Investigation of photodegradation of rhodamine B over a BiOX (X = Cl, Br and I) photocatalyst under white LED irradiation

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Abstract. In this study, a low temperature precipitation method was applied to prepare BiOX (X = Cl, Br and I) nanostructures using HCl, HBr and HI as halogen sources. The as-prepared samples were characterized by various techniques such as X-ray diffraction, field emission scanning electron microscopy, energy dispersive spectroscopy, Fourier-transform infrared spectroscopy, diffuse reflectance spectroscopy and nitrogen adsorption-desorption analysis. The photoactivity of BiOX samples to degrade rhodamine B (RhB) molecules in aqueous solution was investigated under white LED irradiation. The results indicated that BiOCl exhibits a higher photodegradation performance in comparison with BiOBr and BiOI. Ninety six percent of the RhB molecules was degraded over BiOCl after 25 min light irradiation, whereas under the same conditions, the degradation efficiencies of BiOBr and BiOI samples are 62 and 24%, respectively. The enhanced photodegradation activity of BiOCl is ascribed to the higher specific surface area which increases the physical adsorption of RhB molecules on the photocatalyst surface. Moreover, the photodegradation mechanism and the main radical species in the degradation reactions over BiOCl were investigated.

Keywords. Bismuth oxyhalide; photocatalysis; rhodamine B; LED lamp.

1. Introduction

Over the last few decades, with the rapid development of industries the water pollutant has become one of the serious challenges all over the world [1,2]. Up to now, various methods have been applied for wastewater treatment. Among them, the use of semiconductor photocatalysts to degrade organic pollutants under solar light irradiation has received extensive attention [2,3]. Titanium oxide (TiO₂) as a wide band gap semiconductor (3.0–3.2 eV) is one of the most studied photocatalysts. However, TiO₂ which can only be excited under ultraviolet light (UV) is not efficient for the use of solar light [4–6]. In recent years, bismuth oxyhalide (BiOX, X = Cl, Br and I) semiconductors have received significant attention due to its excellent physical and chemical properties, and also its unique layer structure [7–10]. The crystal lattice of bismuth oxyhalide consists of [Bi₂O₂]²⁺ layers sandwiched between two sheets of halide ions, inducing a static internal electric field perpendicular to the [Bi₂O₂]²⁺ and halide ion layers [11,12]. The induced electric field can separate the photogenerated electron-hole pairs along the side of [001], which can lead to the enhancement in the photocatalytic activity for the degradation of dye molecules under UV and visible light irradiation [13–17].

Up to now, a few studies have compared the activity of BiOX (X = Cl, Br and I) for photocatalytic degradation of organic dyes [18–22]. Chang *et al* [18] synthesized crystalline BiOX (X = Cl, Br and I) with the sheet-like morphologies using NaBiO₃ and HX (X = Cl, Br and I) as the raw materials. The photocatalytic activity evaluation indicated that BiOI exhibits the best activity under Xe light irradiation to degrade phenolic endocrine disrupting chemical due to its narrow band gap. Qin *et al* [19] compared the photocatalytic activity of hierarchical BiOX (X = Cl, Br and I) microspheres to degrade methyl orange under visible light irradiation; the results exhibited that BiOI with the smallest band gap shows the best photocatalytic performance. Chen *et al* [20] studied different morphologies of BiOX (X = Cl, Br and I) by a simple precipitation method at room temperature, and indicated that the 3-D flower-like samples show higher photocatalytic activity than the sheet-like morphologies under visible light irradiation. Jia *et al* [21] fabricated BiOX (X = Cl, Br and I) by the solvothermal method using tetrabutylammonium halide as the halogen source, and BiOCl and BiOBr demonstrated higher photocatalytic activity compared with BiOI to degrade RhB and salicylic acid under ultraviolet-visible (UV-Vis) light irradiation. Lv *et al* [22] synthesized BiOX (X = Cl, Br and I) samples with the same nanosheet morphology, BiOCl compared with BiOBr and BiOI exhibited the highest photocatalytic activity to degrade OII, MB

and phenol under UV-Vis irradiation due to its high oxidative activity of generated h^+ radicals, while it nearly has no photoactivity under visible light irradiation. In spite of these efforts, more attempts are still needed to optimize the bismuth oxyhalides for practical application.

On the other hand, the conventional light sources for photocatalytic degradation of organic pollutant over the semiconductor photocatalysts are mercury vapour lamp, xenon lamp and natural solar light. The mercury vapour lamp and xenon lamp light sources have some drawbacks such as heat production, required cooling systems, expensive and low efficiency [23,24]. Moreover, the use of natural solar light has some disadvantages including need a large area for application, high cost of installation and the availability and direction of solar light [23,24]. In recent years, light emitting diodes (LEDs) have attracted much attention due to their advantages such as longer life time, little heating, lower energy consumption and tunable emission wavelength [25,26]. Thus, in this work, the white LED lamp was applied as the visible light source for photodegradation of rhodamine B (RhB) molecules over BiOX ($X = \text{Cl, Br and I}$) photocatalysts. The BiOX composite has been synthesized by a simple precipitation method with the aid of HX ($X = \text{Cl, Br and I}$) as the halogen source. The synthesized BiOX ($X = \text{Cl, Br and I}$) samples were characterized by various techniques. Also, the effect of halide atoms on the photoactivity of BiOX for the degradation of RhB was investigated. The results indicated that the photodegradation of RhB over BiOCl is due to the dye photosensitization effect, while the photodegradation over BiOBr and BiOI can be attributed to both the direct photocatalytic process and dye photosensitization effect. Moreover, the reactive species during photodegradation of RhB over BiOCl were determined.

2. Experimental

All reagents are analytical grade and used without any purification. In a typical experiment, 5 mg polyvinylpyrrolidone (PVP, $M_w = 1,300,000$) was added into 50 ml water. After stirring for 15 min, 1.6 mg HX ($X = \text{Cl, Br and I}$) and 2.134 g $\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$ were dissolved into the solution under magnetic stirring. Then, the solution was diluted by adding 50 ml water. Then, the resulting solution was treated at 70°C for 60 min under stirring for precipitation formation. The precipitate was washed with water several times and dried in an oven at 70°C for 20 h.

The X-ray diffraction (XRD) pattern obtained by the X'Pert Pro model was applied to investigate the crystal structure of the products. Fourier transform-infrared (FT-IR) spectra were recorded by Spectrum RX I (PerkinElmer) in the range of $400\text{--}4000\text{ cm}^{-1}$ with KBr as the reference. The field emission scanning electron microscopy (FESEM, ESCAN MIRA3 XMUFESEM) and energy dispersive spectroscopy (EDS) were applied to investigate the morphology and chemical

composition of BiOCl, BiOBr and BiOI. The N_2 adsorption-desorption analysis was carried out by a BELSORP-mini II to determine the specific surface area and pore size distribution of the products. The optical properties of the products were investigated by diffuse reflectance spectroscopy (DRS, Avaspec-2048-TEC model, Avantes) in the wavelength range of $200\text{--}800\text{ nm}$.

The aqueous RhB solution was used as the pollutant model to compare the photocatalytic activity of BiOCl, BiOBr and BiOI under a white LED lamp (30 W). The distance between the lamp and reaction solution was 10 cm. In each experiment, 20 mg of photocatalyst was dissolved into 60 ml of aqueous RhB solution (10 mg l^{-1}). Prior to irradiation, the suspension was magnetically stirred under dark conditions to ensure the adsorption/desorption equilibrium between the dye molecules and photocatalyst surface. During light irradiation, 2 ml of solution was taken out at 10 min intervals and centrifuged to remove the photocatalyst particles. Then, the residual concentration of RhB in the solution was measured by recording the absorbance at 553 nm using UV-Vis spectroscopy.

3. Results

The crystal structures of the samples were investigated by an XRD technique. Figure 1 displays the XRD patterns of the BiOX ($X = \text{Cl, Br and I}$) powder samples. It is observed that all the diffraction peaks of the as-prepared BiOCl sample can be well ascribed to a pure tetragonal phase of BiOCl (JCPDS No. 06-0249). Also, figure 1b and c exhibits that the XRD diffraction peaks of BiOBr and BiOI samples can be easily indexed to the tetragonal phases of BiOBr (JCPDS No. 09-0393) and BiOI (JCPDS No. 73-2062), respectively. In addition, there are no impurity diffraction peaks, which indicate the phase-purity of the samples.

FT-IR was applied to determine the chemical bonding of samples. Figure 2 shows the FT-IR spectra of BiOCl, BiOBr and BiOI. The characteristic absorption bands at about 526, 518 and 516 cm^{-1} for BiOCl, BiOBr and BiOI, respectively, correspond to the symmetric vibration of the Bi-O bond. In all of the spectra, the bands around 1286, 1432 and $\sim 1640\text{ cm}^{-1}$ are attributed to the C-N, C=C and C=O bonds, respectively [27,28]. The observed bands in the range of $2870\text{--}2960\text{ cm}^{-1}$ can be ascribed to the asymmetric and symmetric stretching vibrations of C-H bonds [27,28]. Moreover, the absorption band at around 3430 cm^{-1} is attributed to the stretching vibration of O-H groups of adsorbed water molecules [27]. Therefore, the FT-IR results confirm the interaction of PVP and BiOX samples.

Figure 3 shows the FESEM images and the corresponding EDS spectra of BiOX ($X = \text{Cl, Br and I}$) samples. It is observed that BiOCl and BiOBr are composed of nanoplates. While the BiOI sample displays a hierarchical flower-like structure with

Figure 1. XRD patterns of (a) BiOCl, (b) BiOBr and (c) BiOI.

Figure 2. FT-IR spectra of (a) BiOCl, (b) BiOBr and (c) BiOI.

an average size of 1 μm . Furthermore, the chemical composition of the BiOX ($X=\text{Cl}$, Br and I) samples was identified by EDS analysis. The EDS spectra are shown in figure 3. This analysis reveals the existence of Bi, O, Cl, Br or I elements, which displays the high purity of the samples.

It is well known that the specific surface area of a semiconductor has a significant impact on its photocatalytic performance. Thus, to study the influence of the specific surface area on the photocatalytic activity, the specific surface area and pore-size distribution of samples were measured by nitrogen adsorption/desorption analysis. Figure 4 shows the nitrogen adsorption/desorption isotherms and the BJH

pore size distribution curves of BiOCl, BiOBr and BiOI. It is observed that all the samples display type IV adsorption isotherm with a H_3 -hysteresis loop in the range of $0.5 < P/P_0 < 1$, which indicates the presence of a mesopore structure [29,30]. The pore volumes of BiOCl, BiOBr and BiOI calculated from BJH plots are about 0.085, 0.076 and $0.029 \text{ cm}^3 \text{ g}^{-1}$, respectively. In addition, the BET surface areas of BiOCl, BiOBr and BiOI are 15.63, 9.41 and $3.68 \text{ cm}^2 \text{ g}^{-1}$, respectively. Therefore, the pore volume and specific surface area of the BiOCl sample are larger than those of BiOBr and BiOI. Therefore, it is predicted that the BiOCl sample has more capacity for surface adsorption of dye molecules, which may enhance the photocatalytic degradation of dye molecules due to the dye photosensitization effect [31].

The optical absorption is another key factor that has a significant effect on the photocatalytic activity of semiconductors. The optical properties of BiOX ($X=\text{Cl}$, Br and I) powder samples were investigated by UV-Vis DRS. Figure 5 shows the DRS spectra of the samples. It can be seen that the optical absorption spectra of BiOCl, BiOBr and BiOI display strong absorption in the UV region, and their absorption edges arise at around 375, 438 and 653 nm, respectively. Thus, the absorption edges of BiOBr and BiOI are in the visible region, while the BiOCl sample exhibits an absorption edge in the UV region. The band gap energies of samples were estimated by the Tauc method according to the below equation [30,31]:

$$\alpha h\nu = A(h\nu - E_g)^{n/2}.$$

In this formula α , $h\nu$, E_g and A are the absorption coefficient, photon energy, band gap energy and constant, respectively. The value of n which exhibits the nature of transition is considered to be 4 for BiOX ($X=\text{Cl}$, Br and I) as an indirect semiconductor [20]. The band gap energy is estimated by extrapolating the tangent line of the $(\alpha h\nu)^{1/2}$ plot in the linear region to intercept the x -axis. The obtained E_g values are about 3.30, 2.82 and 1.72 eV for BiOCl, BiOBr and BiOI, respectively. It can be concluded that the band gaps were shifted to longer wavelengths with increase in the radius of halide atom.

The photocatalytic activity of the samples was evaluated by degradation of aqueous RhB solution under the LED lamp as the visible light source. Typically, 20 mg of each powder samples was added into 60 ml of RhB solution (10 mg l^{-1}) as the pollutant model. Then, after 60 min dark adsorption, the aqueous RhB solution was followed by visible light irradiation. Figure 6a–c shows the changes in the UV-Vis absorption spectra of RhB solution in the presence of various photocatalysts at different irradiation times. It is observed that the intensity of the absorption peak of RhB at 553 nm in the presence of BiOX ($X=\text{Cl}$, Bi and I) gradually decreases with increasing irradiation time, which demonstrates the degradation of RhB molecules. Figure 6d displays the changes in the concentration of RhB solution as a function of irradiation

Figure 3. FESEM images and EDS spectra of (a, b) BiOCl, (c, d) BiOBr and (e, f) BiOI.

Figure 4. Nitrogen adsorption/desorption isotherms and the corresponding pore size distribution (inset) of (a) BiOCl, (b) BiOBr and (c) BiOI.

time over photocatalysts. For comparison the concentration changes of RhB solution in the absence of the photocatalyst are also shown (direct photolysis process). It can be seen that the degradation of RhB solution is negligible in the absence of

Figure 5. (A) UV-Vis diffuse reflectance spectra and (B) the corresponding $(\alpha h\nu)^{1/2}$ vs. photon energy ($h\nu$) plots for the band gap determination of (a) BiOCl, (b) BiOBr, (c) BiOI.

the photocatalyst, while nearly 96% of the RhB molecules can be degraded over BiOCl after 25 min light irradiation. In addition, under the same conditions the degradation efficiencies of BiOBr and BiOI samples are 62 and 24%, respectively. Furthermore, figure 6e displays the plots of $\ln(C_0/C)$ as a function of irradiation time over various photocatalysts. It can be seen that there is a linear relationship between $\ln(C_0/C)$ and irradiation time (s). Thus, the degradation of RhB can be described by pseudo-first-order relation [32–34]:

$$\ln(C_0/C) = kt,$$

where C_0 and C are the concentration of RhB at the initial state and after t time and k is the first rate constant. The rate constants for the RhB degradation over BiOCl, BiOBr and BiOI photocatalysts are 0.122, 0.036 and 0.012 min⁻¹, respectively (figure 6f). Thus, the rate constant of BiOCl is about 3.4 and 10 times higher than that of BiOBr and BiOI, respectively. It can be concluded that the photoactivity of BiOCl to degrade RhB solution under visible light irradiation is much higher than those of BiOBr and BiOI.

Three possible mechanisms can be considered for the photodegradation of dyes in the presence of a semiconductor: (1) direct photolysis of dyes, (2) dye-photosensitization

Figure 6. The temporal evaluation of UV-Vis absorption spectra of RhB solution under white LED irradiation in the presence of (a) BiOCl, (b) BiOBr and (c) BiOI. (d) The photodegradation performance of RhB solution, (e) the plots of $\ln(C_0/C)$ vs. irradiation time and (f) the rate constant (i) in the absence of photocatalyst, and in the presence of (ii) BiOCl, (iii) BiOBr and (iv) BiOI as the photocatalyst under white LED irradiation.

process and (3) direct photocatalytic process [31,35]. As observed, the direct photolysis of RhB is negligible under LED light irradiation. Moreover, when the energy of the incident photon is larger than the band gap of the semiconductor, the electrons and holes are generated in the conduction band (CB) and valence band (VB) of the semiconductor. Then, the photoexcited electrons and holes may directly react with the dyes or produce the reactive radical species for the degradation of dye molecules (direct photocatalytic process) [36,37]. However, based on the DRS results, the band gaps of BiOCl, BiOBr and BiOI are about 3.30, 2.82 and 1.72 eV, respectively. Therefore, BiOCl cannot be excited under visible light irradiation to generate electron-hole pairs for the direct photocatalytic process because of its wide optical band gap, while BiOBr and BiOI are able to excite under visible light irradiation. As a result, the photodegradation activity of BiOCl is attributed to the dye photosensitization process. In the dye photosensitization process, the photoexcited dyes adsorbed on the semiconductor surface can transfer their electrons into the CB of the semiconductor, which may react with O₂ to form the radical species ([•]O₂) for the degradation of dye molecules [38,39]. However, the photodegradation of RhB over BiOBr and BiOI with visible light active band gaps may be due to both the direct photocatalytic process and dye photosensitization process.

Based on the DRS results, the CB potential (E_{CB}) and VB potential (E_{VB}) of BiOX (X=Cl, Br and I) can be predicted

by the following equations [40,41]:

$$E_{CB} = X - E^e - 0.5E_g, \quad (1)$$

$$E_{VB} = E_g + E_{CB}, \quad (2)$$

where X , E^e and E_g are the electronegativity of the semiconductor, the energy of free electrons on the hydrogen scale (~ 4.5 eV) and the band gap energy of the semiconductor, respectively. The X value for BiOCl, BiOBr and BiOI is about 6.64, 6.45 and 6.20 eV, respectively [40,41]. The E_{CB} and E_{VB} are estimated to be 0.49 and 3.79 eV for BiOCl, 0.54 and 3.36 eV for BiOBr and 0.84 and 2.56 eV for BiOI. In addition, the highest occupied molecular orbital (HOMO) and the lowest unoccupied molecular orbital (LUMO) levels of RhB are about 0.95 and -1.42 eV, respectively [41,42]. Figure 7 shows the energy level diagram of BiOX (X=Cl, Br and I), and also the HOMO and LUMO levels of excited RhB under visible light irradiation (RhB^{*}). It is observed that the HOMO energy level of RhB^{*} molecules is above the CB edge potential of BiOX (X=Cl, Br and I). Therefore, it is expected that the excited electrons in the LUMO level of RhB^{*} can readily be transferred to the CB of BiOX (X=Cl, Br and I), which react with adsorbed O₂ molecules on the photocatalyst surface to generate the superoxide radicals ([•]O₂) for the dye degradation process. However, it is well known that the specific surface area of a semiconductor has a very important role in adsorption of dye molecules and also in the photosensitized degradation process [31,43]. From BET analysis, it is

Figure 7. Schematic diagrams of the energy band structure of BiOCl, BiOBr, BiOI and RhB.

Figure 8. The effect of different scavengers on the photodegradation of RhB over BiOCl photocatalyst under white LED irradiation.

observed that the specific surface area and pore volume of the BiOCl sample are much larger than those of BiOBr and BiOI samples. Therefore, BiOCl could provide more active sites for surface adsorption of dye molecules, and resulting in the enhancement in the degradation process.

Figure 9. The cycle experiments of the photocatalytic degradation of RhB over BiOCl under white LED irradiation.

It is well known that different reactive species contribute in the photocatalytic reactions. In order to determine the mechanism for RhB degradation over the BiOCl photocatalyst under visible light irradiation, the main reactive species in photocatalytic reactions should be specified. The radical trapping experiments were applied to explore the main reactive species. *tert*-Butyl alcohol (TBA), ethylene diamine tetra acetic acid disodium (EDTA-2Na) and benzoquinone (BQ) were used as scavengers of hydroxyl radicals ($\cdot\text{OH}$), holes (h^+) and superoxide radicals ($\cdot\text{O}_2$), respectively. One mM of these scavengers was added into aqueous RhB solution before light irradiation. Figure 8 exhibits the effect of different scavengers on the photocatalytic degradation of RhB over the BiOCl photocatalyst. It is observed that the degradation efficiency of RhB strongly decreased when BQ was used as the scavenger. In contrast, when EDTA-2Na or TBA was added as the additive, the photocatalytic degradation of RhB slightly decreases. As a result, it can be concluded that $\cdot\text{O}_2$ has the crucial role in the photocatalytic reactions for the degradation of RhB molecules, while $\cdot\text{OH}$ and h^+ have little effect on photocatalytic reactions. Thus, the main photodegradation reactions over the BiOCl photocatalyst can be described as below:

Furthermore, to establish the stability of the photocatalytic performance of BiOCl under LED light irradiation, the cycle experiments were carried out to degrade RhB over the BiOCl photocatalyst. Figure 9 displays the photocatalytic degradation of RhB over BiOCl after five cycles. It is observed that

BiOCl is very stable during the photocatalytic process and there is no obvious change between cycles.

4. Conclusion

In conclusion, BiOX ($X = \text{C, Br and I}$) nanostructures were successfully fabricated by a simple precipitation reaction using HX ($X = \text{C, Br and I}$) as the halogen source at a low temperature. The effects of different HX on the morphology, chemical bonding, microstructure and optical properties were investigated by various techniques. Based on the DRS results, the E_g values of BiOCl, BiOBr and BiOI are about 3.30, 2.82 and 1.72 eV, respectively. As a result, BiOBr and BiOI are visible light active semiconductors, while BiOCl can be excited under UV irradiation. The results indicated that BiOCl exhibits much higher photoactivity than BiOBr and BiOI for the degradation of RhB. Based on the experimental results, the mechanism of RhB degradation over BiOCl is attributed to the dye photosensitization process, while the photodegradation over BiOBr and BiOI may be due to both the direct photocatalytic process and dye photosensitization process. The higher photodegradation of RhB over BiOCl is attributed to the excellent dye photosensitization process due to its larger specific surface area ($15.63 \text{ cm}^2 \text{ g}^{-1}$) compared with BiOBr ($9.41 \text{ cm}^2 \text{ g}^{-1}$) and BiOI ($3.68 \text{ cm}^2 \text{ g}^{-1}$). In addition, the photodegradation mechanism investigations indicated that the $\bullet\text{O}_2$ radicals play the crucial role in the photocatalytic reactions for the degradation of RhB molecules over BiOCl.

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