Facile synthesis of bismuth terephthalate metal–organic frameworks and their visible-light-driven photocatalytic activities toward Rhodamine B dye

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ABSTRACT
In this work, the bismuth-terephthalate frameworks (Bi-BDC) were prepared by a facile solvothermal approach. The prepared Bi-BDC MOFs were characterized by XRD, TGA, FT-IR, SEM, and UV-vis DRS analyses. The results indicated that the synthesis temperature affected the crystallinity and morphologies of the Bi-BDC frameworks. Notably, the produced Bi-BDC at low temperatures (80 and 100 °C) exhibited enhanced light absorption ability. The photodegradation experiment of rhodamine (RhB) showed that the prepared Bi-BDC-100 sample had the highest removal efficiency of ca. ∼98% after 270 min of visible LED light illumination. In addition, the HPLC-MS analysis revealed that RhB was photocatalytically degraded over the Bi-BDC via the carboxylation and dealkylation mechanism. Furthermore, the removal efficiency of the organic pollutant was optimized by monitoring the effects of catalyst dosage, dye concentration, and pH media. Finally, cyclic tests implied that the synthesized Bi-BDC MOF had good stability and reusability.

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1. Introduction
Water polluted by textile dyes is considered the most severe environmental issue globally (1–5). Rhodamine B (RhB) is widely used among known dyes, and a large amount is released into the environment. RhB is toxic and can cause skin irritation, allergic dermatitis, and cancer. Furthermore, a significant amount of RhB is released into the environment as metabolites, threatening the ecological environment and human health. Thus, removing RhB residues from the environment has received considerable attention (6–9). Many strategies, such as absorption, biological, advanced-oxidation, and heterogeneous photocatalytic methods, have been developed to remove organic contaminants (10–14). Among these methodologies, photocatalytic degradation is a promising route because it is simple, low-cost, and highly effective (1,15,16). Furthermore, the contaminants can be decomposed into minor toxic inorganic matter, CO2, and water during the photocatalytic process (17–19). Conventional photocatalysts, such as TiO2 (20–22); ZnO (23,24); SiO2 (25), MnFe2O4 (26) or Fe3O4 (27) have been extensively studied for photodegradation of organic dyes in water. Nonetheless, these materials showed particular limitations owing to their low porosities.

Recently, metal–organic frameworks (MOFs), a new porous class material, have emerged as potential photocatalysts owing to their attractive properties such as exceptional high porosity, high crystallinity, and tunable pore structures (28–31). Many MOF structures have been applied for photocatalytic processes, including UiO-66(Zr) (32); MIL-100(Fe) (33), MIL100(Fe)/TNF...
(34), MIL-53(Fe) (35), MIL-125(Ti) (36), MIL-125(Ti)/carbon nanotube (37) and Ag/AgCl@CoFe2O4/NH2-MIL-125(Ti) (38). Recently, bismuth-based MOFs with a high valence state of Bi and flexible coordination geometry are promising materials (39). Vilela et al. (40) synthesized the Bi-AzoBTC framework, showing using environmentally friendly water as the only solvent. Shamaila Iram et al. (41) prepared several Bi-MOFs with different linkers, exhibiting good luminescence and gas adsorption properties. Vinh Huu Nguyen et al. (42) synthesized a bismuth-terephthalate framework, which showed a good photocatalytic performance toward Rhodamine B under visible light irradiation. However, research has found that the synthesis of bismuth-based frameworks remains challenging because their structures are sensitive to experimental conditions, including solvent, temperature, and time (43). Furthermore, it is hard to control crystal the nucleation and growth of Bi-MOF particles during the synthesis, challenging in optimizing their performances (44-46).

Herein, the bismuth-terephthalate framework (Bi-BDC) was simply prepared by solvothermal method at varying reaction temperatures. It was realized that the synthesis temperature strongly affected the revolution of both morphology and crystallinity of the products. Furthermore, the reaction temperature also impacted the Bi-BDC band gap value, tuning their photocatalytic activities toward RhB textile dye under LED visible-light illumination. For the first time, the effects of synthesis conditions on Bi-MOF characteristics and their photo-reduction activities were investigated.

2. Experimental

2.1. Materials

1,4-benzenedicarboxylic acid (H2BDC, ≥98%) and bismuth (III) nitrate pentahydrate (Bi(NO3)3·5H2O, ≥ 99.5%) were obtained from Sigma-Aldrich Co., Ltd. N, N-dimethylformamide (DMF, C3H7NO, 99%), and rhodamine B dye (RhB, C28H31ClN2O3, 95%) were purchased from Xilong Scientific Co., Ltd. All chemicals were used as received without further purification.

2.2. Synthesis of Bi-BDC framework

The Bi-BDC framework was prepared by utilizing the solvothermal method. Typically, Bi(NO3)3·5H2O (6 mmol) and H2BDC (9 mmol) were dissolved in 60 ml DMF with constant stirring. The solution was transferred into a Teflon-lined autoclave (V = 100 ml) and sealed in a stainless-steel vessel. The reactor was heated at an assigned temperature (80 – 120 °C) for 48 h. After the reaction, the reactor was naturally cooled down to room temperature. The product was collected by centrifuged and washed with DMF (60 °C) and ethanol (60 °C) to eliminate the remaining reactants. The obtained solids were degassed at 120 °C under vacuum conditions for 12 h before use.

2.3. Characterizations

The crystallographic structures were analyzed using powder X-ray diffraction (LabX XRD-6100, Shimadzu, Japan). The morphology of the samples was analyzed by field-emission scanning electron microscopy (JEOL JSM 7401F, Tokyo, JAPAN). The functional groups were analyzed employing an FT-IR spectrometer (Tensor 27, Bruker, Germany). The synthesized catalysts’ thermal stability was measured in a nitrogen atmosphere at a heating rate of 10°C/min using a thermogravimetric analyzer (TA Instruments, Linseis, North, USA). Finally, the optical properties were analyzed using UV–Vis DRS (Shimazu UV-2450 UV–Vis spectrophotometer, Kyoto, Japan). Metal leading test was performed by ICP-OES analysis.

2.4. Photocatalytic degradation experiment

The photolytic degradation of RhB dye was processed under irradiation of visible LED light (Six Cree® Xlamp® XM-L2 LEDs, Cree, Inc., Durham, NC, USA) with a power of 10 W and a light output of up to 1052 lm. For each run, 30 mg of material was added to the solution and magnetically stirred for 90 min in the dark. After exposure to visible LED light illumination with continuous stirring, 5 ml of the reaction mixture was withdrawn and centrifuged to recover the clear solution. The content of RhB was measured employing a UV-visible spectrophotometer UV-Vis spectroscopy (Cary 60 UV–Vis, Agilent technologies, US) at the absorption wavelength λmax = 554 nm. The absorbance Ao was measured after stirring for 90 min in the dark and represented for the initial concentration C0 of the solution. The absorbance Af after variable periods of illumination was corresponded to the residual concentration Cf. The photodegradation efficiencies of Bi-BDC materials were calculated as following equation: 

\[ H(\%) = \left( \frac{C_0 - C_f}{C_0} \right) \times 100 = \left( \frac{A_o - A_f}{A_o} \right) \times 100 \]

All experiments were performed in triplicate. The data are expressed as average values of three measurements. For the effect of initial concentration and irradiation time to photodegradation efficiency, the 50 mL of RhB with concentrations in a range of 5–20 mg/mL were added to 30 mg Bi-BDC-100 under irradiation time of
0–360 min. The effect of photocatalyst dosage on the photodegradation of RhB was determined by using various amounts of photocatalyst ranging from 10 to 50 mg with 50 mL of 15 mg/mL RhB solution. The effect of pH on the photodegradation efficiency was examined by mixing 30 mg of Bi-BDC-100 and RhB 50 mL of 15 mg/mL, with the pH ranging from 3 to 11. The NaOH and HCl 0.1 M solutions were used to adjust the pH values.

For investigation of samples recyclability of catalyst, after irradiation time of 360 min, 3 mL of reaction solution was taken at the end of the photocatalytic process and the catalyst was separated through centrifugation. After each working cycle, a definite amount volume of RhB was added to the reaction system in order to keep the reaction solution at 50 mL and 15 mg/mL for using in the future experiment.

3. Results and discussion

3.1. Characterizations

Figure 1 depicts the SEM images of the prepared Bi-BDC-80, Bi-BDC-100, and Bi-BDC-120 samples. At a low temperature of 80 °C, the produced Bi-BDC were highly agglomerated particles (Figure 1a). Further increasing to 100 and 120 °C, the samples were rod-shaped crystals of 2–3 µm in length (Figures 1b and c). Figure 2 shows the XRD patterns of the synthesized materials. The XRD patterns of all samples exhibited the characteristic diffraction peaks at 2θ = 7.5, 9.0, 16.5, and 26.0 °, which were assigned to the Bi-BDC framework.
The FT-IR analyses of the samples were performed, as described in Figure 3. The characteristic peaks at 1580 and 1400 cm\(^{-1}\) were attributed to the asymmetric and symmetric stretching vibration of the COO\(^{-}\) groups in the terephthalate linker. Moreover, the vibrational modes at a lower wavenumber of \(\sim770, 650,\) and 500 cm\(^{-1}\) were assigned to Bi–O bonds (49). The results implied that the Bi-BDC frameworks were successfully prepared under the investigated conditions.

Figure 4 shows the TGA analyses of the synthesized Bi-BDC samples. As revealed, the first weight loss at low temperature (<100 °C) was caused by the evaporation of solvents (41,50,51). The second weight loss occurs between 100 and 250°C was ascribable to the release of the solvent molecules adsorbed within the

![Figure 3. FT-IR spectra of the Bi-BDC frameworks prepared at temperatures of 80, 100 and 120 °C.](image)

![Figure 4. TGA analyses of the prepared Bi-BDC frameworks.](image)

![Figure 5. (a) UV-Vis diffuse reflectance spectroscopy and (b) Energy band spectra of the synthesized samples.](image)

![Figure 6. Absorption spectra of RhB solution under various irradiation time (catalyst dosage: 30 mg/L and 50 mL RhB concentration of 15 mg/mL).](image)
pores of Bi-BDC frameworks. Finally, the significant weight loss at around 380 °C was due to the ligand decompositions. These suggest that the resultant Bi-BDC MOFs are stable up to 380 °C, which is consistent with the previous reports (52).

The optical properties of the prepared Bi-BDC-80, Bi-BDC-100, and Bi-BDC-120 samples were explored via their UV-Vis diffuse reflectance spectra, as described in Figure 5. It can be observed that all samples had absorption edges in the range of 350-400 nm, suggesting the materials were strongly stimulated under UV light. Notably, the absorption edge of the produced Bi-BDC-80 and Bi-BDC-100 slightly shifted toward a higher wavelength region, indicating their higher absorption light ability than their counterpart. We hypothesize that low

Table 1. The photodegradation of RhB dye over various materials.

| Material          | Photocatalytic conditions | Removal efficiency (%) | Refs. |
|-------------------|----------------------------|------------------------|-------|
| BiVO₄             | Visible LED 60 W, 200 mL, 15 ppm, 200 min, m_{catalyst} = 0.1 g | 62.4 | (7)  |
| Bi₂WO₆/UiO-66     | Visible halogen 500 W, 30 mL, 0.03 mM, 180 min, m_{catalyst} = 15 mg | 80.0 | (55) |
| Bi-BDC-MW         | Visible 10 W, 50 mL, 3 x 10^{-5}, 50 mL, 360 min, m_{catalyst} = 9 mg | 99.4 | (42) |
| Bi-BCT-DMF/MeOH   | Visible light 10 W, 50 mL, 3 x 10^{-5}, 50 mL, 360 min, m_{catalyst} = 9 mg | 95.1 | (56) |
| Pt/Zn-Ti LDHs     | Xenon lamp 500 W, 100 mL, 10 mg/L, 60 min, m_{catalyst} = 0.10 g | 90.0 | (57) |
| ZnBi₂O₅₂Bi₂O₃     | Halogen 300 W, 100 mL, 10 mg/L, 60 min m_{catalyst} = 1 g/L | 89.2 | (58) |
| TiO₂-MgFe₂O₄      | Halogen lamp 300 W, 100 mL, 50 mg/L, 90 min, m_{catalyst} = 100 mg | 91.0 | (59) |
| Ag₃PO₄/Bi₂WO₆     | Visible a xenon lamp; 200 mL, 1 x 10^{-5}, 80 min, m_{catalyst} = 0.2 mg | 99.5 | (60) |
| BiOCl/rGO         | wLED1.6 W/cm²; 50 mL, 20 mg/L, 25 min, m_{catalyst} = 50 mg | 98.8 | (61) |
| LDH/Bi₂WO₆       | Metal vapor lamp 400 W, 100 mL, 10 mg/L, 75 min, m_{catalyst} = 100 mg | 98.7 | (62) |
| CM/Bi₂O₅/Br/BiOBr | Blue light 405 nm (20 W), 8 mg/L, 100 min, m_{catalyst} = 17 mg | 97.5 | (63) |
| Bi-BDC-100        | Visible 10 w; 50 mL, 1 x 10^{-5}; 360 min, m_{catalyst} = 30 mg | 98.0 | This study |

Figure 7. (a) Photodegradation profile of RhB under various irradiation time, and (b) photodegradation kinetics of pseudo-first-order (catalyst dosage: 30 mg/L and 50 mL RhB concentration of 15 mg/mL).

Figure 8. The proposed photodegradation mechanism of RhB dye over the prepared Bi-BDC under visible LED light irradiation.
temperature could generate more defect sites within the Bi-BDC framework, which enhanced the light response of the materials. The bandgap energy ($E_g$) values of the samples were obtained from the Tauc plots (Figure 5b). Accordingly, the $E_g$ value of Bi-BDC-80, Bi-BDC-100, and Bi-BDC-120 was 3.55, 3.58, and 3.62 eV, respectively.

### 3.2. Photocatalytic degradation of RhB over Bi-BDC frameworks

The photocatalytic experiments were carried out under LED visible light illumination. Figure 6 shows the UV-vis absorption spectra of RhB as a function of sampling time. It can be seen clearly that the RhB absorption intensity decreased with increasing the sampling time. Moreover, the maximum absorption wavelength of RhB slightly shifted from 554 to 500 nm. This could be due to the decomposition of Rhodamine B to RhB N-demethylated during the photocatalytic process (53). The removal efficiency of RhB over the prepared Bi-BDC MOFs was calculated, and the results are shown in Figure 7. In the pre-adsorption stage, the adsorption of RhB onto the catalyst surfaces reached equilibrium for 90 min. After being exposed to light, the removal efficiency of RhB over the synthesized Bi-BDC catalysts rapidly increased and reached equilibrium after 360 min irradiation (Figure 7a). It can be seen that the Bi-BDC MOF prepared at 100 °C exhibited the best photodegradation performance compared to the others. This could be because the Bi-BDC framework formed at a moderate temperature of 100 °C had optimal crystallinity and bandgap energy. After 360 min of visible light illumination, the total removal yield of RhB over Bi-BDC-80 and Bi-BDC-100 were ~ 98 and 96%, respectively, which are comparable with many previously reported materials (see Table 1). The kinetic study revealed that the degradation rate of RhB over the synthesized catalysts followed the order: Bi-BDC-100 (0.017 min$^{-1}$) > Bi-BDC-80 (0.012 min$^{-1}$) > Bi-BDC-120 (0.008 min$^{-1}$) (Figure 7b). The findings indicated that the RhB contaminant was more rapidly degraded over the Bi-BDC-100 catalyst. The reaction mixture was further analyzed by HPLC-MS analysis to reveal the formation of intermediate products during the photodegradation of RhB over the produced Bi-BDC-100 sample. As described in Figure 8, the m/z values of 443, 399, and 355 were observed on the HPLC-MS spectrum. Under visible light irradiation, RhB initially formed a product with the m/z value of 399 via carboxylation, followed by dealkylation to form a product with the m/z value of

![Figure 9](image-url). Effects of experimental conditions on the removal of RhB over the Bi-BDC-100: (a) catalyst dosage (50 mL RhB concentration of 15 mg/mL), (b) initial RhB concentration (catalyst dosage: 30 mg and 50 mL RhB), and (c) pH media (catalyst dosage: 30 mg and 50 mL RhB concentration of 15 mg/mL).
These intermediate products could further decompose to produce low molecular weight byproducts, CO$_2$ and H$_2$O.

### 3.3. Effects of experimental conditions

Figure 9 shows the effects of internal parameters on the RhB removal efficiency over the synthesized Bi-BDC-100 catalyst. As revealed in Figure 9a, the removal efficiency increased with the catalyst dosage, suggesting more active sites for adsorption and photocatalytic process. Figure 9b shows that the initial contaminant concentration affected the adsorption and photodegradation of the Bi-BDC-100 catalyst. At low concentrations, RhB was decolorized by both adsorption and photodegradation. Contrarily, the adsorption and photooxidation were slowed down when loading a high content of dye owing to the saturation of active sites of the catalyst (7,54). Figure 9c demonstrates that media pH strongly impacted the photocatalytic activity of the sample. Accordingly, the photodegradation of RhB onto the catalyst was favored in the acidic media but reduced in the alkaline media. As RhB is known as a cation dye, the accumulation of RhB onto the catalyst would be accelerated at high media pH (pH >7) because of the electrostatic interaction. However, the adsorption of a high content of organic contaminants on the catalyst’s surface could shield and suppress the catalyst’s absorption light, causing a decrease in photocatalytic performance.

### 3.4. Reusability tests

Reusability is one of the crucial aspects of the catalyst. After each removal experiment, the spent catalyst was collected and washed several times with deionized water, then dried at 120 °C for eight hours before reusing as a regenerated catalyst. Figure 10a shows the removal efficiency of RhB over Bi-BDC-100 after three cyclic experiments. As presented, the removal yield of RhB remained ~93% after three cycles, meaning a slight loss compared to the as-prepared catalyst. Figure 10b shows the XRD pattern of the Bi-BDC-100 catalyst before and after the reaction. As revealed, the crystallinity of Bi-BDC-100 remained unchanged during the photocatalysis process. Moreover, the metal ion leading tests at pH = 3, pH = 7, and pH = 11 revealed no trace of Bi in the solution. These findings suggest that the synthesized Bi-BDC MOF was highly stable.

### 4. Conclusions

In conclusion, the Bi-BDC frameworks were simply synthesized by solvothermal synthesis at various temperatures. The results indicated that the synthesis temperature strongly impacted the morphology, crystallite size, and bandgap energy of the produced materials. With increasing the temperature from 80 to 120 °C, the bandgap of the sample increased from 3.55–3.62 eV, respectively. The Bi-BDC prepared at 100 °C exhibited the most excellent photo-reduction rate of ~0.017 min$^{-1}$ toward RhB dye under visible LED light conditions. After 360 min irradiation, the photodegradation efficiency over the Bi-BDC catalyst reached ~98%. Moreover, the synthesized Bi-BDC maintained high photodegradation efficiency and good stability after four cycles. The findings implied that the photocatalytic activities of the Bi-BDC frameworks could be tuned by controlling the synthesis temperature.
Author contributions

Material preparation, data collection and analysis were performed by Pham Hoang Ai Le, Trinh Duy Nguyen, and Huynh Huu Khanh. Writing-original draft, writing-review and editing were conducted by The Ky Vo, Thai Son Pham Quynh and Van-Cuong Nguyen. Data curation, software, and validation were performed by The Ky Vo and Van-Cuong Nguyen. All authors have read and agreed to the published version of the manuscript.

Disclosure statement

No potential conflict of interest was reported by the author(s).

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