



Short communication

## Synthesis of AgCl/Bi<sub>3</sub>O<sub>4</sub>Cl composite and its photocatalytic activity in RhB degradation under visible light

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## ABSTRACT

This work presents a novel composite photocatalyst, AgCl/Bi<sub>3</sub>O<sub>4</sub>Cl, which was prepared using an ion-exchange method. The synthesized composite was characterized by various techniques and its photocatalytic activity was investigated in RhB degradation under visible light irradiation. Results indicated that the introduction of AgCl into Bi<sub>3</sub>O<sub>4</sub>Cl promoted the specific surface area, light absorption performance and the separation efficiency of electron–hole pairs, which resulted in a high photocatalytic activity of the composite. The optimal AgCl/Bi<sub>3</sub>O<sub>4</sub>Cl sample showed a RhB degradation rate of 0.048 min<sup>-1</sup>, which was 2.2 and 2.4 times higher than those of AgCl and Bi<sub>3</sub>O<sub>4</sub>Cl, respectively.

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## 1. Introduction

In recent decades, with the rapid development of industry, science and technology have brought huge economic benefits as well as serious environmental pollution to people. Photocatalysis is considered as the most potential technology to resolve the issue of environmental purification and thus gains much attention. TiO<sub>2</sub> is one of the most studied photocatalyst due to its characteristics of strong photoactivity, high stability and non-toxicity [1]. However, TiO<sub>2</sub> can only absorb <4% of the solar radiation that reaches the earth's surface. This demerit seriously limits its practical application and triggers scientists to discover an active visible-light-driven photocatalyst, such as Ag<sub>3</sub>PO<sub>4</sub> [2], g-C<sub>3</sub>N<sub>4</sub> [3], CaBi<sub>6</sub>O<sub>10</sub> [4] etc.

Bi<sub>3</sub>O<sub>4</sub>Cl is a visible-light-driven semiconductor photocatalyst with an optical band gap of 2.76 eV [5]. Meanwhile, the uniquely layered structure makes the semiconductor hold strong ability in separation of photogenerated charge carriers, which induces its good photocatalytic activity. However, for the purpose of application in industry, the modification is still needed to further improve the photoactivity. For example, Chakraborty et al. prepared WO<sub>3</sub>/Bi<sub>3</sub>O<sub>4</sub>Cl composite and studied the promotion effect of WO<sub>3</sub> [6]. Gao et al. reported that the coupling of BiOCl with Bi<sub>3</sub>O<sub>4</sub>Cl efficiently increased the photocatalytic activity in removal of isopropanol gas [7]. AgCl has been reported as an efficient photocatalyst [8]. Additionally, the plasmonic photocatalyst has the property to act as the co-catalyst. A few of correlative photocatalysts have been synthesized, such as AgCl/SmVO<sub>4</sub> [9] and AgCl/ZnO [10], and they are efficient in degradation of the organic dye. The result

indicates that AgCl may be another good co-catalyst for Bi<sub>3</sub>O<sub>4</sub>Cl photocatalyst.

Based on the above analysis, it is inferred that AgCl/Bi<sub>3</sub>O<sub>4</sub>Cl can be a potential candidate for photocatalysis, and AgCl/Bi<sub>3</sub>O<sub>4</sub>Cl has not been reported ever before. This expectation leads us to prepare a novel photocatalyst AgCl/Bi<sub>3</sub>O<sub>4</sub>Cl via a simple precipitation method followed by ion exchange between Bi<sub>3</sub>O<sub>4</sub>Cl and AgNO<sub>3</sub> solution. Rhodamine B (RhB) aqueous solution is chosen as the model compound to estimate the photocatalytic activity of the composite. The experimental result shows that the loading of a small amount of AgCl greatly enhances the photocatalytic ability of Bi<sub>3</sub>O<sub>4</sub>Cl.

## 2. Experimental section

## 2.1. Catalyst preparation

All chemicals are purchased commercially and used without any further purification. Pure AgCl was synthesized via a precipitation method with AgNO<sub>3</sub> and NH<sub>4</sub>Cl as the raw materials [9]. BiOCl was prepared using the same method except that the raw materials were Bi(NO<sub>3</sub>)<sub>3</sub>·5H<sub>2</sub>O and hydrochloric acid [11]. Bi<sub>3</sub>O<sub>4</sub>Cl powder with pale-yellow color was synthesized by the solid reaction between Bi<sub>2</sub>O<sub>3</sub> and the prepared BiOCl at 700 °C for 6 h [5]. AgCl/Bi<sub>3</sub>O<sub>4</sub>Cl photocatalyst was synthesized by an ion-exchange method. In a typical process, 0.0203 g AgNO<sub>3</sub> was dissolved in 80 mL de-ionized water with magnetic stirring at room temperature. Then, 1.0 g Bi<sub>3</sub>O<sub>4</sub>Cl was added and stirred for 30 min. Afterwards, the mixture was poured into a Teflon-lined stainless-steel autoclave (100 mL capacity) and incubated in an oven at 80 °C for 4 h. After completion of the reaction, the precipitates were collected by centrifugation, washed several times with water and

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ethanol, and finally dried at 60 °C to obtain 8% AgCl/Bi<sub>3</sub>O<sub>4</sub>Cl composite. Other AgCl/Bi<sub>3</sub>O<sub>4</sub>Cl samples with different AgCl molar content were prepared using the same method.

## 2.2. Photocatalytic reaction

The photocatalytic activities of the AgCl/Bi<sub>3</sub>O<sub>4</sub>Cl photocatalysts were tested by the degradation of Rhodamine B (RhB) under visible-light irradiation. A 350 W spherical Xe lamp equipped with UV and Infrared filters (420 nm <  $\lambda$  < 800 nm) was employed as the light source. The volume of the RhB solution is 100 mL and the catalyst amount is 0.2 g. Prior to irradiation, the mixture was agitated for an hour to ensure adsorption/desorption equilibrium at room temperature. At given irradiation time intervals, samples were periodically withdrawn and centrifuged to remove photocatalyst for analysis. The filtrates were then analyzed by a UV–vis spectrophotometer ( $\lambda = 554$  nm) to determine the RhB concentration. The examination experiment process of reactive species is similar to the photodegradation experiment. A quantity of scavengers was introduced into the RhB solution prior to addition of the catalyst. Chemical oxygen demand (COD) was measured using a potassium permanganate solution as the oxidizing agent in a strong acid medium.

The information about the characterizations of the synthesized samples is listed in supplementary materials.

## 3. Results and discussion

The BET experiment indicates that both Bi<sub>3</sub>O<sub>4</sub>Cl (0.4 m<sup>2</sup>/g) and AgCl (0.2 m<sup>2</sup>/g) exhibit significantly low specific surface areas, whereas the surface area of AgCl/Bi<sub>3</sub>O<sub>4</sub>Cl composite is higher than pristine materials. For the samples of AgCl/Bi<sub>3</sub>O<sub>4</sub>Cl with the AgCl content of 2%, 4%, 6%, 8% and 10%, the BET value is 1.9, 1.4, 1.5, 2.4 and 1.2 m<sup>2</sup>/g, respectively. The increased surface area suggests that the AgCl/Bi<sub>3</sub>O<sub>4</sub>Cl samples may hold stronger ability for adsorption of reactants, which is beneficial for the photocatalytic reaction.

The photoabsorption performance of AgCl/Bi<sub>3</sub>O<sub>4</sub>Cl composites was characterized by a UV–vis diffuse reflectance spectrophotometer (DRS). As shown in Fig. 1a, the absorption edge of Bi<sub>3</sub>O<sub>4</sub>Cl locates at 456 nm which is larger than that of AgCl (410 nm), indicating its stronger photoabsorption ability for visible light. Via the equation of  $E_g = 1240 / \lambda$ , the optical band gap of Bi<sub>3</sub>O<sub>4</sub>Cl and AgCl can be estimated to be 2.71 and 3.02 eV, respectively. Both of them are consistent with the previous results [5,9]. The introduction of AgCl into Bi<sub>3</sub>O<sub>4</sub>Cl can promote the light absorption ability slightly, which may be ascribed to the surface plasmon resonance (SPR) effect of Ag nanoparticles (NPs) since Ag NPs are usually accompanied with AgCl [9,10]. With the increases of AgCl content, the photoabsorption performance of AgCl/Bi<sub>3</sub>O<sub>4</sub>Cl is improved. Fig. 1b shows the X-ray diffraction (XRD) patterns of Bi<sub>3</sub>O<sub>4</sub>Cl, AgCl, and 8% AgCl/Bi<sub>3</sub>O<sub>4</sub>Cl composite. In order to make clarity, only the three samples are listed. Pure Bi<sub>3</sub>O<sub>4</sub>Cl has six strong peaks at  $2\theta = 23.9, 29.1, 29.7, 31.4, 31.6$  and  $45.3^\circ$ , indicating its monoclinic structure (PDF#36-0760). AgCl is in its cubic structure (PDF#31-1238) and shows the characteristic peaks at  $27.8, 32.2, 46.2$  and  $54.8^\circ$ . For AgCl/Bi<sub>3</sub>O<sub>4</sub>Cl composite, the XRD patterns are nearly the same as those of pure Bi<sub>3</sub>O<sub>4</sub>Cl except for the two weak peaks at  $27.8$  and  $32.2^\circ$ , indicating the formation of AgCl via the ion exchange during the hydrothermal process.

The morphology of AgCl/Bi<sub>3</sub>O<sub>4</sub>Cl was investigated using scanning electron microscope (SEM) and transmission electron microscopy (TEM). Fig. 2 shows the SEM and TEM images of pure Bi<sub>3</sub>O<sub>4</sub>Cl and 8%AgCl/Bi<sub>3</sub>O<sub>4</sub>Cl. Pure Bi<sub>3</sub>O<sub>4</sub>Cl appears to be irregular blocks with a size of 2–4  $\mu\text{m}$ . These blocks are composed of a lot of nanoflakes (Fig. 2a), corresponding to the layer structure of Bi<sub>3</sub>O<sub>4</sub>Cl. The high resolution (HR) TEM further proves that these flakes have smooth surface (Fig. 2b). Different from Bi<sub>3</sub>O<sub>4</sub>Cl, AgCl/Bi<sub>3</sub>O<sub>4</sub>Cl composite displays a three-dimensional hierarchical micro/nanostructure consisted of

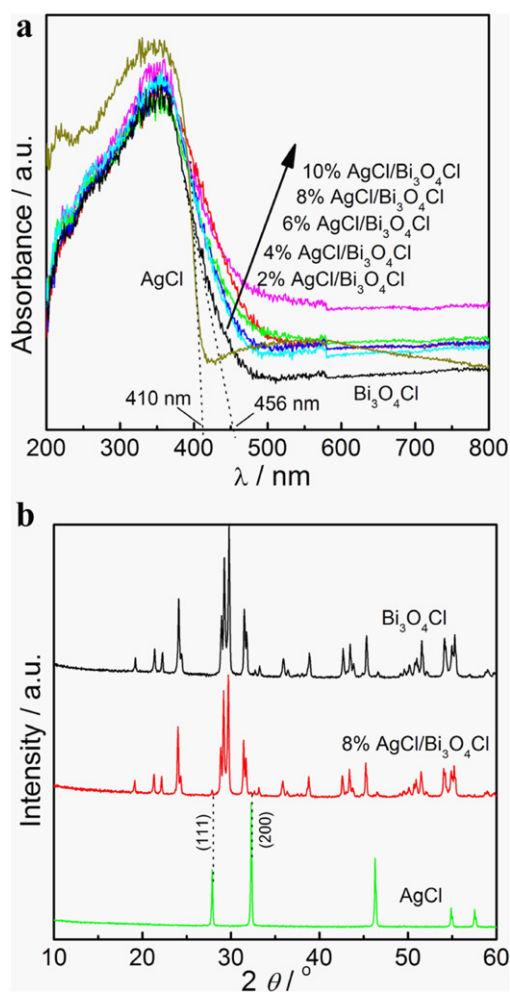


Fig. 1. UV–vis spectra of AgCl/Bi<sub>3</sub>O<sub>4</sub>Cl photocatalysts (a) and XRD patterns of Bi<sub>3</sub>O<sub>4</sub>Cl, AgCl, 8%AgCl/Bi<sub>3</sub>O<sub>4</sub>Cl composite (b).

numerous nanosheets. It suggests that the tightly compressed Bi<sub>3</sub>O<sub>4</sub>Cl nanosheets are reconfigured during the hydrothermal reaction. The changed morphology may be the reason of the increased BET surface area of AgCl/Bi<sub>3</sub>O<sub>4</sub>Cl composite. Fig. 2d shows the HRTEM morphology of the AgCl/Bi<sub>3</sub>O<sub>4</sub>Cl sample. The lattice fringe of 0.2896 nm corresponds to the (411) crystal plane of Bi<sub>3</sub>O<sub>4</sub>Cl. The nanoparticles adhered on Bi<sub>3</sub>O<sub>4</sub>Cl can be assigned to AgCl since they disappear after a while of electron beam irradiation.

Fig. 3 shows the X-ray photoelectron spectra (XPS) of Bi<sub>3</sub>O<sub>4</sub>Cl, AgCl and 8%AgCl/Bi<sub>3</sub>O<sub>4</sub>Cl composite. All signals of C, Bi, Cl, O and Ag are detected in the survey XPS of the AgCl/Bi<sub>3</sub>O<sub>4</sub>Cl composite (Fig. 3a), indicating its hybrid structure, which is consistent with the XRD and TEM experiments. The Ag 3d<sub>5/2</sub> and 3d<sub>3/2</sub> binding energies (BE) of AgCl are located at 367.4 eV and 373.5 eV, respectively (Fig. 3b), which are very close to the reported values of AgCl [12]. As compared to the pure AgCl, the Ag 3d peak of AgCl/Bi<sub>3</sub>O<sub>4</sub>Cl exhibits a slightly positive shift from 367.4 eV to 367.8 eV. Given that the BE value of Ag 3d<sub>5/2</sub> of metallic Ag is 368.2 eV [13], the positive shift may originate in the formed Ag nanoparticles. This result accords well with the DRS experiment and the previous reports [9,14].

The photocatalytic activity of AgCl/Bi<sub>3</sub>O<sub>4</sub>Cl photocatalyst was evaluated by decomposing RhB under visible-light irradiation. Fig. 4a displays the changes of the RhB concentration versus the reaction time over AgCl/Bi<sub>3</sub>O<sub>4</sub>Cl photocatalysts. The blank test shows that RhB is stable under visible light irradiation, indicating that the photolysis of RhB is negligible. P25 exhibits poor activity due to its insensitivity to visible light. AgCl and pure Bi<sub>3</sub>O<sub>4</sub>Cl show high photocatalytic activities, and

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