Synthesis and Characterization of Ag-Doped BiOI Microflowers and Its Application

Nuengruethai Ekthammathat*, Pornchai Pornharuthai, Anukorn Phuruangrat

***Abstract*—**A series of silver doped bismuth oxyiodide (Ag-doped BiOI) have been successfully synthesized by microwave irradiation method. The suitable condition was adjusted synthesizes with pH 3 at 180 W for 3 h. Then, phase purity and morphologies of as-synthesized 0-3 mol% were characterized by XRD and SEM technique, respectively. The morphologies of products were microflowers in the variety size about 1.0-2.0 μm. Finally, we investigate the photocatalytic properties by degradation of rhodamine B (Rh B) under visible light irradiation for 120 min. The results show 1 mol% Ag-doped BiOI has the highest decolorization efficiency about 97.27 %. It could be concluded that Ag^+ ion has been affect to the structure and properties of BiOI.

***Keywords*—** BiOI, photocatalyst, metal-doped

I. Introduction

In recent years, renewable energy and environmental issues are highlight topic in the world. Since the discovery of photo-electrochemical splitting of the water on titanium dioxide (TiO_2) by Fujishima and Honda in 1972, the semiconductor-based photocatalyst have been researched [1]. Although TiO_2 has limited about wide band gap energy, which could only make efficient response to UV light region and it has high recombination of photogenerated electrons and holes [2]. So, there are considerate of the finding out a simple and environmentally friendly photocatalyst which works well under visible light region. Till now, there are many reported about new photocatalysts and its applications, Bi-based is the one of photocatalyst which has been attracted. For example, in 2016, K. Zhao *et al.* [3] fabricated hierarchical BiOI-chitosan nanoplate microspheres for solar cells application. Then 2017, C.K. Huang *et al.* [4] reported that $BiVO_4$ combine with silver ion (Ag^+) exhibited enhance synergic photodegradation activity toward methylene blue (MB) under visible light irradiation. There means Ag^+ ion can improve Bi-based compounds to high performance photocatalytic property.

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Although, the photocatalytic activities are depends on composition, structure, morphology and size of the materials [5]. So, the morphology control and metal-doped are the simple strategies to develop the properties of semiconductor photocatalyst.

So, in this study, we synthesized BiOI by a simple microwave irradiation method with 180 W for 3h. Ag was doped to improve the photocatalytic properties. Then, characterized as-synthesized by XRD, SEM, and TEM. Finally, we investigate the photocatalytic properties by degradation of rhodamine B (Rh B).

II. Procedure or Experiment

A. Synthesis

All the reagents of this research are analytical grade and use without further purification. A series of Ag-doped BiOI were synthesized from bismuth nitrate ($Bi(NO_3)_3{\cdot}5H_2O$) and potassium iodide (KI) as starting materials, dissolved in 150 ml deionize water. And $AgNO_3$ was added to substitute ($Bi(NO_3)_3{\cdot}5H_2O$) with series 0, 1, 2, and 3 mol%. The pH of precursor solutions was adjusted to pH 3 by 3 M NaOH. Subsequently, the solution was microwave irradiation processed at 180 W for 3 h. At last, the precipitates were filtrated, wash with DI water and ethanol, and dried at 80 °C for 24 h.

B. Characterization

The crystallinity and phases identification of the Ag-doped BiOI were characterized by the X-ray diffractometer (XRD, Philips X'Pert MPD) using a CuKα radiation at 45 kV and 35 mA in the range of 25-60 deg. The size and morphology of as-synthesized Ag-doped BiOI was studied by Low vacuum scanning electron microscope (LV-SEM, JEOL IT300) operating at 20 kV.

C. Photocatalytic activity test

Photocatalytic activities of the products were studies by decolorization of rhodamine B (RhB) in an aqueous solution under visible light irradiation. The photoreactor used light source as Philips Lifemax 32W 6200K Cool Daylight. In a typical degradation experiment, 100 ml RhB solution with a concentration 10^{-5} M and 100 mg as-prepared Ag-doped BiOI were added into a 250 ml Pyrex Erlenmeyer flask, and then stirred continuously in the dark for 30 min to ensure adsorption/desorption equilibrium of RhB on the surfaces of

the photocatalyst. The equilibrium concentration of RhB was used as the initial value for the photodegradation processes. The light was turned on to initiate the photocatalytic reaction. At certain time intervals, 5 ml solution were sampled and centrifuged to remove the particles. The degradation of organic dye was monitored by measuring the absorbance of the solution using the UV-visible spectrophotometer with DI water as reference and measure at wavelength of 570 nm. The decolorization efficiencies [6] were calculated by equation (1)

$$\text{Decolorization efficiencies (\%)} = \frac{C_0 - C_t}{C_0} \times 100 \quad \text{(eq. 1)}$$

III. Results and Discussion

This research has been synthesized and characterized the series of Ag-doped BiOI. They were synthesized by the microwave irradiation method which the simple and rapid method compared with the conventional method such as hydrothermal method. The suitable condition synthesizes was adjusted pH 3 at 180 W for 2 h. The products were red-brown powder. The phase purities and crystallinity were investigated by XRD technique. The results show 0, 1, 2, and 3 mol% Ag-doped BiOI were pure tetragonal BiOI phase with apace group P4/nmm in accordance with the JCPDS No. 10-0445 [7]. The diffraction peaks at 2Θ = 29.64, 31.65, 37.05, 37.39, 45.37, 46.48 and 51.34 can be indexed to (102), (110), (103), (112), (200), (201) and (114) crystallographic plan of BiOI phase, respectively, as shown in Fig. 1. We observed the intensities of XRD spectra were decreased after increased the content of Ag from 0 to 3 mol% but cannot detected impurity phase such as Ag, AgI. It could be conclude that Ag^+ ions were inserted in crystal structure of BiOI as well.

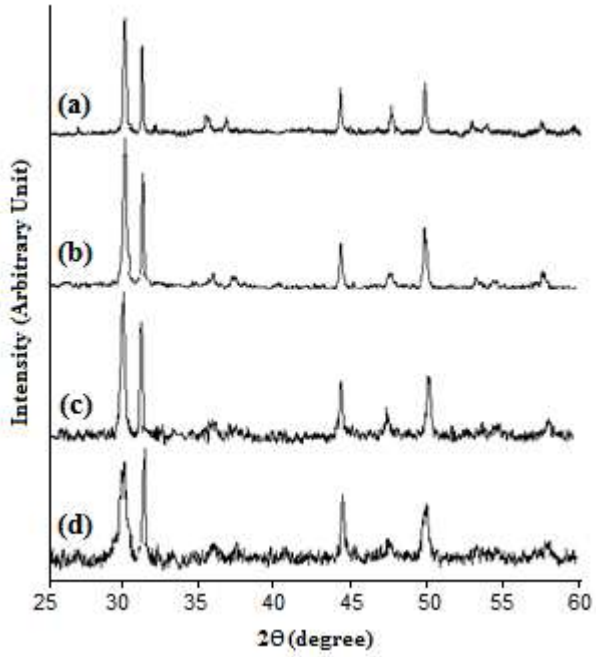

Fig. 1 XRD patterns of (a) 0, (b) 1, (c) 2, and (d) 3 mol% Ag-doped BiOI which synthesized by microwave irradiation method with 180 W 3 h.

SEM images (Fig. 2) show the morphologies of the products are microflowers in varieties size about 1.0-2.0 μm which combined from nanoplates. The contents of Ag^+ ions have a little affected to the morphology of the products such as the aggregation of nanoplates.

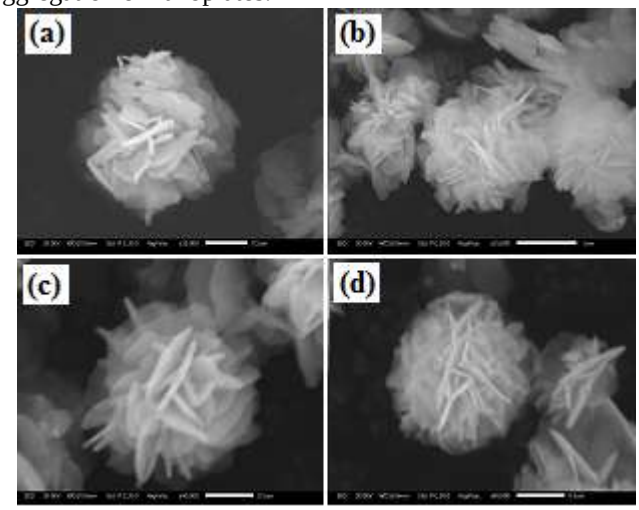

Fig. 2 SEM images of (a) 0, (b) 1, (c) 2, and (d) 3 mol% Ag-doped BiOI which synthesized by microwave irradiation method with 180 W 3 h.

The photocatalytic activities of Ag-doped BiOI were evaluated by the degradation of dye under visible light irradiation for 120 min. An organic dye molecules that we are selected to test was RhB, because it high structural stability, easy to observe by UV-visible spectrophotometer and non-photolysis [8]. It is well known that the photodegradation which occurred in this reaction is photocatalytic process.

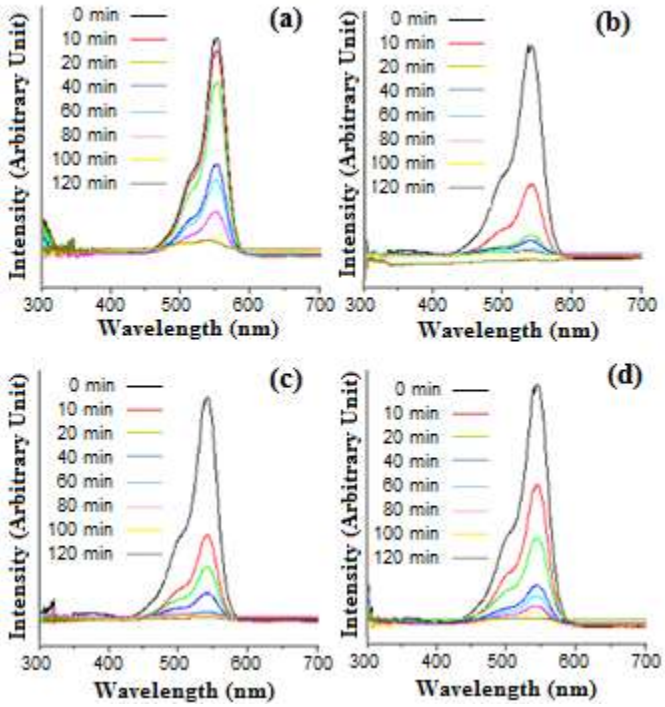

Fig. 3 Absorption spectra of RhB solution photocatalyzed by (a) 0 (b) 1 (c) 2, and (d) 3 mol% Ag-doped BiOI under visible light irradiation within 120 min.

Fig. 3 shows the absorption spectra of RhB solution photocatalyzed by (a) 0 (b) 1 (c) 2, and (d) 3 mol% Ag-doped BiOI under visible light irradiation within 120 min., optimize the characteristic peaks at 570 nm. An example color of solutions was changed from pink color to transparent by 1 mol% Ag-doped BiOI which synthesized by microwave irradiation method with 180 W 3 h, as shows in Fig 4.

Fig. 4 The decolorization of RhB by 1 mol% Ag-doped BiOI which synthesized by microwave irradiation method with 180 W 3 h

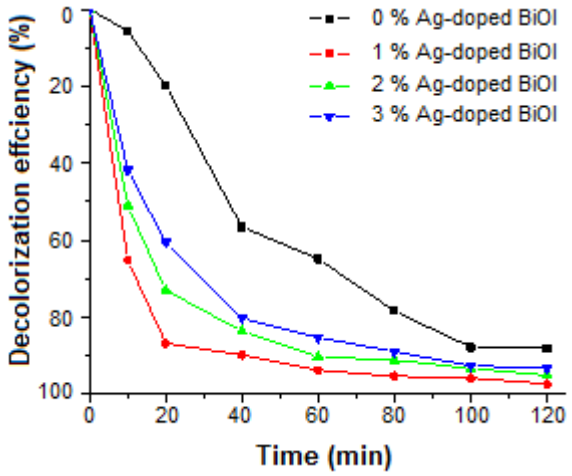

Fig.5 Decolorization efficiency of the RhB solution photocatalyzed by 0, 1, 2, and 3 % Ag-doped BiOI under visible light within 120 min.

Then, we calculated the delorlorization efficiency from eq. 1 and plot graphs show in Fig. 5. It can be seen that the decolorization efficiency (%) of 0, 1, 2, and 3 mol% Ag-doped are 88.08, 97.27, 96.80, 95.06, and 93.30 %, respectively. So, it can be concluded that 1 mol% Ag-doped BiOI have the highest decolorization efficiency and the highest rate. Ag^{+} ion has been affect to the structure and properties of BiOI. The photocatalytic activities were developed by doping the metal or metal ion due to metal ions get the Schottky barrier at semiconductor or interstitial defect [9].

IV. Conclusions

In this research, the series of 0, 1, 2, and 3 mol% Ag-doped BiOI microflowers photocatalyst have been successfully synthesized by a simple microwave irradiation method with pH 3 at 180 W for 3 h. They have an enhancement of photocatalytic performance under visible light irradiation. The highest photocatalytic properties is 1 mol% Ag-doped BiOI with 97.27%.

Acknowledgment

The authors gratefully acknowledge Bansomdejchaopraya Rajabhat University (BSRU) for financial support and Science and Applied Science Center (SASC), faculty of science and technology for facility support.

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