Photocatalytic activity of RBi$_2$O$_4$NO$_3$ (R: Tb, Dy, Er, Gd, and Ho) for phenol degradation under visible light irradiation

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Abstract
Rare-earth metal bismuth oxynitrates, RBi$_2$O$_4$(NO$_3$) (R: Tb, Dy, Er, Gd, and Ho), were prepared by hydrothermal reactions using hydrated sodium bismuthate (NaBiO$_3$·nH$_2$O) and rare earth nitrates. The optical band gap of the Tb compound was 2.10 eV, and those for the Dy, Er, Gd, and Ho compounds ranged between 2.92 and 2.94 eV. All the compounds were yellowish white in color, except the Tb compound, which was yellowish brown. Surface area of all the compounds varied from 2.46 to 8.17 m$^2$/g. The Tb compound showed the highest photocatalytic activity for phenol degradation under visible light irradiation than other compounds (Dy, Er, Gd, and Ho). At elevated temperatures, TbBi$_2$O$_4$(NO$_3$) was changed to (Tb$_{0.333}$Bi$_{0.667}$)O$_{1.50}$ having a fluorite-type structure, which exhibited poor photocatalytic activity for phenol degradation under visible light irradiation.

Key-words: Rare earth bismuth oxynitrites, Photocatalyst

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1. Introduction

Trivalent Bismuth (Bi$^{3+}$) based oxides and oxyhalogens have been paid the attention for the photocatalysis research because of their visible-light-responsive property. The origin of visible light response comes from the sp hybridization of O 2p and Bi 6s orbitals and/or revised lone pair model.1–5 Form this motivation we have prepared few trivalent bismuth based oxide such as Na$_{0.32}$Bi$_{1.68}$Ti$_{2.04}$–(OH)$_{0.44}$,6 Pb$_{0.35}$Bi$_{0.65}$O$_{1.59}$,7 Bi$_{3.33}$(VO$_4$)$_2$O$_2$,8 among them Pb$_{0.33}$Bi$_{0.67}$O$_{1.59}$ and Bi$_{3.33}$(VO$_4$)$_2$O$_2$ exhibited photocatalytic properties for degredation of phenol under visible light.7,8) There are many other bismuth oxide based compounds BiPO$_4$,9 BiVO$_4$,10 BiFeO$_3$,11 Bi$_2$MoO$_6$,12 Bi$_2$WO$_6$,13 BiOX (X = Cl, Br, I),14–16 Bi$_2$O$_3$Cl,17 Bi$_3$S$_2$O$_15$,18 Bi$_2$MoO$_6$,19 Bi$_2$MoO$_6$,20 BiIO$_4$/BiOBr,21 BiVO$_4$/BiOBr,22 BiO/Bi$_2$O$_7$Cl$_2$,23 can enhance photocatalytic activity under visible light irradiation. Heterojunction composites of bismuth based oxides AgBr/BiPO$_4$,19 Bi$_2$MoO$_6$,20 Bi$_2$O$_3$Cl,23 can enhance photocatalytic activity performance. Besides heterojunction formation, rare earth elements doped in the bismuth based oxides Yb:BiPO$_4$,24 La:BiFeO$_3$,25 (La, Yb):BiVO$_4$,26–28 Eu, Yb:Ce:BiOX (X = Cl, Br, I),28–31 La, (Er, Yb):Bi$_2$O$_3$Cl$_2$ (Er, Yb):Bi$_2$MoO$_6$,32–35 Yb, (Ce, Nd, Pr or Sm):Bi$_2$WO$_6$,36–37 Ce:BiOBr,38 Nd:Bi$_2$Fe$_4$O$_9$,39 can also improve the photocatalytic degradation of organic pollutants [phenol, methylene blue (MB), methyl orange (MO), rhodamine B]. Incorporation of rare earth elements either in A-site or B-site of the single perovskite Bi$_{0.84}$RE$_{0.16}$FeO$_3$ (RE = La, Gd),40 double perovskite Ba$_2$RBiO$_6$ (R = La, Ce, Nd, Sm, Eu, Gd, Dy),41 layer perovskite Bi$_{1.25}$Mo$_{0.75}$Ti$_2$O$_7$ (R = La, Sn, Nd, and Eu)42 and pyrochlore Bi$_2$RNbO$_7$ (R = Y, Rare Earth)43,44 structures were studied for the photocatalytic properties for degradation of organic pollutants (MB, MO) and water splitting. We had prepared rare-earth bismuth oxynitrites, RBi$_2$O$_4$NO$_3$ (R: Y, Tb, Dy, Er, Gd, and Ho) with the layered structure, where trivalent Bi and rare earth elements are present in the crystal structure. The crystal structure of the rare-earth bismuth oxynitrite was refined by the neutron powder diffraction data,45 but their photocatalytic activities were not been evaluated yet.

Herein, we will describe the photocatalytic activities of RBi$_2$O$_4$(NO$_3$) for phenol degradation under visible light irradiation; we also discuss the formation of a fluorite-type (Tb$_{0.333}$Bi$_{0.667}$)O$_{1.50}$ which was obtained by heating TbBi$_2$O$_4$(NO$_3$) at 800 °C. Additionally, the fluorite-type (Tb$_{0.333}$Bi$_{0.667}$)O$_{1.50}$ crystal structure was refined by using synchrotron powder X-ray diffraction (SPXRD) data and checked the photocatalytic activity for phenol degradation under visible light irradiation.

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2. Experimental

NaBiO$_3$·$n$H$_2$O and R(NO$_3$)$_3$·$n$H$_2$O (R: Tb, Dy, Er, Gd, and Ho) with 1:4 molar ratio was placed in a teflon-lined autoclave (70 ml) with 40 ml distilled water. The autoclave was then closed and heated at 200 °C for 4 days to complete the hydrothermal reaction. After cooling the autoclave, the solid products were separated by filtration, washed with distilled water, and dried at 60 °C. All the starting materials [NaBiO$_3$·$n$H$_2$O, R(NO$_3$)$_3$·$n$H$_2$O] were purchased from Kanto Chemical Co., Inc. (Tokyo, Japan). The products were characterized by X-ray Diffraction (XRD), using a Miniflex-600 Rigaku diffractometer with nickel-filtered Cu Kα radiation ($\lambda$ = 1.54056 Å). SPXRD measurements were performed at the BL02B2 beamline of SPring-8, Hyogo, Japan. Data were collected at constant wavelength ($\lambda$ = 0.412999 Å). Crystal structure was refined using the Rietveld program RIETAN-FP and visualized using VESTA software. Diffuse-reflectance spectra were collected using the JASCO V-550 spectrometer, and converted using the Kubelka-Munk function. The oxidation states for Tb and Bi atoms were determined by X-ray photoelectron spectroscopy (XPS, JEOL-9200). Surface areas were measured by N$_2$ adsorption (BELSORP-mini, Nippon BEL). Aqueous phenol solution (20 ppm) was prepared in ultrapure water and the catalyst was added at a concentration of 3 g/L. The solution was magnetically stirred in the dark and under visible light irradiation ($\lambda$ ≥ 420 nm), and phenol concentration was determined by liquid chromatography in a time-dependent manner (JASCO LC-2000) at every 30 min interval, where (30–60) min of elapsed time under dark condition and (90–240) min of elapsed time under visible light irradiation.

3. Result and discussion

3.1 XRD and crystal structure of RBi$_2$O$_4$NO$_3$

Rare-earth bismuth oxynitrates, RBi$_2$O$_4$NO$_3$ (R: Tb, Dy, Er, Gd, and Ho) were prepared by hydrothermal reactions. The crystal structure of the Y compound which was isostructural with RBi$_2$O$_4$NO$_3$, was refined by using the neutron diffraction data. The XRD pattern of the Y compound obtained from the crystallographic information file (CIF) is shown in Fig. 1(a). Single phase of RBi$_2$O$_4$NO$_3$ was confirmed by comparison the XRD patterns [Fig. 1(b)] with YBi$_2$O$_4$NO$_3$. The X-ray powder patterns were indexed in the tetragonal system with $a$ ~3.89 Å and $c$ ~10.20 Å. From the Energy-dispersive X-ray spectroscopy (EDX) analysis, the molar ratio of Bi/R in the products was found to be two. Crystal structure of RBi$_2$O$_4$NO$_3$ was displayed in Fig. 1(c). The crystal structure of RBi$_2$O$_4$NO$_3$ is consisted of (M$_3$O$_4$)$^+$ layers (M: R, Bi1, Bi2) and (NO$_3$)$^-$ groups, which are alternate stacking along the $c$-axis as displayed in Fig. 1(c). R is coordinated by eight oxygen atoms formed a cubic polyhedron, whereas Bi1 and Bi2 atoms formed square antiprism and also coordinated by eight oxygen atoms [Fig. 1(c)]. The R atom is surrounded by four different sites of oxygen atoms as shown in the Fig. 1(c) but Bi1 and Bi2 surrounded by three different sites of oxygen atoms. The SEM images of the starting compound and the Tb compound are shown in Fig. S1. The starting compound had a flake like particle with ~10 μm, and the Tb derivative shows an irregular shape particle with submicron size. The other rare earth metal compounds had also similar morphology.

3.2 Optical properties of RBi$_2$O$_4$NO$_3$

Optical absorption spectra of RBi$_2$O$_4$(NO$_3$) (Fig. S2) show that the absorption edges lie within the visible region. The color of the samples were yellowish white, except for the Tb compound, which was yellowish brown. The band gap of the Tb compound was lower (2.10 eV) than that of the other compounds.
than those of the other compounds (2.92–2.94 eV), as shown in Fig. 2. The Tb compound exhibited two-steps absorption spectra (Fig. S2) probably due to the presence of two oxidation states (Tb$^{3+}$ and Tb$^{4+}$), as shown in the XPS data (Fig. S3). The room-temperature electrical resistivity of a pelletized sample of the Tb compound was $\sim$100 $\Omega$m, and the temperature dependence of electrical resistance showed semiconducting behavior. EDX analysis indicated that the Tb/Bi ratio was $\sim$0.5, and the chemical composition was deduced to be (Tb$^{3+}$x, Tb$^{4+}$1-x)Bi$_2$O$_4$x(NO$_3$)$_1$-x, where $x = 0.33$ was calculated from XPS analysis.

### 3.3 Photocatalytic property of RBi$_2$O$_4$NO$_3$

The photocatalytic activities of RBi$_2$O$_4$(NO$_3$) were evaluated in terms of phenol decomposition at an initial concentration of 20 ppm, using a 0.15 g sample in 50 mL of ultrapure water. Schematic representation of the photocatalytic experiment under visible light irradiation, including the experimental conditions, is shown in Fig. 3(a). Time dependence of C/C$_0$ in the dark and under visible-light irradiation ($\lambda \geq 420$ nm) is shown in Fig. 3(b). The suspensions were magnetically stirred in the dark (60 min), and the phenol adsorption for all the samples were measured. During this period, the decrease in phenol concentration was negligible. The Tb compound showed the fastest decomposition rate for phenol under visible light irradiation, probably because it has the lowest band gap among the studied catalysts. We were unable to measure the surface area of the Er compound because of the extremely low nitrogen adsorption, resulting in the lowest photocatalytic activity. The Dy, Gd, and Ho compounds showed comparable photocatalytic activities due to similar band gaps and surface areas (Table S1). Previously, Li et al. reported the photocatalytic activity of Tb doped Bi$_2$MoO$_6$ exhibited better than pure Bi$_2$MoO$_6$ for phenol degradation. The DOS calculation of these (RBi$_2$O$_4$NO$_3$) compounds did not lead to a reasonable result because of heavy atoms such as rare earth metal and bismuth atoms. DOS calculations and photocatalytic mechanism under investigations.

### 3.4 Photocatalytic property of Tb compounds heated at different temperatures

At elevated temperatures (above 600 °C), the Tb compound was changed to (Tb$_{0.333}$Bi$_{0.667}$)O$_{1.50}$, having a fluorite-type structure [Fig. 4(a)], by releasing of NO and O$_2$ similar to that of YBi$_2$O$_4$NO$_3$. Photocatalytic activity of the heated Tb-compound were checked for phenol (20 ppm) under visible light ($\lambda \geq 420$ nm). Time dependence of C/C$_0$ in the dark and under visible-light irradiation ($\lambda \geq 420$ nm) is shown in Fig. 4(b). First, the
suspensions (phenol solution and catalyst) were magnetically stirred in the dark (60 min), and the phenol adsorption for all the samples were negligible. Untreated Tb-compound (Tb-RT) showed highest photocatalytic activity for phenol degradation. However, photocatalytic activity gradually decreased for the samples with increasing the temperature from 200–800 °C as shown in Fig. 4(b). This tendency may be due to the agglomerated of powder particles at higher temperature, which could influence the photocatalytic property. UV–vis spectra of the heated Tb compounds at elevated temperatures are shown in Fig. S4. The band gaps and surface areas of the heated Tb compounds at different temperatures are shown in Table S2.

3.5 Crystal structure refinement of (Tb0.333Bi0.667)O1.50

The fluorite-type (Tb0.333Bi0.667)O1.50 contained only Tb3+ and Bi3+, which is confirmed by the XPS data (Fig. S5). The SPXRD pattern of the (Tb0.333Bi0.667)O1.50 can be indexed to a fluorite-type structure [Fm$ar{3}$m (#225)] with a cubic cell of $a = 5.49405(4)\,$Å. The corresponding SPXRD Rietveld refinement pattern is shown in Fig. 5, with the crystal structure of this new compound shown in the inset. The final R-factors were $R_{wp} = 7.98\,$% and $R_p = 6.30\,$%. Oxygen (O2) occupancy (0.75) at the 8c site was kept fixed for charge balance. The (Tb3+, Bi3+)-O2 mean distance in (Tb0.333Bi0.667)O1.50 (2.379 Å) was shorter than that of (Bi3+)-O2 in fluorite-type-Bi2O3 (2.453 Å) due to the lower ionic radius of Tb3+ than that of Bi3+ for the same coordination number. The crystallographic data and structural model parameters are summarized in Tables 1 and 2, respectively.

Table 1. Crystal data for (Tb0.333Bi0.667)O1.50

| Chemical formula | (Tb0.333Bi0.667)O1.50 |
|-----------------|-----------------------|
| Radiation type, $A$ (Å) | Synchrotron X-ray, 0.412999 |
| Temperature (°C) | 25 |
| Crystal System | Cubic |
| Space group | Fm$ar{3}$m (No. 225) |
| Lattice parameters (Å) | $a = 5.49405(4)$ |
| Volume ($\Omega$) | 165.836(2) |
| Formula weight (g/mol) | 216.31 |
| Calculated density (g/cm$^3$) | 8.66 |
| Z Value | 4 |
| $R_{wp}$ (%) | 7.98 |
| $R_p$ (%) | 6.30 |
| $R_B$ (%) | 6.11 |
| $R_F$ (%) | 7.75 |
| $S$ | 4.45 |

Table 2. Structural parameters of (Tb0.333Bi0.667)O1.50

| Atom | Site | $x$ | $y$ | $z$ | $g$ |
|------|------|----|----|----|----|
| Tb   | 4a   | 0  | 0  | 0  | 0.333* |
| Bi   | 4a   | 0  | 0  | 0  | 0.667* |
| O    | 8c   | 0.25 | 0.25 | 0.25 | 0.75* |

*denotes a fixed parameter, $g$ = Occupancy.
ments at SPring-8, which were performed with approval of
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ature to 800 °C.

Figure 6 shows the plot of unit-cell volume against the ionic radii of cations for fluorite-type oxides. The fluorite-type oxides were classified into two groups of typical fluorite-type oxides and rare-earth bismuthates. The former showed a linear relationship between the unit-cell volume and ionic radii of cations, and the latter showed a deviated relationship. \(\text{Tb}_{0.333}\text{Bi}_{0.667}\text{O}_{1.50}\) was included in the bismuthate group, and the deviation from the linear relationship was considered to be caused by the stereo active lone pair of \(\text{Bi}^{3+}\). Thermal stability of the compound was observed up to 800 °C, which was observed from the SPXRD as shown in Fig. 6(b).

4. Conclusion

Single-phase \(\text{RBi}_2\text{O}_4(\text{NO}_3)\) (R: Tb, Dy, Er, Gd, and Ho) compounds were hydrothermally synthesized and their photocatalytic activities were evaluated in terms of phenol degradation under visible light irradiation. The Tb compound possessed the lowest band gap, and thus, exhibited the highest photocatalytic activity for phenol degradation up to 210 min among the synthesized compounds. The Tb compound was changed to fluorite-type \(\text{Tb}_{0.333}\text{Bi}_{0.667}\text{O}_{1.50}\) compound, when heated to 600 °C. Crystal structure of this fluorite-type compound was refined from the SPXRD.

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