



Degradation of Aniline by Bismuth Oxyiodide (BiOI) under Visible Light Irradiation



ABSTRACT

Visible-light-driven porous spherical photocatalyst bismuth oxyiodide (BiOI) was successfully prepared by facial hydrothermal synthesis. The obtained catalyst was characterized using x-ray diffraction (XRD), ultraviolet-visible diffuse reflection (UV-Vis DRS) and scanning electron microscope (SEM). The experiment which used the visible-driven catalyst to degrade aniline wastewater investigated the effects of initial concentration of aniline and reaction time on the removal efficiency of aniline. The highest photodegradation efficiency of aniline was 97.3% with a BiOI dosage of 1 g L⁻¹ and initial aniline concentration of 50 mg L⁻¹ after 2 h of visible light irradiation. The chemical oxygen demand (COD) of the solution decreased substantially and the high chemical oxygen demand removal efficiency achieved 71%. In addition, the investigation of different scavengers demonstrated that h⁺ and •O₂⁻ are the main reactive species in the photodegradation of aniline. The fate of nitrogen was investigated by ion chromatography. The high photodegradation and mineralization capability efficiency suggest BiOI is a promising photocatalyst for the degradation of organic pollution in the practical application.

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INTRODUCTION

Aniline, as an important organic chemical raw material and chemical product, is widely used in dyes, pharmaceuticals, pesticides, explosives and rubber, which seriously pollutes the environment and is harmful to human's health (Ye *et al.* 2009). It has been listed as one of the 129 priority pollutants by U.S. Environmental Protection Agency (Li and Xie 2007) and it has been included in China's "blacklist of environmental priority pollutants" (Zhang *et al.* 2007). Aniline, containing a benzene ring, is too stable to resist degradation. The photocatalytic method is one of the most advanced oxidation process (AOPs) for the degradation of aniline in aqueous solutions (Calza *et al.* 2004; Baran *et al.* 2006; Hu *et al.* 2007). This method is cheap and chemically stable. However, titanium oxide (TiO₂), a commercially-used photocatalyst may have some limitations, as it is only stimulated under the UV light and has a fast recombination of photo-generated electron-hole pairs (Gomez-Solis *et al.* 2012). Therefore, the development of photocatalyst that respond to visible light is necessary to maximize the use of visible light.

Visible photocatalytic oxidation technology, is an environment-friendly technology, that has caught the

attention of the public with the advantages which are demonstrated as it breaks toxic and hazardous organic pollutants (Jiang *et al.* 2015). Due to the wide band gap, traditional photocatalyst needs to be activated through ultraviolet that limits its application (Chou and Chen *et al.* 2015). Therefore, it is necessary to research and develop some new efficient visible-light-driven photocatalysts to replace it. Some relevant researches in this area have become a hot topic in recent years.

Recently, bismuth oxyhalides (BiOX, X=F, Cl, Br, I), ternary oxide semiconductors, are considered as candidates for photocatalytic applications owing to its layer structure and remarkable photocatalytic activities (Paola *et al.* 2012). The band gap values of BiOX was approximately 3.19, 2.75 and 1.76 eV for bismuth oxihloride (BiOCl), bismuth oxibromide (BiOBr) and bismuth oxyiodide (BiOI), respectively (Chang *et al.* 2010). Among them, BiOI, with the narrow band gap, has the strongest absorption under visible light irradiation. Researches mostly focus on the synthetic method of BiOX and commonly choose universal organic pollutants, such as methyl orange (MO) and rhodamine B (Rh B), to investigate their photocatalytic

performance (Su *et al.* 2010; Wu *et al.* 2011; Jiang *et al.* 2010; Shi *et al.* 2012).

The study of degradation of aniline with BiOI, a stubborn environmental pollutant, could promote photocatalytic applications in wastewater treatment. In this study, BiOI is synthesized and characterized by X-ray diffraction (XRD), ultraviolet-visible diffuse reflection spectrum (UV-Vis DRS) and scanning electron microscope (SEM). The aniline aqueous solution containing BiOI was degraded under visible light as well as the degradation or mineralization efficiency of aniline were analyzed by UV-Vis Spectrophotometry, ion chromatography and the measure of chemical oxygen demand (COD). The effects of aniline initial concentration and catalyst dosage were also investigated.

MATERIALS AND METHODS

Chemicals

Aniline, bismuth nitrate pentahydrate ($\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$), potassium iodide (KI), polyvinyl pyrrolidone (PVP-K30) and TiO_2 P25 were all purchased from Aladdin Chemistry Co. Ltd. Ammonium oxalate and benzoquinone were purchased from Tianjin Kemiou Chemical Reagent Co., Ltd., isopropanol was bought from Tianjin Yongda Chemical Reagent Co. Ltd. All of the solvents and chemicals used in the experiment were of analytical grade and used without further purification.

Synthesis and Characterization of photocatalyst

The synthesis of BiOI was adopted from Hao *et al.* (2012). Aniline, bismuth nitrate pentahydrate ($\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$) and KI were added in a 50 ml aqueous solution containing PVP 6.7 g L^{-1} . The suspension was magnetically stirred in the dark for 10 min and then transferred into a 50 ml Teflon-lined stainless steel autoclaved up to 80% of the total volume. The autoclave was heated at 150°C for 1 h and then naturally cooled to room temperature. The precipitates were collected by centrifugation and washed by deionized water for several times, and then dried at 70°C for 10 h.

X-ray powder diffraction (XRD) analysis of the as-prepared samples was carried out on a Bruker D8 Advanced X-ray diffractometer using $\text{Cu K}\alpha$ radiation ($\lambda = 0.15442 \text{ nm}$) at a scanning rate of 5° min^{-1} in the 2θ range of 10 – 80° . Scanning electron microscopy (SEM) images were observed on a Philips Quonxe-2000 microscope. UV-vis diffuse reflectance spectroscopy (UV-Vis DRS) results of the samples were performed in the range of 200 – 800 nm on a Hitachi U-3010 spectrophotometer equipped with an

integrating sphere attachment. Barium sulfate (BaSO_4) was used as a background in the experiment. The morphology and chemical composition of the samples was investigated by transmission electron microscope (TEM) (FEI Ltd., TECNAIG2F20-S-TWIN) using an acceleration voltage of 200 kV .

Photocatalytic experiments

The visible photo degradation of aniline was carried out under simulated visible light irradiation. Simulated solar irradiation was provided by a 350 W xenon lamp (Nanjing Xujiang Machine-electronic Plant, China) with a 420 nm cutoff filter (Nanjing Xujiang Machine-electronic Plant, China). A certain amount of BiOI sample (ranging from 0.20 to 4.0 g L^{-1}) was added into 50 ml of aniline solution with different initial concentrations (varying from 10 to 90 mg L^{-1}). Before irradiation, the suspension was magnetically stirred in the dark for 1 h to achieve the aniline adsorption-desorption equilibrium. Then the solution was exposed to visible light for 2 h . At given time intervals, about 4 mL of the suspension was taken out and passed through a $0.45 \mu\text{m}$ fiber for analysis.

Concentrations of aniline in all aqueous solutions were analyzed with a Shimadzu LC-20A high performance liquid chromatography (Japan) equipped with UV detector (SPD-M20A, Shimadzu, Japan) set at 236 nm . The column used was a reversed-phase C18 column. Mobile phase was a mixture of acetonitrile-water ($55:45, \text{V}:\text{V}$) and the flow rate was set at 0.50 mL min^{-1} . The peak identification was based on the retention time and the UV spectrum of external standards.

Chemical oxygen demand (COD) was measured with potassium dichromate after the sample was digested with WMX-IIA COD microwave digestion system (Shaoguan keli experimental instrument Co. Ltd. China). Concentration of NO_3^- was determined by ion chromatography on a model system (Dionex ISI-900) with anion chromatography column (AS-14), anion guard column (AG-14) and chemical suppressor (AMMS 30). The $8.0 \text{ mM Na}_2\text{CO}_3/1.0 \text{ mM NaHCO}_3$ acted as a leachate with 0.8 mL min^{-1} of flow velocity and dilute sulphuric acid as a regeneration solution.

RESULTS AND DISCUSSION

Characterization of BiOI

Crystal structure. The XRD patterns provide the crystal structure and phase information of the as-synthesized samples. In the full-range of 10 – 80°C , the diffractive patterns of the samples are quite in similar angles compared

with those of the standard tetragonal structure for BiOI (JCPDS No. 73-2062), but the peaks of BiOI are broader, reflecting its smaller particle size (**Figure 1**). The particle size can be calculated based on Scherrer's formula. (Shen 2008):

$$D = K\lambda / \beta \cos \theta \quad (1)$$

Where D is the average particle size, K is the constant ($K=0.890$), λ is the X-ray wavelength, θ is the diffraction angle and β is the full-width at half maximum. The average particle size of BiOI is 14 nm, which is in consistency with the result reported by the previous literature (Hao and Xiao *et al.* 2012).

Photoabsorption property. The sample exhibit strong absorption in the visible light region with an absorption edge at 700 nm (**Figure 2**). The band gap energy (E_g) can be estimated according to the equation of $E_g = 1240 \lambda^{-1}$ and the band gap energy is calculated to be 1.77 eV which is close to Chang *et al.* (2010) research ($E_g = 1.76$ eV). The band gap of BiOI has an obvious blue shift compared with that of TiO_2 , indicating that the as-synthesized BiOI has suitable band gap energy for photocatalytic decomposition of organic contaminants under visible light irradiation.

Surface morphology

The SEM images demonstrated that BiOI microspheres could be obtained when the reaction was performed in optimal conditions and the photocatalyst BiOI is synthesized by a controllable method for the preparation of inorganic materials in hydrothermal conditions (**Figure 3**).

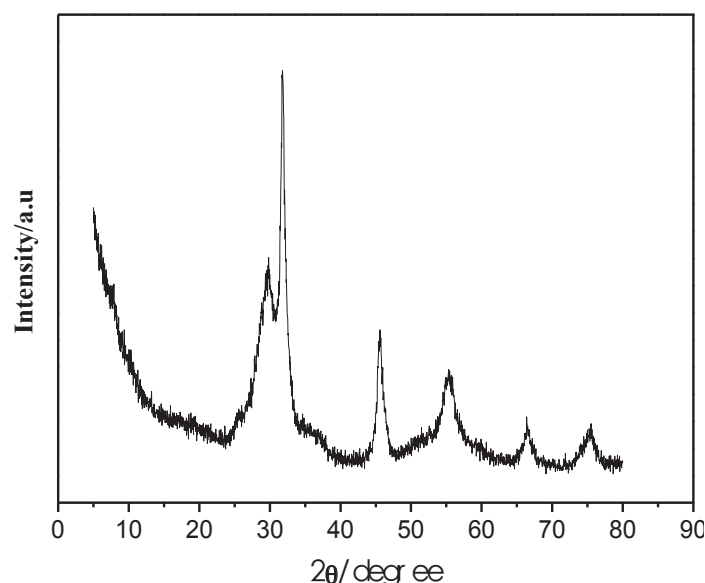


Figure 1. XRD pattern of the BiOI and standard tetragonal structure.

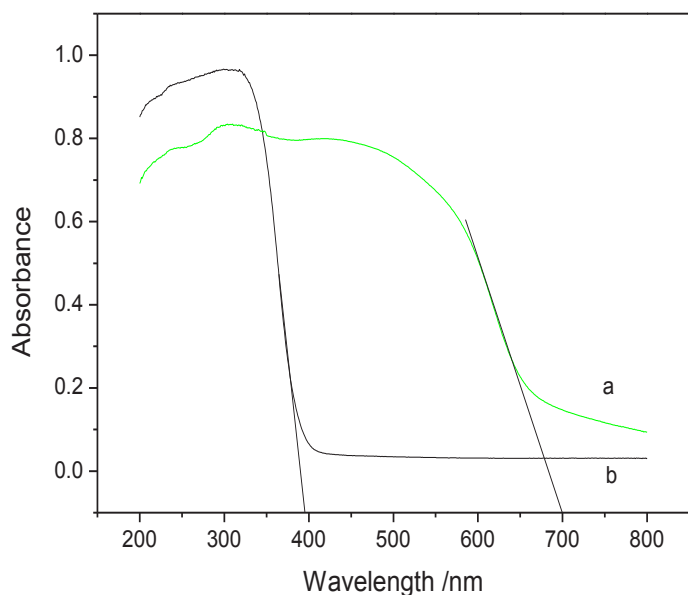


Figure 2. UV-Vis diffuse reflectance spectra of (a) TiO_2 and (b) BiOI.

The observation suggests that the main morphology of the sample consists of a large quantity of microspheres with an average thickness of 1.6 μm and even spherical size (**Figure 3A**). Especially, from the high-magnification SEM image (**Figure 3C**), the sample is ideal microspheres. The highest magnification SEM image (**Figure 3D**) exhibits the hollow architecture of the microspheres BiOI, demonstrating that the BiOI has large specific surface area and high adsorption property.

TEM and EDX analysis. The morphological structure of BiOI sample was further analyzed by TEM (**Figure 4A and B**). The TEM image (**Figure 4A**) shows that microspheres are composed of nanorod. The as-prepared BiOI sample owns hollow architecture, which is consistent with the SEM observation (**Figure 4B**). The elemental mapping analysis (**Figure 4c to f**) confirms that Bi, O and I existed in the catalyst. In addition, the EDS spectrum demonstrates that the elements of O, Bi, I, and Cu were detected in the BiOI sample, where Cu comes from copper grid.

Photocatalytic degradation of aniline

Evaluation of the photocatalytic activity. The time dependence of the removal ratio of aniline on the BiOI under visible-light irradiation was investigated and compared with TiO_2 P25 (a commercial, highly active, mixed phase titania photocatalyst). The degradation curve of aniline sample with that of TiO_2 P25 and without any photocatalyst (Blank) due to the restriction was plotted (**Figure 5**). It is clearly shown that aniline barely degraded without any photocatalyst (Blank), whereas the BiOI exhibited a much higher activity than that with TiO_2 P25 under visible-

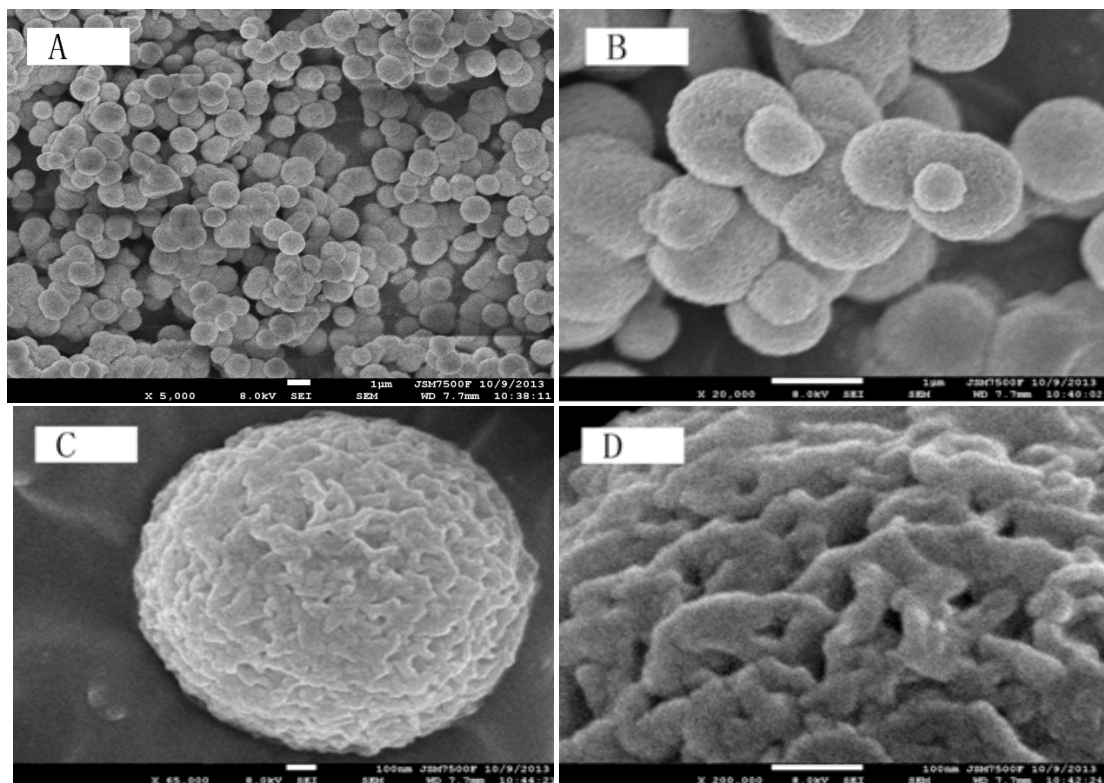


Figure 3. The Surface morphology of BiOI.

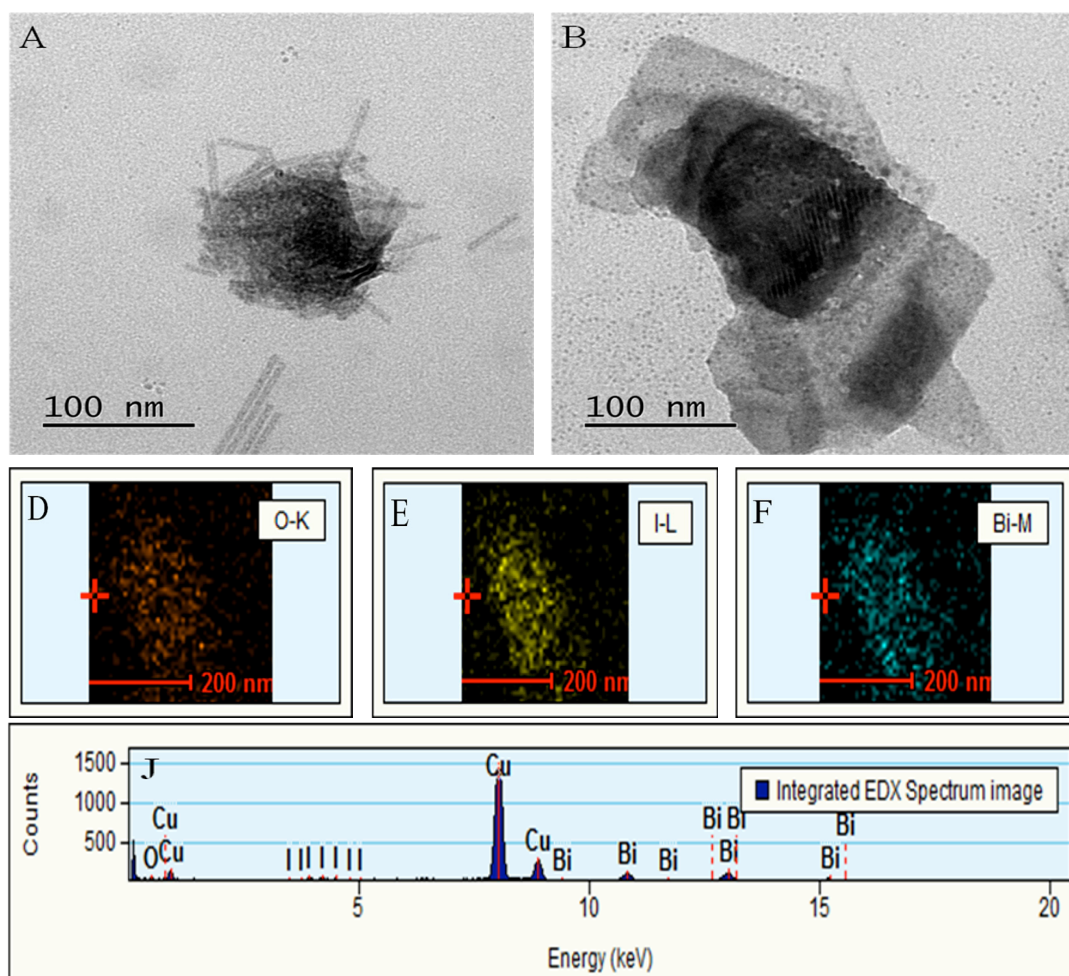


Figure 4. TEM images of BiOI (A, B); mapping of O (D), I(E), Bi(F); EDX spectrum of BiOI(J).

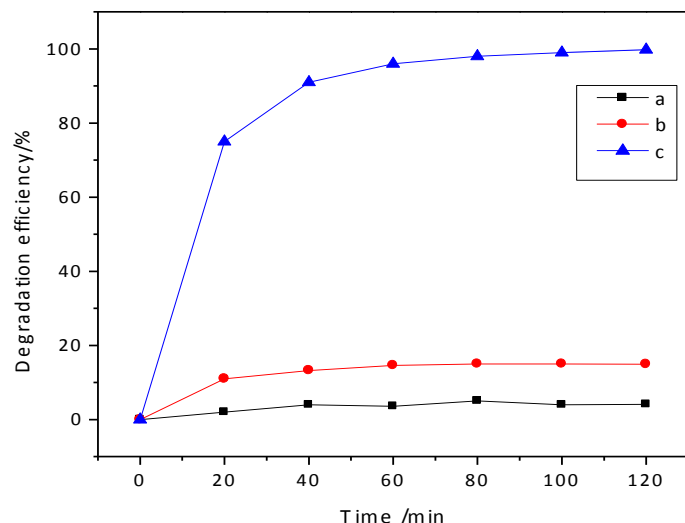


Figure 5. Degradation of aniline as a function of irradiation time at 1 g L^{-1} catalyst loading, 50 mg L^{-1} initial aniline concentration in the system of (a) no catalyst/visible light, (b) TiO_2 P25/visible light, (c) BiOI/visible light.

light exposure. After irradiation for 120 min, only 15% of aniline was removed by Degussa P25 while the removal efficiency of the as-synthesized BiOI reached 99%, which demonstrates that the BiOI is an excellent visible-light-driven photocatalyst. The low visible light photocatalytic activity of TiO_2 P25 is attributed to limited utilization of visible light because of its large band gap (Xiao *et al.* 2012).

Effects of the initial conditions on photocatalytic degradation. Different initial concentrations of aniline solutions, ranging from 10 to 90 mg L^{-1} , were used to observe the effect of varying aniline concentration on the degradation efficiency. The results of different aniline initial concentration on the degradation efficiency clearly show that the lower the aniline concentration, the higher the efficiency of the aniline decomposition (Figure 6). An explanation for this result is that as the initial concentration of aniline increases, more and more organic substances can be adsorbed on the surface of photocatalyst, thus the photogeneration of reactive oxygen species will be reduced since there is overloading of the active sites. In addition, increasing aniline concentration leads to an increase of the amount of incident photons which are absorbed by the pollutant molecules and never reach the photocatalyst surface (Konstantinou and Albanis 2004, Behnajady *et al.* 2006).

In addition, the effect of the catalyst dosage on the degradation efficiency was also studied by varying the catalyst amounts from 0.2 to 4.0 g L^{-1} at 1 g L^{-1} catalyst loading after 2 h of simulate illumination irradiation. The degradation efficiency of aniline increased significantly

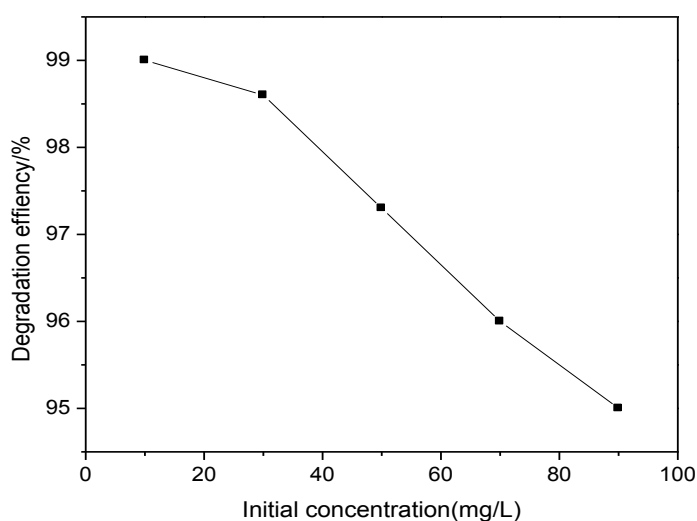


Figure 6. The effect of different aniline initial concentration on the degradation efficiency at 1 g L^{-1} catalyst loading, 2h of simulate illumination irradiation.

as the catalyst concentration increased from 0.2 to 1 g L^{-1} and then decreased slightly with a further increase in the catalyst concentration (Figure 7). It is not difficult to understand that a lower catalytic activity exhibited a low catalyst dosage because of the smaller amount of catalytic active sites. However, if the dosage is much higher than the optimal value, an increase in the opacity and light scattering of the catalyst and a decrease in the number of catalytic surface active sites will arouse the degradation efficiency of the aniline to no longer increase or even decline. This is likely caused by aggregation of the catalyst particles (Gondal *et al.* 2010). Therefore, 1 g L^{-1} was selected as the optimal catalyst dosage of the photocatalytic reaction.

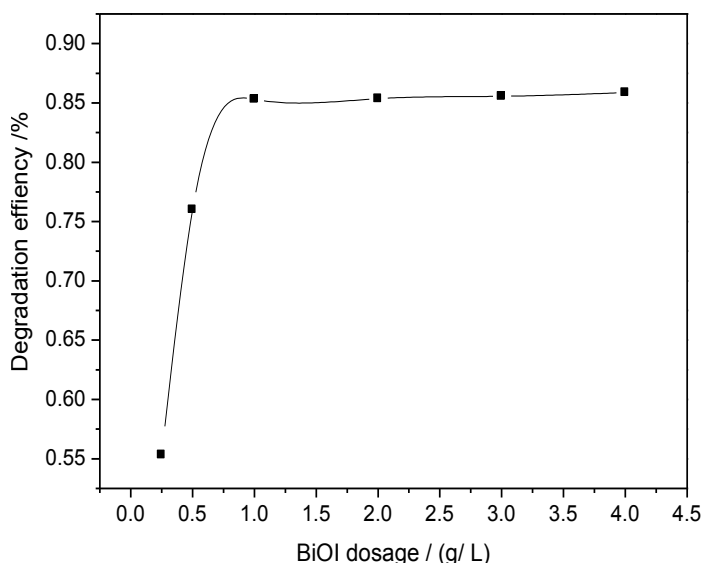


Figure 7. Effect of catalyst dosage (initial aniline concentration = 50 mg L^{-1} , irradiation time = 2h).

The mineralization of aniline. It is well known that the complete decomposition of photocatalytic reaction is to generate CO_2 and ions. The extent of mineralization of aniline is studied following the equation of Kaniou *et al.* (2005). In the wastewater treatment field, The removal of COD is more important than degradation efficiency of aniline as COD value can be used to evaluate the amount of total organic compounds including the organic intermediates formed in the reaction. Therefore, the change of COD can reflect the mineralization of wastewater (Wu and Englehardt 2015).

The concentration of COD was chosen as a mineralization index to characterize the aniline degradation. It is observed that 71% of the COD was eliminated after 140 min of irradiation, indicating that a portion of the aniline were mineralized in this process, which is important for the practical application of BiOI photocatalysts to avoid secondary pollution (Figure 8A).

While significant decrease of the aniline concentration is in the first 20 min, the COD removal of aniline solution

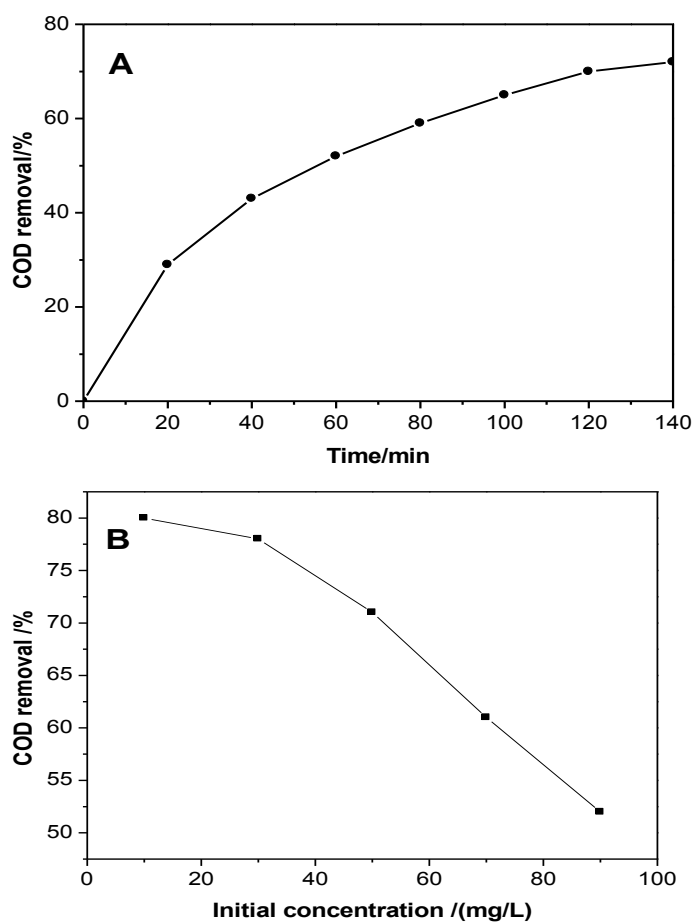


Figure 8. COD removal of the solution as a function of (A) irradiation time and (B) initial concentration of aniline (catalyst dosage=1 g L⁻¹).

reduced slowly, which shows that macromolecule of aniline is destroyed at the first step of the degradation and further oxidation of intermediate requires longer illumination.

It is observed that during the experiment, the COD decreases substantially with the increase of aniline concentration and the final COD removal of the initial 10, 30, 50, 70 and 90 mg L⁻¹ aniline concentration are 80%, 78%, 71%, 61% and 52%, respectively, exhibiting high mineralization of BiOI at low concentration (Figure 8B).

When the initial concentration is 50 mg L⁻¹, the organic nitrogen conversion to the NO_3^- is an efficient process and about 60% of the nitrogen is recovered in the form of NO_3^- , indicating other nitrogen-containing organic compounds still exist in the solution (Figure 9). In the process of photocatalytic oxidation of aniline, the predominant inorganic nitrogen is NH_4^+ ions while NO_3^- is the predominant inorganic nitrogen in the BiOI/visible light system that was used in the study. This indicates a stronger oxidizing ability of BiOI (Kaniou *et al.* 2005). Other nitrogen-containing organic compounds are still present in the solution. The other nitrogen elements of aniline may exist in the way of NH_4^+ or organic nitrogen (Nitoi *et al.* 2015). The final oxidation states of nitrogen on the photo-degradation also depend on the experimental concentration and nature of the substrate.

Photocatalytic mechanism

It is well known that a series of reactive species

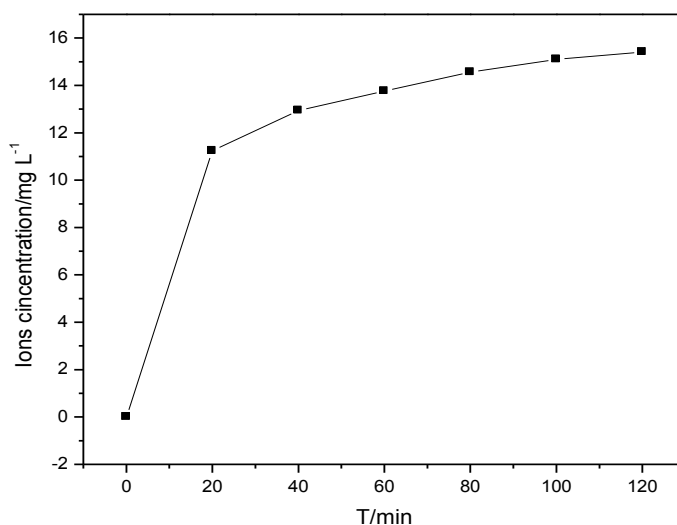


Figure 9. NO_3^- formation as a function of irradiation time at 50 mg L⁻¹ aniline initial concentration, 1 g L⁻¹ catalyst dosage and 2 h of simulate illumination.

including hydroxyl radicals ($\bullet\text{OH}$), active holes (h^+) and superoxide radicals ($\bullet\text{O}_2^-$) play an essential role in the process of photodegradation of organic pollutants. Various trapping agents (6 mmol L⁻¹ ammonium oxalate for h^+ , 1 mmol L⁻¹ benzoquinone for $\bullet\text{O}_2^-$, 10 mmol L⁻¹ isopropanol for $\bullet\text{OH}$) were introduced in the photocatalytic degradation of aniline to clarify the underlying photocatalytic mechanism (Lin *et al.* 2012). The photodegradation of aniline were not suppressed largely by the addition of $\bullet\text{OH}$ scavenger isopropanol, indicating that the free hydroxyl radicals was not the main reactive species in the oxidation process (Figure 10). While the photodegradation was inhibited significantly in the presence of benzoquinone and ammonium oxalate, $\bullet\text{O}_2^-$ and h^+ were the main reactive species in the photodegradation of aniline. A possible photocatalytic pathway of the BiOI sample was proposed as follows:

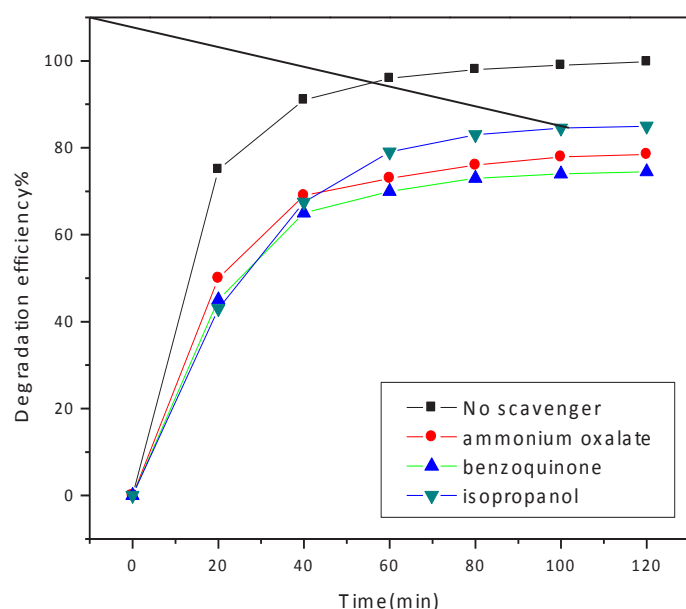
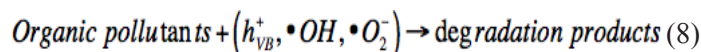
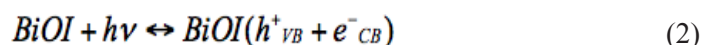


Figure 10. Effects of different scavengers on degradation of aniline in the presence of BiOI.

CONCLUSIONS

In the study, the degradation of aniline under visible light by BiOI has been demonstrated. Under the optimization of experimental condition of 50 mg L⁻¹ aniline concentration and 1 g L⁻¹ catalyst dosage, the aniline concentration decreases quantitatively after 2 hours of visible light irradiation. COD removal shows little intermediate organic species remain in the solution. The organic nitrogen is recovered in the form of nitrate. The system of BiOI could be applied in the elimination of aniline under visible light illumination, exhibit high photodegradation efficiency and good mineralization capacity. In addition, the quenching investigation of different scavengers demonstrated that h^+ and $\bullet\text{O}_2^-$ reactive species are the main reactive species in the photodegradation of aniline. The degradation path of aniline is not yet clear, which needs further researches. The degradation of aniline by BiOI provides a potential direction of practical application of organics removal in wastewater.

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