STUDYING IMPACT OF CITRIC ACID-BISMUTH NITRATE PENTAHYDRATE RATIO ON PHOTOCATALYTIC ACTIVITY OF BISMUTH OXIDE PREPARED BY SOLUTION COMBUSTION METHOD

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ABSTRACT

Bismuth oxide can be synthesized by solution combustion method. One of parameters that must be considered on the synthesis using this method is fuel-oxidant ratio (ϕ) since it influences the physicochemical properties such as morphology, the particle size and crystal structure of the obtained products which further affect on their photocatalytic activity. This research used citric acid as fuel and $\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$ as oxidant. This study aims to investigate impact of citric acid-bismuth nitrate pentahydrate (fuel-oxidant) ratio on the physicochemical properties and photocatalytic activity of the obtained products on degradation of methyl orange. The results showed that all the obtained products contained the mixture of $\alpha\text{-Bi}_2\text{O}_3$ (monoclinic) and $\beta\text{-Bi}_2\text{O}_3$ (tetragonal) with irregular shape coral reef-like and in agglomerate form. In addition, the fuel-oxidant ratio does influence the products' particle size in which the particle size of the products prepared by a ratio of $\phi > 1$, $\phi = 1$, and $\phi < 1$ are about 5-8 μm , 6-10 μm , and 9-14 μm , consecutively. Result of photocatalytic activity shows product synthesized with a ratio of $\phi > 1$ has the highest photocatalytic activity.

Keywords: Bismuth Oxide, Solution Combustion, Fuel-oxidant, Photocatalyst, Photocatalysis

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INTRODUCTION

Bismuth oxide (Bi_2O_3) is a metal oxide with a pale yellow solid¹ and melting point of 825 °C.² Bismuth oxide (Bi_2O_3) has good optical and electrical characteristics such as refractive index, high dielectric permittivity and electrical conductivity.³ Furthermore, bismuth oxide has wide energy band gap (2 - 3.96 eV).⁴ Therefore, due to its potential properties, bismuth oxide is used in various applications for examples: solid oxide fuel cell,⁵ electrode,⁶ gas sensor,⁷ and degradation of dye molecules through photocatalysis.⁸⁻¹³

This metal oxide can be prepared by several methods, such as precipitation,^{8,9,14,15} solution combustion,^{10,16} hydrothermal,¹⁷ and sol gel methods.¹⁸ In this study, bismuth oxide was prepared by solution combustion method because it has several advantages such as fast and easy processing, shorter reaction times, high purity and homogeneity of the product, and nanosized powder product.¹⁹ Solution combustion method is a synthesis method whose main components consist of oxidant and fuel, and the process involves high temperature.¹⁹

Factors that can influence the synthesis using the solution combustion method include the ratio of fuel-oxidant, temperature, flame type and the amount of gas produced. In this study, bismuth oxide was prepared by solution combustion method with variation of fuel-oxidant ratio (ϕ). The fuel-oxidant (ϕ) ratio can affect the morphology, crystal structure, and particle size of the resulting products. The fuel-

oxidant (ϕ) ratio determines the gas amount formed. Gas formed during the reaction disintegrates the cluster and forms pores in the material. The greater the amount of the fuel, the more gas formed; consequently, the greater pores formed as well. Moreover, the resulting particle size is also smaller since the gas formed inhibits the growth of the particles.¹⁹

La et al.¹⁶ synthesized bismuth oxide using solution combustion method with variation of fuel-oxidant ratio 1.2, 0.8 and 0.6. The fuel used was citric acid, while the oxidant used was bismuth nitrate pentahydrate. The resulting product was β -Bi₂O₃ (tetragonal) at the ratio of fuel-oxidant 1.2 and 0.8, while the ratio of fuel-oxidant 0.6 resulted in a mixture of α -Bi₂O₃ (monoclinic) and β -Bi₂O₃ (tetragonal). In term of particle size, product synthesized using a ratio of fuel-oxidant 1.2 had a material size of 20-30 nm, while for the fuel-oxidant ratio of 0.8 generated large agglomerated product. Eventhough La et al.¹⁶ synthesized bismuth oxide using solution method with variation of fuel-oxidant ratios, the impact of difference ratio of fuel-oxidant on the photocatalytic activity has not been explored yet. Therefore, in this study, bismuth oxide fabrication was carried out by solution combustion method with a variety of fuel-oxidant (ϕ) ratios, namely $\phi > 1$, $\phi = 1$, and $\phi < 1$ in order to determine the impact of the ratio of fuel-oxidant on the physicochemical properties and photocatalytic performance of the obtained products. The oxidant and fuel used were bismuth nitrate pentahydrate and citric acid, respectively.

EXPERIMENTAL

Materials

Materials used were bismuth nitrate pentahydrate (Bi(NO₃)₃·5H₂O) from Sigma-aldrich, HNO₃ (65%, v/v), NH₄OH (25%, v/v), polyethylene glycol (PEG 20000), methyl orange, citric acid monohydrate (C₆H₈O₇·H₂O) and distilled water purchased from Merck Index, Indonesia without further purification and in analytical grades.

General procedure

Synthesis of bismuth oxide by solution combustion method follows the method recommended by La et al.¹⁶ with slight modification. Fuel-oxidant (ϕ) ratios used in this study were $\phi > 1$ with a value of 1.2; $\phi = 1$, and $\phi < 1$ with a value of 0.8.

Synthesis of bismuth oxide with fuel-oxidant ratio $\phi > 1$ started with adding of 2.9106 gram Bi(NO₃)₃·5H₂O in 10 mL of HNO₃ 0.04 M. The mixture was stirred until homogeneous with speed of 1000 rpm. Next, 0.04 gram of PEG and 1.471 gram of citric acid was added into the mixture and stirred in 1200 rpm for 5 minutes until the solution was in transparent form. The clear transparent solution formed was then heated at 300 °C for 8 hours. The formed product was calcined at 700 °C for 4 hours. As a result, a yellow powder was formed and used for further characterization and photocatalytic activity test. This procedure was also performed on the synthesis of bismuth oxide with variations in the fuel-oxidant ratio $\phi = 1$ and $\phi < 1$ by changing the amount of Bi(NO₃)₃·5H₂O which was added.

Characterisation Method

The characterization techniques used for products analysis were XRD, FTIR, TGA and SEM. The XRD instrument used CuK α as the radiation source, 30 kV voltage, 30 mA electricity, the length of radiation wave X-ray 1.54178 Å and 2 θ ranging from 10° to 90°. Diffractograms from XRD analysis were compared with joint comitte on powder diffraction standard (JCPDS) database to identify the crystal structure of products. FTIR Prestige 21 (Shimadzu) with wavenumber scanning of 400-4000 cm⁻¹ was applied to obtain FTIR spectra. A thermogravimetric curves were recorded from TGA 4000 Perkin Elmer at the temperature range of 50 – 700 °C with heating rate of 5 °C/min under nitrogen stream. SEM images were obtained using JEOL-JSM-6510LV microscope.

Photocatalytic Activity Test

Photocatalytic performance test of the obtained products was conducted in a reactor photocatalyst, using methyl orange 5 ppm. It was undertaken without and with light irradiation. For the photocatalytic activity test without light irradiation, 50 mL of methyl orange was added 0.1 gram of the obtained product. Subsequently, the mixture was stirred in medium speed (667 rpm) for 2 hours. While for photocatalytic

activity test under ultraviolet (UV) light the procedure was the same however the samples were under UV irradiation in different times that were 2, 4, 6, 8, and 10 hours. After that, the mixture was filtered and filtrate was analyzed using UV-Vis spectrophotometer to determine the absorbance which further to calculate degradation percentage of methyl orange by bismuth oxide.

RESULTS AND DISCUSSION

Synthesis of Bismuth Oxide Using Combustion Method

Bismuth oxide synthesis started with dissolving bismuth nitrate pentahydrate ($\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$) in nitric acid solvent and formed homogeneous suspension with cloudy white in colour. The suspension was then added polyethylene glycol (PEG) and citric acid then stirred at high speed (1200 rpm) to form transparent solution. According to Li et al.,²⁰ the role of PEG is to prevent agglomeration; PEG is to slow the rate of diffusion of solutes from the nucleus formed to the surface of the crystal, while the diffusion process is a rate determinant during crystal growth. By the slow crystal growth, the product generated would have a more homogeneous morphology and smaller particle size.

The transparent solution formed had been heated at 300 °C for 8 hours. This process produced gases of CO_2 , N_2 , and H_2O .²¹ The transparent solution heated for 8 hours resulted in powder with brownish yellow mixed with black in colour as seen in Fig.-1. The black colour in the samples indicated the presence of carbon derived from citric acid. During the heating process there was also the formation of purplish pink smoldering flame. Smoldering type flame cannot be long-lasting, sometimes it doesn't even appear. This flame is formed from the release of heat from the combustion process.¹⁹

Powder with yellow in colour was formed when calcination at 700 °C for 4 hours was applied as presented in Fig.-2. This yellow colour powder indicated physical appearance that bismuth oxide was formed. Percentage yields of bismuth oxide with a ratio of $\phi > 1$, $\phi = 1$, and $\phi < 1$ were 66.43%, 74.26% and 65.71%, consecutively. According to Mukasyan et al.,²¹ the mechanism occurred during the calcination process is :

Fig.-1: The Physical Appearance Of The Transparent Solution of (a) $\phi > 1$, (b) $\phi = 1$, (c) $\phi < 1$ After Heating For 8 Hours

Fig.-2: The Physical Appearance Of The Products With ratio of (a) $\phi > 1$; (b) $\phi = 1$; (c) $\phi < 1$ after Calcination

Fig.-3: Diffractograms of Products Synthesized With Fuel-Oxidant Ratio of (a) $\phi > 1$, (b) $\phi = 1$, (c) $\phi < 1$

In order to identify that the bismuth oxide synthesis was successful, the products were then analyzed using XRD to determine the product's crystal structures. Figure-3 presents the diffractograms of products synthesized with a variety of fuel-oxidant ratios.

The presence of α - Bi_2O_3 is indicated by a peak at 2θ of 27.10, 32.92, and 46.05 for product with ratio of $\phi > 1$; at 2θ of 27.13, 32.90, and 46.10 for product with ratio of $\phi = 1$, and at 2θ of 27.16, 32.93, and 46.07 for product with ratio of $\phi < 1$. Those data are consistent with the JCPDS database number 41-1449 for α - Bi_2O_3 . The presence of β - Bi_2O_3 is marked with a peak condition at 2θ of 25.49, 42.11, and 54.51 for product with a ratio of $\phi > 1$; at 2θ of 25.50, 42.15, and 54.57 for product with ratio of $\phi = 1$, and at 2θ of 25.56, 42.21, and 54.59 for product with ratio of $\phi < 1$. This is consistent with the JCPDS database with number 27-0050 for β - Bi_2O_3 . The crystal structures of the products synthesized using solution combustion method with a variation ratio of $\phi > 1$, $\phi = 1$ and $\phi < 1$ are a mixture of α - Bi_2O_3 (monoclinic) and β - Bi_2O_3 (tetragonal). This result is in agreement with La et al¹⁶ report. This suggests that the variation of the ratio of fuel-oxidant (ϕ) has no effect on the crystal structure of the obtained products.

The FTIR spectroscopy technique was also applied to identify the functional groups indicating the presence of bismuth oxide. Figure-4 shows the FT-IR spectra of all the products synthesized in different fuel-oxidant ratio. The intense band at 2250-2100 cm^{-1} region is attributed to the vibration mode of the alkyne.²² The presence of vibration mode C-C attributed to alkyne is probably because of incomplete reaction of citric acid as fuel yet the intensity of this vibration mode is low. Furthermore, the peak at 844 cm^{-1} is due to Bi-O stretching vibration.²³ The intense band at 1200 - 1700 cm^{-1} possibly arises due to stretching vibrations of the NO_3 ion, which may come from bismuth nitrate $\text{Bi}(\text{NO}_3)_3$.²⁴ All the products show no significant difference in FTIR spectra.

Changes of the samples before calcination as seen in Fig.-1 that occurred during the calcination process can be identified using TGA analysis by calculating mass changes as a result of the gradual increase in temperature. One of the samples before and after calcination as shown in Fig.-1 and Fig.-2 respectively was analyzed using TGA. Figure-5 shows the TGA curves of the sample before (Fig.-5a) and after calcination (Fig.-5b). TGA curve of the sample before calcination shows that there are three regions of mass weight loss on the TG curve. The first area at the temperature of 34.0-50.8°C is associated with

the decomposition of bismuth nitrate pentahydrate ($\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$).²⁵ The second area occurred at the temperature of 265-319.8 °C which may be due to reduced or lost organic matter such as citric acid. The total loss of organic matter is in the region of ~ 398 °C. The relative mass changes of the sample before calcination were 1.64292 (%), 3.36388 (%), and 2.14636 (%). While the TGA curve of sample after calcination shows no relative mass change significantly which is probably the formation of bismuth oxide was almost complete. Compared to TGA curve of sample before calcination, the relative mass change of sample after calcination is smaller than that before calcination.

Fig.-4: FT-IR Spectra Of Products Synthesized By Citric Acid-Bismuth Oxide Ratio of (a) $\phi=1$; (b) $\phi<1$; (c) $\phi>1$

The morphology and particle size of the products were then analyzed using SEM. Figure-6 shows the SEM images of the products. It can be seen that all samples have similar morphology, that is irregular shapes, coral reefs-like and in agglomerate forms, with the size of 5-8 μm , 6-10 μm , and 9-14 μm for products synthesized with $\phi>1$, $\phi=1$, and $\phi<1$ consecutively. The bigger the fuel-oxidant ratio (ϕ), the smaller the particle size. The fuel-oxidant (ϕ) ratio determines the amount of gas formed. The gas formed during the reaction disintegrates the cluster and form pore in the material. The greater the amount of the fuel, the more gas formed; consequently, the resulting particle size is smaller since the gas formed inhibits the growth of the particles.¹⁹

Photocatalytic Activity

The products were applied for methyl orange degradation under ultraviolet light by time interval of 2 – 10 hours. The absorbance of methyl orange solution after photocatalysis was measured using UV-Vis spectrophotometry with a maximum wavelength of 465 nm.

Fig.-5: Thermogram Bismuth Oxide (A) Before Calcination; (B) After Calcination

Fig.-6: SEM Images of Products Synthesized With Citric Acid-Bismuth Nitrate Pentahydrate of (A) $\Phi > 1$, (C) $\Phi = 1$, (E) $\Phi < 1$ With The Magnification of 200x; And of (B) $\Phi > 1$, (F) $\Phi < 1$ (D) $\Phi = 1$ With Magnification of 1000x.

Figure-7 shows the percentage of degradation of methyl orange after photocatalysis using the three samples. It shows that during the 10 hours of the photocatalysis, products synthesized with a ratio of $\phi > 1$, $\phi = 1$, and $\phi < 1$ degraded methyl orange for 38.53%, 13.34% and 38.02% consecutively. Products with a ratio of $\phi > 1$ have the best photocatalytic activity for methyl orange degradation because the high fuel-oxidant ratio (ϕ) generated the smaller particle size of product.¹⁹ The smaller particle size the greater the surface area; consequently contact between methyl orange and particles is also high. As a result, photocatalytic activity increases. This is in agreement with SEM image as presented in Fig.-6a and 6b that product synthesized with fuel-oxidant ratio of $\phi > 1$ has the smaller particles compared to other products.

The mechanism of methyl orange degradation has been proposed by Liu et al.²⁶ When the bismuth oxide is illuminated by light, the electrons will be excited from the valence band to the conduction band, and the hole electrons will be formed in the valence band. Then OH^- ions that come from water adsorbed on the surface of bismuth oxide react with holes of electrons and form $\bullet\text{OH}$ radicals and H^+ ions.²⁶ On the semiconductor surface, electrons in the conduction band react with atmospheric O_2 to form $\bullet\text{O}_2^-$. In addition, $\bullet\text{O}_2^-$ can be another source of $\bullet\text{OH}$ formation after protonation process. Radical of hydroxyl ($\bullet\text{OH}$) is a potential substance for oxidation of methyl orange into simpler molecules that are CO_2 and H_2O when the reaction is complete.²⁶

Wang et al.^[27] reported that the activity of dye molecules degradation generally follows the first-order kinetics, which is formulated by the equation:

$$\ln C_t = \ln C_0 - kt$$

with k is degradation rate constant in the first-order kinetics model (s^{-1}); C_0 and C_t is methyl orange concentration at $t = 0$ and t time (ppm), respectively.

Figure 8 shows the first order kinetics model of the degradation of methyl orange. The degradation rate constant of methyl orange by-products synthesized with citric acid-bismuth nitrate pentahydrate ratio of

$\phi > 1$, $\phi = 1$ and $\phi < 1$ are 97.20 s^{-1} , 47.52 s^{-1} , and 95.76 s^{-1} , respectively. Product with a ratio of $\phi > 1$ has the highest reaction rate constant degradation of methyl orange, it is in accordance with the highest photocatalytic performance of the methyl orange degradation as seen in Fig.-7.

Fig.-7: The Degradation Percentage of Methyl Orange By All The Products

Fig.- 8: First Orde Reaction Kinetic on The Methyl Orangedegradation

CONCLUSION

The citric acid-bismuth nitrate pentahydrate ratio affected the photocatalytic activity of the bismuth oxide prepared by solution combustion method. The photocatalytic performance of obtained products with a ratio of $\phi > 1$ showed the highest activity on the degradation of methyl orange under light irradiation. Meanwhile, the crystal structures of the products synthesized with a variation ratio of $\phi > 1$, $\phi = 1$ and $\phi < 1$ are a mixture of $\alpha\text{-Bi}_2\text{O}_3$ (monoclinic) and $\beta\text{-Bi}_2\text{O}_3$ (tetragonal) suggesting that the variation of citric acid-bismuth nitrate pentahydrate (fuel-oxidant) ratio (ϕ) has no effect on the crystal structure of the products. However, this fuel-oxidant ratio does influence the particle size of the products.

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