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Effect of Calcination Temperature on The Morphology and Photocatalytic Activity of Bismuth Oxide

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Abstract. Bismuth oxide can be synthesized by sol-gel (SG) method with variety of calcination temperature. Bismuth oxide (Bi_2O_3) is a pale-yellow solid which has 2 to 3.96 eV band gap, high photocatalytic activity, and electrical conductivity so that this material has the potential properties as the basic material of solid electrolyte fuel cells and photocatalyst. In this research, synthesis of bismuth oxide was undertaken using sol-gel method with variety of calcination temperatures, i.e. 500 °C, 600 °C, dan 700 °C. Calcination temperature is one factor that affects the synthesis product, so this research aims to determine the effect of calcination temperature to the resulting synthesis products. The synthesis of bismuth oxide by this method was conducted by dissolving Bi(NO3)3.5H2O and citric acid in HNO3, then added PEG 6000. The results showed that bismuth oxide synthesized using sol gel method with variety of calcination temperature have different crystal structure. While, the photocatalytic activity indicated that bismuth oxide synthesized with calcination temperature at 700 0C gave best performance on degradation of methyl orange.

INTRODUCTION

Bismuth oxide (Bi_2O_3) is a metal oxide with a pale yellow solid [1], which has melting point on 825 °C [2]. Bismuth oxide (Bi_2O_3) has good optical and electrical characteristic such as refractive index, high dielectric permittivity and electrical conductivity. Furthermore, bismuth oxide has an energy band gap between 2 to 3.96 eV [3]. Therefore, bismuth oxide can also be used in various other applications such as solid oxide fuel cell, gas sensor, and photocatalytic[4]. Bismuth oxide can be synthesized by several methods, for example precipitation method [5-7], solution combustion method [1, 8, 9], hydrotermal method [10], and sol-gel method [11, 12].

In this study, bismuth oxide is synthesized using sol-gel method because this method has many advantages, such as homogeneity and good purity, low synthesis temperature because in certain materials can be done in room temperature[13], the size of the crystal is relatively small around 100 nm because the size distribution can be controlled [14]. Sol-gel method influenced by several factors, such as calcination temperature. Calcination temperature is one factor that affects the synthesis product, wherein the high calcination temperature and duration of calcination have a significant effect on the nature of crystallinity, structure and surface of the product[14].

In this research, synthesis of bismuth oxide was undertaken using sol-gel method with variety of calcination temperatures, i.e. 500 °C, 600 °C, dan 700 °C. This research aims to determine the effect of calcination temperature to the resulting synthesis products. The resulting product was then characterised using XRD to identify the crystal structure, SEM is to identify the morphology of the material and also tested as photocatalyst for methyl orange degradation under ultraviolet light.

EXPERIMENTAL METHODS

The materials used in this research: Bismuth nitrate pentahydrat ($Bi(NO_3)_3.5H_2O$) from Sigma-aldrich Other reagents used were nitric acid (65%, v/v), polyethyleneglycol (PEG 6000), citric acid monohydrate ($C_6H_8O_7.H_2O$), methyl Orange bought from Merck Index, Indonesia and Aquadest.

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Synthesis of bismuth oxide using sol-gel method was undertaken using the procedure reported by Astuti et al [12] with slight modification. 4 grams of $Bi(NO_3)_3.5H_2O$ and citric acid with ratio molar of 1:1 were dissolved in 50 ml HNO_3. The mixture is stirred until homogeneous with 1000 rpm. The mixture added 2 ml PEG 6000 then heated at 100°C until yellow gel formed (stirring 40 hours with 667 rpm), then yellow gel was dried in the oven at 100 °C for 12 hours until swollen. The formed product will be calcined at 500 °C, 600 °C, dan 700 °C for 4 hours. A yellow powder was obtained after calcination and used for further characterization using XRD (XRD Bruker using CuKα as the radiation source with 2 θ ranging from 10° to 90° and the lengh of radiation wave X-ray 1,54056 Å), SEM (JEOL-JSM-6510LV). The diffractograms XRD is fitted using origin software, the fitting result will compared with Joint Comitte on Powder Diffraction Standars (JCPDS) database. The formed product also tested for degradation of methyl orange.

Photocatalytic activity test of the synthesized Bi_2O_3 is conducted in a reactor photocatalyst, using methyl orange 5 ppm. For the photocatalytic activity test without light, 0,1 gram of bismuth oxide is added to 50 mL methyl orange. Then, stir the sample in medium speed (667 rpm) for 2 hours. While for photocatalytic activity test with ultraviolet light (UV-A (352 nm) at 15 watt is conducted for 2 hours, 4 hours, 6 hours, 8 hours, and 10 hours.

RESULT AD DISCUSSION

Synthesis of Bismuth Oxide using Sol-Gel Method

The result of synthesis bismuth oxide using sol gel method can be seen in Fig 1. After stirring for 40 hours and heated at 100°C, yellow gel formed. The swelling brownish yellow sol is formed after yellow gel drying in the oven for 12 hours. Then the formed product will be calcined at 500 °C, 600 °C, dan 700 °C for 4 hours. Then the formed product will be calcined at 500 °C, 600 °C, dan 700 °C for 4 hours.

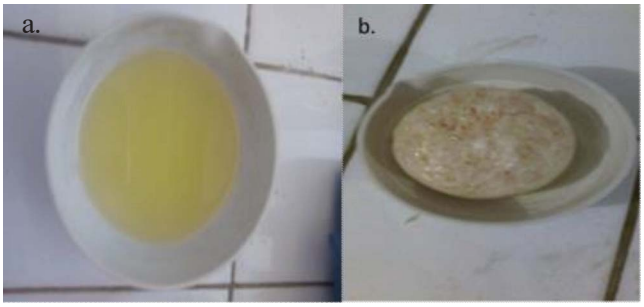

FIGURE 1. The result of bismuth oxide synthesis: (a) After stirring for 40 hours, (b) After drying in the oven at 100 °C

The color of the powder turned to be yellow after calcination for 4 hours. The result are shown in Fig. 2. The result showed that the higher the temperature of calcination, the crystals produced the more yellow. The product has different colors because bismuth oxide produced has different structure.

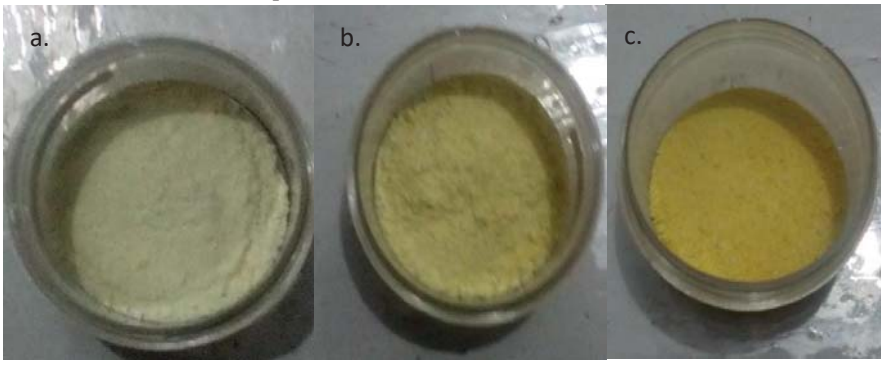

FIGURE 2. The result of bismuth oxide synthesis after calcination at: (a) 500°C (b) 600°C (c) 700°C

X-Ray Diffraction (XRD)

The XRD characterization results were obtained by comparing some of the highest peak samples with the peak of the JCPDS database with numbers 41-1449 for α-Bi_2O_3, 45-1344 for γ-Bi_2O_3, and 27-0054 for BiO. The diffractograms of bismuth oxide synthesized with a variety of calcination temperature can be seen in Fig.3. It can be seen that the calcination temperature at 500 °C resulted in a mixture of a α-Bi_2O_3 (monoklinik), γ-Bi_2O_3 (*body center cubic*), dan BiO. Bismuth oxide synthesized with calcination temperature at 600 °C resulted in a mixture of α-Bi_2O_3 (monoclinic), γ-Bi_2O_3 (*body center cubic*), dan BiO. Then bismuth oxide synthesized with calcination temperature at 700 °C resulted in α-Bi_2O_3 (monoclinic). The single phase observed might be due to the high temperature applied. As a result, the gel formed was converted completely into α-Bi_2O_3 (monoclinic).

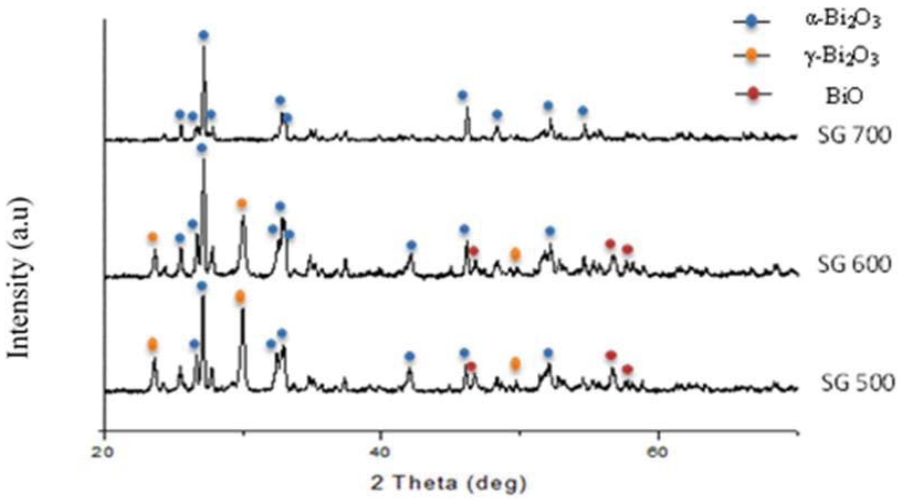

FIGURE 3. Diffractogram XRD bismuth oxide synthesized with variety of calcination temperature

Scanning Electron Microscope (SEM)

SEM images presented in Fig. 4 show that bismuth oxide synthesized using variety of calcination temperature has relatively homogeneous surface with different shape and size.

The surface of the calcinated bismuth oxide at 500 °C has a shape like multi-layered cauliflower and agglomerate. The size of the material is 0.5-2 μm. The surface of the calcinated bismuth oxide at 600 °C has a shape like cauliflower composed of thin, multi-layered plates, and agglomerates. The size of the material is 1-2.25 μm. Then the surface of the calcinated bismuth oxide at 700 °C has a shape like coral reefs, porous and agglomerates. The size of material is 2.5-10.2 μm. According to Cabot et al. (2004) [15] at a temperature of 700 °C is a transformation to form a crystal structure α-Bi_2O_3, the growth of crystals resembles coral reefs.

Photocatalytic Activity of Bismuth Oxide

Bismuth oxide is tested as a photocatalyst for the degradation of methyl orange under ultraviolet light by time inteval of 2-10 hours. The result of photocatalytic obtained from the absorbance of methyl orange of photocatalytic results is measured using UV-Vis spectrophotometry with maximum wavelength at 465 nm. From Fig.5. we concluded that the longer of the photocatalytic process, concentration of methyl orange will be smaller because methyl orange is degraded. The photocatalytic at 10 hours showed the best result that is able to reduce the concentration of methyl orange which was initially 4,965 ppm to concentration 3,184 ppm for SG 500, 3,325 ppm for SG 600, and 2,667 ppm for SG 700 and with methyl orange which degraded SG 500, SG 600, and SG 700 respectively as much as 35.860%, 34.842%, and 46.287%.

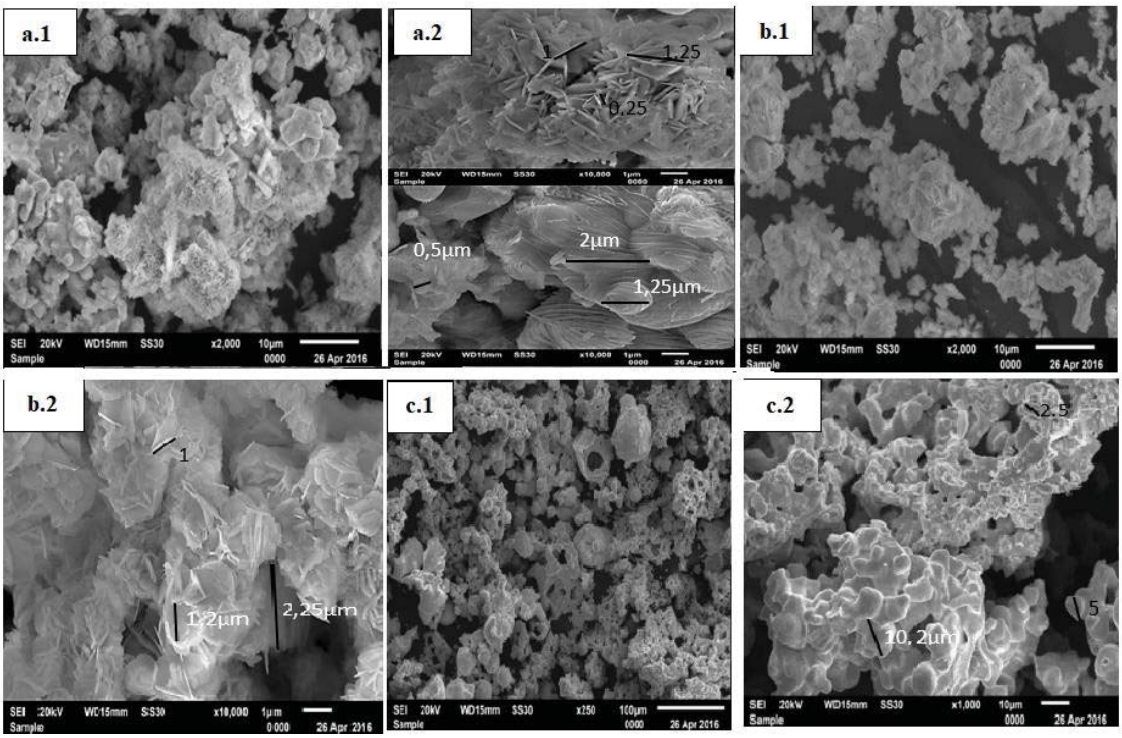

FIGURE 4. SEM images of bismuth oxide synthesised in different calcination temperature : (a.1) 500 °C magnification of 2000x, (a.2) 500 °C magnification of 10.000x, (b.1) 600 °C magnification of 2000x, (b.2) 600 °C magnification of 10.000x, (c.1) 700 °C magnification of 250x, (c.2) 700 °C magnification of 1000x.

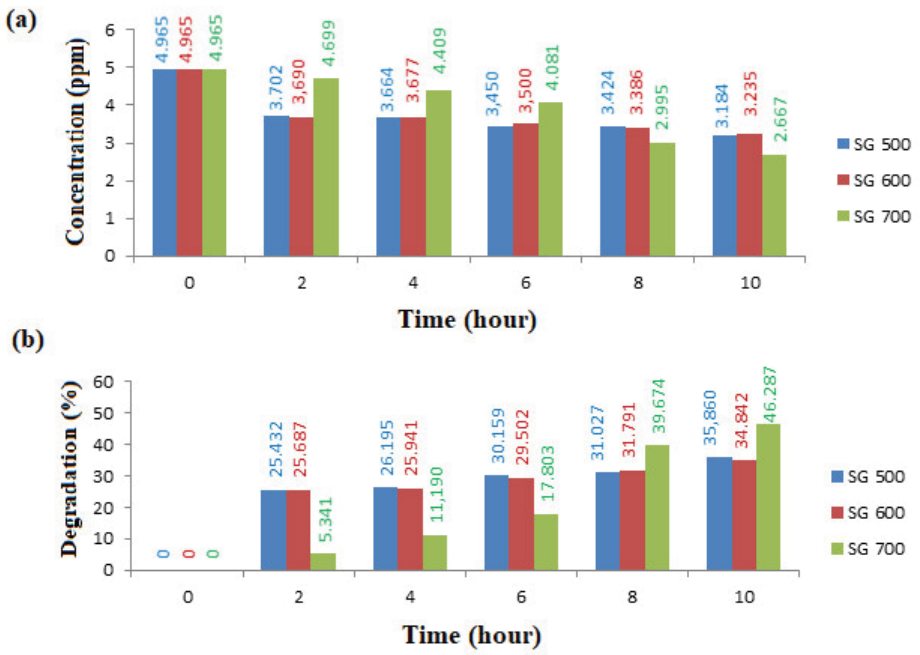

FIGURE 5 Photocatalitic result of bismuth oxide (a) Effect of the presence of photocatalyst on methyl orange degradation; (b) Percentage degradation of methyl orange by bismuth oxide catalyst with variety of calcination temperature

According Yuanhua et al. (2011) [16], the dye degradation activity generally follows the order reaction kinetics 1, namely:

$$ln\ C_t = \ln C_0 - k\ t \quad (1)$$

with k = rate constant at orde 1 (s^{-1}), C_0 = initial consentration methyl orange solution (ppm), and C_t = the concentration of methyl orange solution at certain time. Based on graphic kinetics degradation of methyl orange by bismuth oxide catalyst, we can see in the Fig.6 can be obtained by linear equation, so that known the rate reaction rate degradation (k) methyl orange which can be known from slope (m).

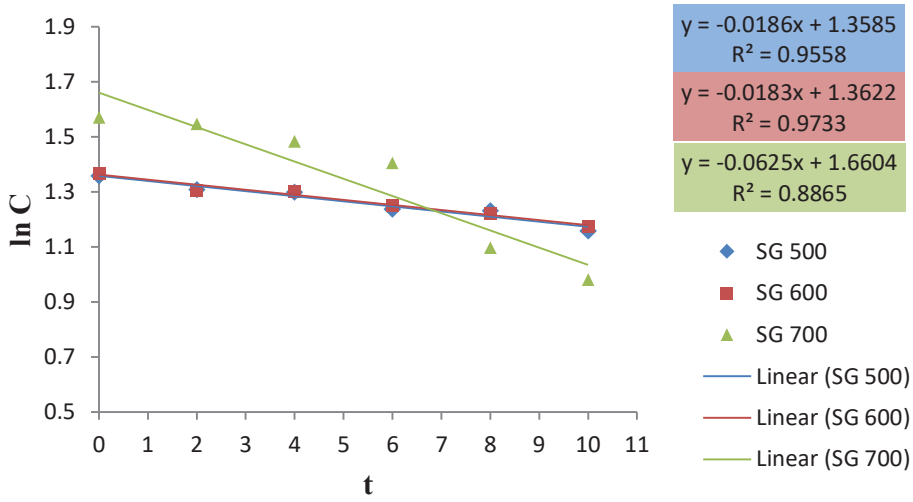

FIGURE 6. Graph of kinetics study of methyl orange degradation by bismuth oxide with variety of calcination temperature

Bismuth oxide synthesized with calcination temperature at 700 °C has the highest rate constant, we can see Fig 7. it is suitable with the literature[15] because the product has a homogeneous crystal structure that is α-Bi_2O_3 (monoclinic) and porous so it has the best of degradation activity of methyl orange.

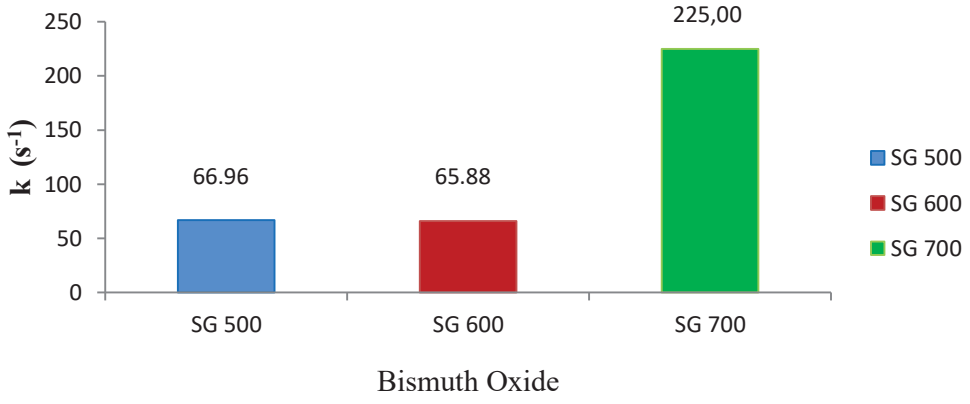

FIGURE 7. The rate constant of methyl orange degradation by bismuth oxide with variety of calcination temperature

CONCLUSION

Bismuth oxide was successfully synthesised using sol gel method. The calcination temperature influenced the physicochemical properties including crystal structure, morphology and size of particles. Moreover, the photocatalytic activity test showed that calcination temperature at 700 ^{0}C gave best performance on degradation

of methyl orange. Eventhough the particle size of the product synthesized in this condition is bigger than others, the morphology of product depicted the presence of pores resulting in higher adsorption of methyl orange.

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