performance was also attributed to a larger surface area of nanoparticle samples compared to that of bulk samples. Tian et al. [5] later have studied the degradation of arsenite under visible-light irradiation of $\text{Bi}_2\text{Sn}_2\text{O}_7$ and proposed a possible mechanism for arsenite oxidation. $\text{Bi}_2\text{Sn}_2\text{O}_7$ also displays strong photocatalytic activity for photodegradation of various pollutants, including methane [6], rhodamine B (RhB) [7,8], phenol [8] and isobutene [9–11].

$\text{Bi}_2\text{Sn}_2\text{O}_7$ is known to occur in three polymorphs, depending on temperature [12,13]. The α–phase (monoclinic) is thermodynamically stable at room temperature up to 90°C, while β-$\text{Bi}_2\text{Sn}_2\text{O}_7$ (face-centered cubic) is found between 90°C and 680°C. At temperature higher than 680°C, $\text{Bi}_2\text{Sn}_2\text{O}_7$ exists in the γ-phase that belongs to ideal (cubic) pyrochlore structure [12]. However, a variety of recent studies of $\text{Bi}_2\text{Sn}_2\text{O}_7$ nanoparticles prepared by the hydrothermal method [4,8,14].
have indicated that γ-Bi$_2$Sn$_2$O$_7$ becomes favored even at low temperatures (180–240°C). Furthermore, despite numerous experimental reports using the hydrothermal method to prepare Bi$_2$Sn$_2$O$_7$, to our knowledge there is no report on the Bi$_2$Sn$_2$O$_7$ preparation using a microwave-assisted method. Here, we have employed a fast microwave-assisted route to synthesize Bi$_2$Sn$_2$O$_7$ in a wide range of temperature. The samples obtained were characterized and the photocatalytic performance was investigated by the decomposition of RhB under visible light. The result have shown that a sample annealed at 500°C exhibited the best photocatalytic activity in comparison with the remaining ones. The Bi$_2$Sn$_2$O$_7$ photocatalyst displays good stability over test cycles.

2. Materials and methods

2.1. Synthesis

Bi$_2$Sn$_2$O$_7$ nanoparticles were prepared by a fast microwave-assisted method. The advantages of this method were shown in previous studies [15–18]. Typically, the precursor solution was prepared by mixing $5 \times 10^{-3}$ mol of tin (IV) chloride pentahydrate (SnCl$_2$·5H$_2$O) and $5 \times 10^{-3}$ mol of bismuth nitrate (Bi(NO$_3$)$_3$·5H$_2$O) in 100 mL of distilled water under magnetic stirring for 30 min at room temperature. The pH value of the precursor solution was adjusted to 12 through the addition of NaOH solution. The obtained solution was transferred to a 150 mL bottle and heated by a microwave oven at a power of 750 W for 20 min. After microwave processing, the solution was cooled down to room temperature naturally. The obtained precipitate was separated by centrifugation, then washed in distilled water and ethanol for several times, and dried in an oven at 70°C for 24 h. Finally, the as-prepared powders were annealed for 5 h at different temperatures from 300°C to 700°C with a heating rate of 10°C·min$^{-1}$.

2.2. Characterization

The as-prepared samples were characterized by powder X-ray diffraction (XRD) on a Siemens D5005 X-ray diffractometer. The thermal behavior was examined using a DTG-60H-Shimadzu instrument differential scanning calorimeter (DSC) in the range of 25–700°C with the heating rate of 10°C·min$^{-1}$ under flowing air. The morphology of the samples was investigated by a S4800-Hitachi electron microscope (FE-SEM). The absorption spectra of the Bi$_2$Sn$_2$O$_7$ nanoparticles were investigated using UV-Vis diffuse reectance spectroscopy (DRS) (Jasco V670 spectrophotometer). The Brunauer-Emmet-Teller (BET) surface area was determined using a Micromeritics Tristar 3000 V6.04 nitrogen adsorption apparatus. The photoluminescence (PL) spectra of samples were recorded by a fluorescence spectrophotometer (Spectrofluorometer Horiba Jobin Yvon NanoLog) using a 150 W xenon lamp as the light source. The total organic carbon (TOC) concentration during the photodegradation of RhB was analyzed by a TOC analyzer (TOC-TNM 1, Shimadzu) after filtration through a 0.22 μm filter. The Raman spectroscopy analysis was performed with a Horiba LabRAM HR Evolution spectrometer at an excitation wavelength of 532 nm.

3. Results and discussion

3.1. Structural and morphological characterization

Fig. 1 shows the diffraction patterns of the as-prepared and annealed samples at different temperatures from 300°C to 700°C. The XRD pattern of the as-prepared sample shows the BiOCl phase (marked as asterisk) as a result of a chemical reaction between Bi$^{3+}$ and Cl$^-$ ions in solution [19]. In addition, the weak crystalline phase of γ-Bi$_2$Sn$_2$O$_7$ (characteristic peaks at 2θ = 28.8°, 33.4°, 47.9°, 56.9° - JCPDS card number: 88-0496) is also observed. After annealing at 300°C, the peaks corresponding to the BiOCl phase have almost disappeared. Further increasing the annealing temperature from 400°C to 700°C resulted in an increase of diffraction peak intensities of γ-Bi$_2$Sn$_2$O$_7$ along with a gradual reduction in the line width, which implies an improvement in crystallinity. Moreover, no impurity phases were detected at annealing temperature above 400°C. We estimated the average crystalline size of the Bi$_2$Sn$_2$O$_7$ nanopowder using the Scherrer equation to the (222) diffraction peaks at 2θ = 28.8°. Table 1 shows an increase in the average crystalline size from 6 to 15 nm with increasing annealing temperature.

The crystallization process can be further characterized by means of DSC and TGA analysis. As shown in Fig. 2, the DSC data exhibits two pronounced peaks at 108 and 345°C that correspond to endothermic and exothermic processes, respectively. The exothermic peak is in the range of temperatures at which the rapid weight loss (6 wt%) in the TGA curve occurs due to the evaporation of free adsorbed water (see inset of Fig. 2), while the exothermic one (between 200 and 400°C with a smaller weight loss of 3%) can be assigned to a strong crystallization of the Bi$_2$Sn$_2$O$_7$ phase. This result agrees with the diffraction observation in the temperature range from 300 to 400°C shown in Fig. 1.

The morphology of the Bi$_2$Sn$_2$O$_7$ nanocrystallines as a function of annealed temperatures was investigated by FE-SEM as displayed in Fig. 3. All the samples comprise of spherical nanoparticles with an average grain size of ca. 40–60 nm (Table 1). To evaluate the temperature-dependent specific surface area of Bi$_2$Sn$_2$O$_7$ we carried out N$_2$ adsorption-desorption isotherm measurement. Table 1 shows that Bi$_2$Sn$_2$O$_7$ obtained at 400°C possesses the highest specific surface area of 94.8 m$^2$/g. The specific surface area in turn reduces to 55.8, 31.7 and 16.2 m$^2$/g as the annealing temperature increases to 500°C, 600°C, and 700°C, respectively.

3.2. Optical properties

The diffuse reflectance spectra of the Bi$_2$Sn$_2$O$_7$ samples were measured to explore the influence of temperature on optical properties. As illustrated in Fig. 4, for the as-prepared sample, the absorption curve shows a unique steep absorption edge at a wavelength of 385 nm ($E_g = 3.22$ eV) which could be assigned to the electronic interband transition of the BiOCl phase [20]. The absence of the Bi$_2$Sn$_2$O$_7$ adsorption edge in this sample may be due to the low crystallinity (see Fig. 1). At 300°C, in addition to the absorption edge of BiOCl we observed the exponential Urbach tail near the optical band edge as a result of the occurrence of defect states or impurity levels [21]. At higher temperatures no signal of the BiOCl phase was detected; instead, the absorption edge of the Bi$_2$Sn$_2$O$_7$ phase occurs at wavelengths in the range 500–550 nm along with the Urbach tail. Notably, with increasing treatment temperature the absorption edge of Bi$_2$Sn$_2$O$_7$ becomes significantly steeper, indicating the improvement of the degree of
crystallinity, accompanied by the growth in grain size and/or the decrease in defect levels close to the band edges. Considering the indirect transition of Bi$_2$Sn$_2$O$_7$, the Tauc plot for the absorption spectra was used to determine the optical band gap. Fig. 4b shows a slight increase of $E_g$ from 2.16 to 2.55 eV as the temperature was increased. This band gap value is smaller compared to that reported by Wu [4], Xu [22] and co-workers (2.76–2.78 eV) but is in agreement with that reported by Liu et al. [8] and Li et al. [14] using a hydrothermal method.

The room temperature photoluminescence emission spectra of the Bi$_2$Sn$_2$O$_7$ nanoparticles are depicted in Fig. 5a. The PL spectra show a broad emission band in the range of 450–750 nm with center peaks located at around 530 nm. This was attributed to a band-to-band transition, which is directly related to the recombination of photo-induced electron-hole pairs in semiconductors [23]. Hence, the lowest intensity of this peak for the Bi$_2$Sn$_2$O$_7$ sample annealed at 300°C corresponds to an inhibition of electron-hole recombination, which could lead to enhance photocatalytic activities. In addition to the intraband transition peak, several other peaks (at 580, 628, 670 nm) are revealed by fitting Lorentzian functions to the PL spectrum (Fig. 5b). To our best knowledge, there is no report about the origin of these PL peaks of Bi$_2$Sn$_2$O$_7$. Here, in combination with the occurrence of the Urbach level in the absorption spectra we propose that the three peaks may originate from defect or impurity levels in the energy band.

### 3.3. Photocatalytic activities

The photocatalytic activities of Bi$_2$Sn$_2$O$_7$ nanoparticles under visible light irradiation were investigated using RhB solution as a test contaminant. In Fig. 6a, we show different stages of the UV-Vis spectra of RhB solutions in the presence of Bi$_2$Sn$_2$O$_7$ nanoparticles annealed at 500°C. In order to evaluate the photocatalytic efficiency we employed the ratio $C_t/C_0$ with $C_0$, $C_t$ being the RhB concentration at initial time ($t = 0$) and after irradiation time $t$, respectively, for a specific peak (i.e. at wavelength 554 nm). For comparison of degradation efficiency, we used a standard photocatalyst, titanium dioxide (TiO$_2$ Degussa, P-25), as a catalyst for the degradation of RhB under visible light irradiation. As shown in Fig. 6b, the concentration ratio for the Bi$_2$Sn$_2$O$_7$ sample that is annealed at 500°C exhibits a rapid decrease compared to that of other as-synthesized samples and is also higher than the standard photocatalyst of TiO$_2$. In other words, the Bi$_2$Sn$_2$O$_7$ sample annealed at 500°C might have the highest photocatalytic efficiency in the same

### Table 1
Mean crystalline size and grain size are determined from XRD analyses and FE-SEM images, respectively; and specific surface areas are obtained by BET measurements at different annealing temperatures of 400°C, 500°C, 600°C, and 700°C.

<table>
<thead>
<tr>
<th>Annealing temperature (°C)</th>
<th>400</th>
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<td>Crystal size$_{XRD}$ (nm)</td>
<td>6</td>
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<td>40</td>
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<td>Surface area (m$^2$/g)</td>
<td>94.8 ± 0.4</td>
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### Fig. 2.
DSC curve (red line) and TGA curve (see inset) of as-prepared Bi$_2$Sn$_2$O$_7$ at heating rate of 10°C·min$^{-1}$. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

### Fig. 3.
Morphology of Bi$_2$Sn$_2$O$_7$ samples annealed at different temperatures of a) 400°C, b) 500°C, c) 600°C, and d) 700°C.

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experimental setups. It is well-known that the photocatalytic activity of a semiconductor is driven by the competition between various factors [1,4], including (i) the specific surface area, (ii) the crystallinity of nanomaterials, and (iii) the grain size. The photocatalytic reactions in the sample with a higher specific area, or equivalently a large number of surface-absorbed reactants, are assumed to be faster. However, the large surface area which acts as surface defective sites could cause a faster electron-hole recombination rate, resulting in a reduction in photocatalytic efficacy. In addition, the higher the crystallinity level of nanoparticles, the fewer the defects, and the higher the photocatalytic efficiency is [1]. On the other hand, the crystallinity that can be improved by temperature treatment usually lead to the aggregation of small nanoparticles that in turn decreases the surface area. In our case, the higher photocatalytic efficiency in the Bi2Sn2O7 sample prepared at 500 °C compared to that in the remaining ones is determined by the competition between the surface area (see Table 1) and the recombination of the charge carriers (Fig. 5).

It is known that a probably photodegradation pathway of RhB is largely driven by the N-deethylation process and the destruction of chromophores, followed by ring opening and mineralization [24–26]. Zhuang and co-workers [27] indicated that the blue-shift and broadening of the 554 nm absorption peak as a function of irradiation time were considered as an evidence of chemical bonding breaking in RhB molecular [28]. Specifically, this hypsochromic is attributed to a stepwise deethylation of RhB to produce intermediate products, including $N,N,N'$-triethyl rhodamine (539 nm), $N,N'$-diethyl rhodamine (522 nm), $N$-ethyl rhodamine (510 nm), and rhodamine (498 nm) [27]. For the RhB/Bi2Sn2O7 system, as shown in Fig. 6a, the maximal absorption peak shifts to shorter wavelengths. After 180 min of illumination, the final photodegraded product has a maximal absorption peak at 510 nm corresponding to an incompletely N-deethylated outcome of RhB, N-ethyl rhodamine [27]. The N-deethylation efficiency is estimated to be 80% by a calculation based on the absorption spectra. In order to investigate the mineralization of RhB during the photocatalytic degradation process, the TOC in the solution was measured. The efficiency of RhB mineralization was then calculated by the equation, $\frac{\text{TOC}_0 - \text{TOC}_F}{\text{TOC}_0} × 100\%$, where TOC0 and TOCF are the TOC value of the initial RhB solution and of the degraded RhB solution, respectively. For the RhB/Bi2Sn2O7 sample prepared at 500 °C, the mineralization efficiency is determined to be ca. 15%. The results suggest that the RhB degradation process over Bi2Sn2O7 is dominated by the contribution from N-deethylation. In this work, we did not attempt to further identify the photodegradation intermediate products. A detailed analysis of the reaction intermediates was given elsewhere (e.g. Refs. [24–28]).

Fig. 4. a) UV-Vis DRS of the Bi2Sn2O7 nanoparticles annealed at different temperatures from 400°C to 700°C. b) Dependence of $(\alpha h\nu)^{1/2}$ versus photon energy $h\nu$ of the Bi2Sn2O7 nanoparticles.

Fig. 5. a) Photoluminescent spectra of the Bi2Sn2O7 samples annealed at various temperatures and b) Lorentzian fitting curves of photoluminescent spectrum of Bi2Sn2O7 sample annealed at 400°C.
More information about the RhB photocatalytic degradation under visible light irradiation can be extracted from the Raman measurements. Fig. 7 shows a sequence of Raman data of RhB aqueous solutions recorded as a function of reaction time during the degradation over the 500°C annealed sample. The vibration fingerprints of RhB [29–31] can be clearly observed after subtraction of fluorescence background signals. In Fig. 7, we label the bands centered at 1355, 1506, 1541, and 1647 cm⁻¹ as A; 413 cm⁻¹ as B; 622 and 933 cm⁻¹ as C; 664 and 705 cm⁻¹ as D; 984, 1193 and 1276 cm⁻¹ as E; and 1054 cm⁻¹ as F. The A band corresponds to xanthene ring (XR) stretching. B is XR deformation and NH₂ oscillation. C represents XR and phenyl ring (PHR) stretching. D is attributed to C-H out of the plane of the XR. E is C-H bending of the XR. F is PHR stretching. One can see that each Raman peak seems to remain at the same position, indicating that the changes in chemical compositions of RhB during the photocatalytic degradation process have little effect on the basic vibrational modes. Another interesting feature is that the intensity of the A bands decrease rapidly after 180 min of irradiation (see the inset in Fig. 7), which is in agreement with the UV-Vis results. However, in other bands (like C, E and F bands), their intensity fluctuates with respect to irradiation time. The results also confirm the existence of various intermediate products and the complexity of the RhB photocatalytic process. A complete degradation of dye and by-products to form carbon dioxide and water, therefore, needs a further investigation of the degradation behavior of the resulting compounds, which is beyond our current study.

For practical applications of the photocatalyst, the reusability and stability are important parameters. The recycle the photocatalytic performance of the Bi₂Sn₂O₇ samples annealed at 500°C is depicted in Fig. 8. The result indicates that in comparison to the first run, the photocatalytic activity losses were around 1%, 2.7%, and 4% for the second, third, and fourth run, respectively. Therefore, it can be concluded that the Bi₂Sn₂O₇ nanoparticles annealed at 500°C have relatively high and stable performance photocatalytic activity.

4. Conclusions

In summary, the Bi₂Sn₂O₇ nanoparticles have been successfully synthesized using a fast microwave-assisted technique. Different from the existence of three polymorphous as a function of temperature in previous reports [12,13], i.e α-, β-, and γ-phases where the γ phase was known to be favored at high temperature above 600°C, we found a unique phase of γ-Bi₂Sn₂O₇ in the range of thermal treatment temperatures of 300–700°C. At lower temperatures, the BiOCl phase was apparently detected in addition to the low crystallinity γ phase. As treatment temperature increases, the crystallinity is significantly improved and the crystal size increase slightly contrast to a decrease of the specific surface area. We have investigated the photocatalytic activity and the complexity of the RhB photocatalytic process. A complete degradation of dye and by-products to form carbon dioxide and water, therefore, needs a further investigation of the degradation behavior of the resulting compounds, which is beyond our current study.

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Fig. 7. Raman spectral changes for RhB solution as a function of visible light irradiation time over the as-synthesized Bi₂Sn₂O₇ at 500°C. For clarity, only Raman spectra at three different irradiation times are shown. The inset is a magnification of the pronounced peak centered at 1647 cm⁻¹. The vibrational modes are labeled as A-F, as described in the text.

Fig. 8. Cycling runs of the photocatalytic degradation of RhB under visible light irradiation with the presence of Bi₂Sn₂O₇ catalysts annealed at 500°C.
of Bi$_2$Sn$_2$O$_7$ nanoparticles in the degradation of RhB solutions and observed that there exists an optimal synthesis temperature of 500°C for the enhanced photocatalytic reaction. TOC measurement results indicated that RhB could be mineralized to CO$_2$ after 180 min of degradation. The mineralization efficiency is evaluated to be 15%, which is much smaller than the N-deethylation of RhB (80%). On combinations of the better separation of the photogenerated charge carriers, signaled by a weak photoluminescence, we concluded that for Bi$_2$Sn$_2$O$_7$ nanoparticles obtained by the microwave-assisted method the recombination of the charge carriers was the dominant process.

References


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