Bi$_2$O$_3$ nanoparticles: synthesis, characterizations, and photocatalytic activity

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Abstract. In this study, Bi$_2$O$_3$ nanoparticles have been successfully synthesized using Jatropha multifida L. leaf extract (JMLE). Bi$_2$O$_3$ nanoparticles were characterized by Fourier Transform Infrared (FT-IR) spectroscopy, X-Ray Diffraction (XRD), UV-Vis Diffuse Reflectance Spectroscopy (UV-Vis DRS), Scanning Electron Microscope-Energy Dispersive X-Ray (SEM-EDX) and Transmission Electron Microscope (TEM). These various characterizations were performed to analyze the functional groups, structural, optical, morphological, composition, and photocatalytic properties. The synthesized Bi$_2$O$_3$ have monoclinic structure, optical band gap of 3.34 eV, agglomerated morphology, and particle size of 17.26 nm. Photocatalytic activity of Bi$_2$O$_3$ nanoparticles was investigated for the degradation of methylene blue (MB) under UV light irradiation with degradation percentage of 92.53%.

Keywords: Bi$_2$O$_3$, Jatropha multifida, photocatalytic activity, methylene blue

1. Introduction

Bi$_2$O$_3$ has many advantages as anode, semiconductor, catalyst, and photodegradation of organic dyes [1–4]. Bi$_2$O$_3$ nanoparticles have been synthesized by sol gel, successive ionic layer adsorption and reaction (SILAR), redox reaction, and green synthesis methods [1, 5–7]. Bismuth oxide has less toxic, good conductivity, large energy band gap, and good photocatalyst activity [4]. Therefore, Bi$_2$O$_3$ nanoparticles are synthesized as photocatalyst to decompose some organic dyes.

Currently, the synthesis of nanoparticles is mostly by green synthesis. It aims to reduce the use of hazardous chemicals, which pollute the environment. Green synthesis methods are environmentally friendly, low cost, and the used material is easy to obtain. Metal oxide nanoparticles that has been synthesized with green synthesis method are ZnO/CuO, Fe$_3$O$_4$, CdO, and La$_2$O$_3$ [8–11]. One of the raw materials is plant leaf extract.

Jatropha multifida is a kind of traditional plants in Indonesia. It is used as antiseptic, antioxidant, antibacterial, antimicrobial, and others [12–17]. Jatropha multifida also contains secondary metabolites such as alkaloid, flavonoid, tannin, steroid, phenol, and saponin [16, 18].

Synthesis of Bi$_2$O$_3$ nanoparticles with aqueous fraction of plant extracts has not been studied. In this study, we reported the synthesis of Bi$_2$O$_3$ nanoparticles with Jatropha multifida leaf extract (JMLE) and their photocatalytic activity under UV light irradiation. Photocatalytic activity of Bi$_2$O$_3$ nanoparticles was performed for methylene blue (MB) degradation.

2. Materials and methods

2.1. Materials

Bismuth nitrate alkaline Bi$_2$O(OH)(NO)$_3$ (Merck, Germany) was used as precursor. Jatropha multifida leaves were collected from Conservation Unit of Biopharmaceutical Cultivation (UKBB) Study Center.
of Tropical Biopharmaca, Bogor Agriculture Institute, Bogor, Indonesia. All reagents and solvents, which used in the investigation, were in analytical grade.

2.2. Preparation of *Jatropha multifida* leaf extract

*Jatropha multifida* leaf was washed, dried and mashed. 50 mg of leaf powder was macerated in 250 mL of methanol by stirring every day for 7 days. The methanol fraction was partitioned with hexane, and it was evaporated. The extract was added with deionized water as *Jatropha multifida* leaf extract (JMLE) stock solution.

2.3. Phytochemical screening

Each fraction of leaf extract was phytochemically tested to determine the secondary metabolites content such as alkaloids, flavonoids, steroids, terpenoids, tannins, polyphenols, and saponins.

2.4. Synthesis of Bi$_x$O$_y$

0.1 mmol of Bi$_2$O$_3$(NO$_3$)$_3$ was added into JMLE solution, then stirred at 80 °C for 2 h. The results were centrifuged for 30 min. The formed particles were rinsed with deionized water and recentrifuged for 15 min. The centrifuged result was dried and calcined at 500 °C for 3 h to obtain Bi$_x$O$_y$ powder.

2.5. Characterization of Bi$_x$O$_y$

The Fourier Transform Infrared (FT-IR) spectrophotometer (IR Prestige-21 Shimadzu) was performed to determine functional groups in JMLE and Bi$_x$O$_y$ nanoparticles. UV-Vis Diffuse Reflectance Spectroscopy (DRS) Shimadzu 2400 was used to investigate the optical studies at the wavelength range of 200–800 nm. Scanning Electron Microscopy (SEM) Zeiss Evo MA10 and Tunneling Electron Microscopy (TEM) JEOL JEM 1400 were used to know the surface morphology and the particle size of nanoparticles. Energy Dispersive X-Ray (EDX) was used to determine the composition of nanoparticles. X-Ray Diffraction (XRD) characterization (PANalytical EMPYREAN) was used to determine the crystal structure at 20 range of 10°–90°. The photodegradation of methylene blue was investigated using UV-Vis spectrophotometer (Shimadzu 2600) at the wavelength of 200–800 nm.

2.6. Photocatalytic activity of Bi$_x$O$_y$

The photocatalytic activity test of Bi$_x$O$_y$ nanoparticle was carried out for the degradation of methylene blue under UV light irradiation (Gold star PLB 9 W-BLB, 170-240 V). All experiments were performed at room temperature for 150 min.

3. Results and discussion

3.1. Phytochemical screening

The phytochemical screening was performed to determine the content of secondary metabolites such as alkaloids, flavonoids, tannins, polyphenols, steroids, terpenoids, and saponins. Methanol fraction and aqueous fraction of JMLE contain alkaloids, flavonoids, tannins, polyphenols, and saponins. While, hexane fraction contains alkaloid and saponin. Hence, phytochemical screening of JMLE aqueous fraction show the positive results for alkaloids as the main secondary metabolite in Bi$_x$O$_y$ nanoparticle synthesis. This result is demonstrated in further analysis by FT-IR spectroscopy.

3.2. FT-IR analysis

FT-IR spectroscopy was performed to determine the functional group of JMLE and Bi$_x$O$_y$ nanoparticles at the wavenumber 400–4000 cm$^{-1}$. Figure 1 shows FT-IR spectra of Bi$_x$O$_y$ nanoparticles and JMLE. The sharp absorption peaks at 1383 cm$^{-1}$ is assigned to the C–O vibration and 3420 cm$^{-1}$ as –OH stretching from peak shift at JMLE that may still be left on the surface of Bi$_x$O$_y$ nanoparticles. The vibration of Bi–O is shown at wavenumber 825, 586, and 495 cm$^{-1}$. It has the conformity with C–O vibration modes at 1388 cm$^{-1}$ [4]. The wavenumber of 400–650 cm$^{-1}$ and 3433 cm$^{-1}$ also correspond to BiO, octahedron and –OH vibration, respectively as reported by Yuvakkumar and Hong [20]. The absorption peaks of JMLE assign to the stretching vibration modes of O–H at 3320 cm$^{-1}$ and the stretching vibration of C–H at 2930 cm$^{-1}$. These two vibrations demonstrate the presence of saponin as reported by Venkatesan et al. [21]. C=O group vibration at 1610 cm$^{-1}$ and C–N stretching vibration at 1076 cm$^{-1}$ show the presence of saponin and alkaloid, respectively. The vibration mode of C–C stretching is assigned at 1406 cm$^{-1}$. These results are in line with the previous studies that the vibration
3.3. XRD analysis
XRD pattern of Bi$_2$O$_3$ nanoparticles gives a strong and sharp peak, indicating that Bi$_2$O$_3$ nanoparticles are crystals phase as shown in figure 2. The peaks are related to the monoclinic crystals phase of Bi$_2$O$_3$ suitable with COD No. 96-152-6459 at the 2θ value of 24.55°, 25.75°, 26.92°, 27.34°, 33.18°, 35.00°, 35.42°, and 37.56°. The average crystallite size of Bi$_2$O$_3$ nanoparticles can be calculated by Debye Scherrer equation and obtained the average crystal size of 69.24 nm.

3.4. SEM-EDX and TEM analysis
Figure 3a shows the SEM images of Bi$_2$O$_3$ nanoparticles with the magnification of 25,000 times, indicating the morphology of Bi$_2$O$_3$ nanoparticles are agglomerated. Figure 3b shows TEM images with the scale bar of 50 nm with the morphology of Bi$_2$O$_3$ nanoparticles are cubic like and agglomerated. The particle size of Bi$_2$O$_3$ is 17.26 nm. According to the EDX spectrum as shown in figure 3c, the quantitative EDX analysis indicates the atomic ratios of Bi and O are 2:3, which is confirmed as Bi$_2$O$_3$.

3.5. UV-Vis DRS analysis
UV-Vis DRS was performed to investigate the optical studies and to determine the band gap energy of semiconductor materials. The reflectance value is converted by the Kubelka Munk equation. The band gap energy can be determined by plot Tauc method. Figure 4 shows Bi$_2$O$_3$ nanoparticles with the band
The band gap energy \( E_g \) of Bi\(_2\)O\(_3\) nanoparticles.

**Figure 4.** The band gap energy \( E_g \) of Bi\(_2\)O\(_3\) nanoparticles.

Based on the band gap energy value, Bi\(_2\)O\(_3\) nanoparticles can work as photocatalyst under UV light irradiation [4].

**3.6. Photocatalytic activity of Bi\(_2\)O\(_3\)**

The photocatalytic activity of Bi\(_2\)O\(_3\) nanoparticles was observed for the degradation of methylene blue under UV light irradiation. The UV-Vis absorption peak decreases at \( \lambda_{\text{max}} \) of 664 nm for 150 min as shown in figure 5. The degradation percentage of methylene blue was obtained at 92.53% using Bi\(_2\)O\(_3\) nanoparticles under UV light irradiation.

The UV light irradiation activates Bi\(_2\)O\(_3\) nanoparticle to form electron-hole pairs. The catalyst, which is exposed by UV light irradiation will produce hole in the valence band and electron in the conduction band. It causes the excitation of electrons from the valence band to the conduction band. The hole which reacts with H\(_2\)O will form •OH, while the reaction with O\(_2\) will produce superoxide radicals (O\(_2\)•) [26]. The reaction will degrade methylene blue to the smaller molecules.

**4. Conclusions**

Bi\(_2\)O\(_3\) nanoparticles have been successfully synthesized by green synthesis method using *Jatropha multifida* leaf extract (JMLE) as a weak base source and capping agent. The phytochemical screening shows that JMLE contains alkaloid, flavonoid, tannin, polyphenol, and saponin. FT-IR characterization shows the vibration of Bi-O at the wavenumber 825, 586, and 495 cm\(^{-1}\). The band gap energy of Bi\(_2\)O\(_3\) nanoparticles is 3.34 eV and its morphology is agglomerated with the particle size of 17.26 nm. The photocatalytic activity test of Bi\(_2\)O\(_3\) nanoparticles has been conducted for degradation of methylene blue at 92.53% for 150 min.

**Acknowledgements**

The authors would like to thank University of Indonesia for funding this research through PITTA Grant University of Indonesia with contract No. 2339/UN2.R3.1/HKP.05.00/2018.

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