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Submission date: 22-Oct-2020 10:23AM (UTC+0700)
Submission ID: 1422762632
File name: C2MS_Astuti_2020_IOP_Conf._Ser.__Mater._Sci._Eng._833_012061.pdf (599.19K)
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Character count: 13453

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IOP Conf. Series: Materials Science and Engineering **833** (2020) 012061 doi:10.1088/1757-899X/833/1/012061

Photocatalytic Performance of Bismuth Oxide Prepared by Citric Acid-Fueled Solution Combustion on Decolorisation of Organic Dye Molecules

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Abstract. Bismuth oxide can be applied as photocatalyst. The purpose of this study was to synthesize bismuth oxide using the solution combustion method, identify the characteristics of bismuth oxide using FTIR and XRD, and determine the photocatalytic activity of the obtained bismuth oxide to decolour Remazol Black B and Rhodamine B dye molecules. The results showed that bismuth oxide was formed with yellow colour after calcination convinced with the presence of Bi-O and Bi-O-Bi functional groups and having 95.38% similarity using Match software. The photocatalytic activity test shows that the obtained bismuth oxide enabled to decolour RBB, RhB dyes well.

1. Introduction

Bismuth Oxide is a material that has many advantages and can be applied in various fields including solid oxide fuel cells, gas sensors, high temperature super conductor materials, functional ceramics and photocatalyst materials [1]. One of methods for bismuth oxide synthesis is solution combustion [2]. This method has various advantages such as fast and simple process, as well as easy to do [3].

The solution combustion method is a synthesis method whose main components consist of oxidants and fuels, with processes that involve high temperatures [3]. Fuels commonly used in the solution combustion method are citric acid [2], urea [4], glycine [5], and hydrazine [6]. While, the oxidant used in the method solution combustion is metal nitrate because the nitrate group acts as an oxidizing agent and has great solubility and allows for the homogenization of larger product yields [4].

Y. Astuti et al [7] synthesized bismuth oxide using solution combustion method from bismuth subnitrate (Bi5O(OH)9(NO3)4) and citric acid as oxidant and fuel, respectively. The results showed that bismuth oxide enable to decolor methyl orange by 38% for 12 hours with a UV lamp. In addition, by this method, synthesis of bismuth oxide with bismuth nitrate pentahydrate as oxidant and citric acid as fuel was also undertaken [8]. Specifically, the effect of a variation of moles ratio of fuel-oxidant on the photocatalytic activity of obtained products for the decolorization of methyl orange dyes was investigated. The results showed that the moles ratio 1 presented a good photocatalytic activity in methyl orange decolorization. Therefore, in this paper the performance of bismuth oxide synthesized using solution combustion method with moles ratio 1 for decolorization of other dye molecules, namely, rhemazol black B (RBB) and rhodamine B (RhB) will be presented. Both dyes are usually applied for Batik coloring. It should be borne in mind that almost 40% dyes applied for Batik coloring are disposed into environment [9]. In a huge amount of these dyes makes the environment unfriendly for human. Therefore, it must be overcome, for example by using photocatalyst bismuth oxide. The investigation is

expected to contribute on science about performance of bismuth oxide in decolorizing other dyes besides methyl orange.

2. Experimental Methods

2.1. Materials

The materials used were bismuth nitrate pentahydrate from Sigma-Aldrich USA, distilled water, nitric acid from Merck Germany, citric acid Merck Germany, Remazol Black B and Rhodamine B (RhB) from Sigma-Aldrich USA.

2.2. Research Procedure

2.2.1. Synthesis of Bismuth Oxide. Bismuth oxide synthesis was undertaken using the solution combustion method. As much as 2.42 g bismuth nitrate pentahydrate was dissolved in 10 mL nitric acid 65% then added 1.26 g of citric acid. The mixture was stirred until dissolved and then heated at temperature of 300 °C for 8 hours. Obtained powder (Figure 1a) was allowed to cool then calcined for 4 hours at 700 °C. This procedure is based on Y Astuti report [8].

2.2.2. Product Characterization. The obtained products were then characterized using FTIR and XRD. FTIR analysis was carried out on the products before and after calcination to identify the presence of Bi-O or Bi-O-Bi functional groups. The obtained product after calcination was also characterized using XRD to convince that bismuth oxide was successfully formed.

2.2.3. Photocatalytic Activity. Photocatalytic activity of product after calcination was then determined by applying it to decolour RBB and RhB dye molecules. As much as 0.1 g of synthesized bismuth oxide was added into 50 mL RBB 25 ppm and subsequently was irradiated using UV-A (3.94-3.1 eV) at time variation of 1, 2, 3, 4, and 5 hours. This procedure was applied for RhB (5 ppm) solution as well. After irradiation, the solution was centrifuge to separate bismuth oxide powder and dye solution. The filtrate was then analyzed using spectrophotometer to determine the absorbance change of the solution after photocatalysis. This absorbance was further used to calculate the percentage of decolorization by applying formula:

$$decolourization\ percentage = \left(\frac{C_0 - C_t}{C_0}\right) \times 100\ \% \quad (1)$$

with C_0 = initial concentration of dye molecule (ppm), C_t = final concentration of dye molecule (ppm) at t time.

In addition, to convince that prepared bismuth oxide enabled decolourising RBB and RhB, these dyes were also irradiated using UV-A for 5 hours without the presence of bismuth oxide. Photocatalysis of both dyes using commercial bismuth oxide was also undertaken at a time variation of 0.5 to 3 hours with 0.5 hour interval.

3. Result and Discussion

3.1. Synthesis and structural analysis of the obtained products

The result showed that the yellow powder in the obtained product after heating for 8 hours, Figure 1a, is very little indicating bismuth oxide was formed. It is dominated by black and dark green powder indicating that there is still carbon in the sample derived from citric acid. After calcination for 4 hours at a temperature of 700oC, the mixed color powder turned to yellow powder as shown in Figure 1b. It is indicated that bismuth oxide was formed.

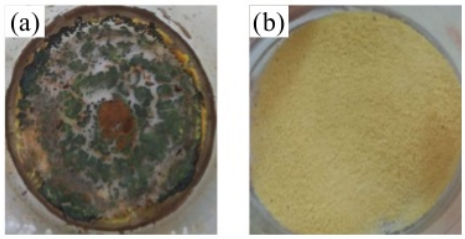

Figure 1. The obtained products (a) after 8 hour heating and (b) after 4 hour calcination.

The obtained products before and after calcination were analyzed using FTIR and the results can be seen in Figure 2. Product before calcination showed there is a peak at 850 cm-1 wavenumber attributed to Bi-O-Bi functional group [10] and a peak at the wavenumber of 1387 cm-1 indicating the existence of the Bi-O vibration mode [10]. These peaks convinced that bismuth oxide was formed in the product before calcination even though the product was physically not only yellow but mixed color as presented in Figure 1a. The mixed color product indicating there are still many impurities derived from bismuth nitrate pentahydrate and citric acid as starting material shown by the appearance of many peaks besides Bi-O-Bi and Bi-O. The peaks at wave numbers 1080 and 1132 cm-1 on sample before calcination are assigned as vibration modes of CO stretching [11] coming from citric acid. Moreover, peaks at wavenumbers 1525 and 1587 cm-1 were also present attributed to vibrations of OH functional group [11]. The stretching OH vibration was also observed at wavenumber of 3455 cm^{-1}. In addition, peak at 1300 cm-1 is also assigned as vibrations of N-O from NO3 [12]. Due to the impurities presence, the product was then calcined at 700 °C. FTIR spectra among the products and starting materials can be seen in Figure 2.

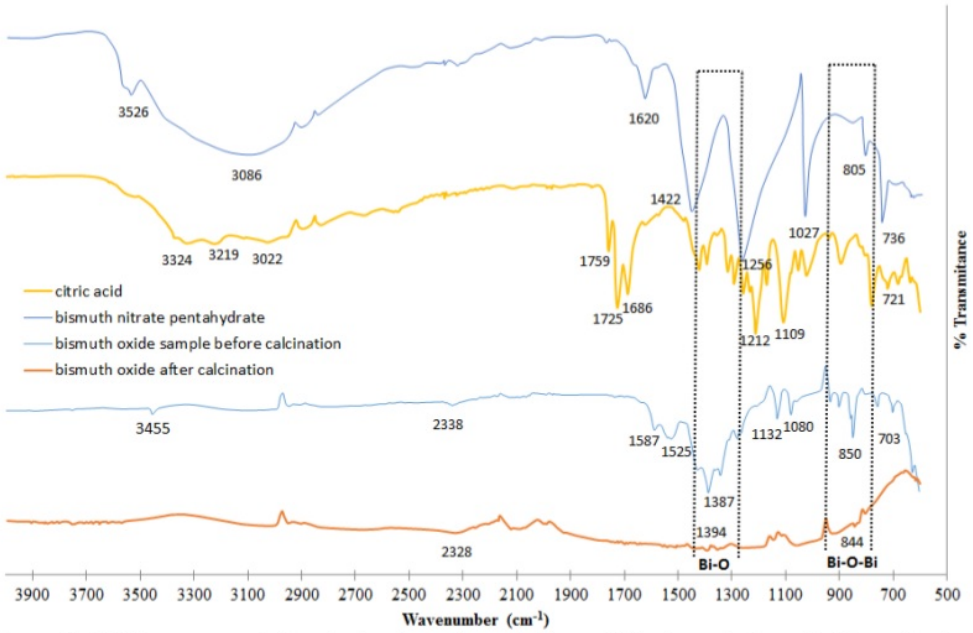

Figure 2. FTIR spectra of the obtained products before calcination, citric acid, bismuth nitrate pentahydrate and the obtained products after calcination.

FTIR spectrum of sample after calcination showed that there is a peak at wavenumber of 844 and 1394 cm-1 indicating the formation of Bi-O-Bi bond and Bi-O respectively [10]. These results are in

accordance with FTIR spectra of pure bismuth oxide [8, 13]. In the meantime, other peaks were not observed in this FTIR spectrum. Therefore, these peaks confirmed that the functional groups of the impurity largely disappeared, and the product obtained after calcination be purer than the product before calcination.

Fu et al [12] reported that in the wave number of 1300~ there was not only a Bi-O group but there was also a NO3- group as stated previously. The FTIR spectrum of sample before calcination has a peak with sharper intensity than that of sample after calcination. The intensity of this peak in both samples can be calculated through the ratio of absorbance approach at the wavenumber of around 1400-1300 cm-1 with a control wavenumber of about 2300 cm-1. The comparison of peak intensity of both samples is presented in Table 1.

Table 1: Comparison of absorbance in wave numbers of 1300 cm-1 in samples before and after calcination and control group

Treatment	Wavenumber absorption 1300 cm^{-1}: Control group
Before Calcination	4.18
After Calcination	1.25

Based on Table 1, it can be seen that the ratio of absorbance decreases after calcination. The decrease is assumed to occur because the NO3- group observed at wavenumber of 1300 cm-1 was lost as a result of the calcination process.

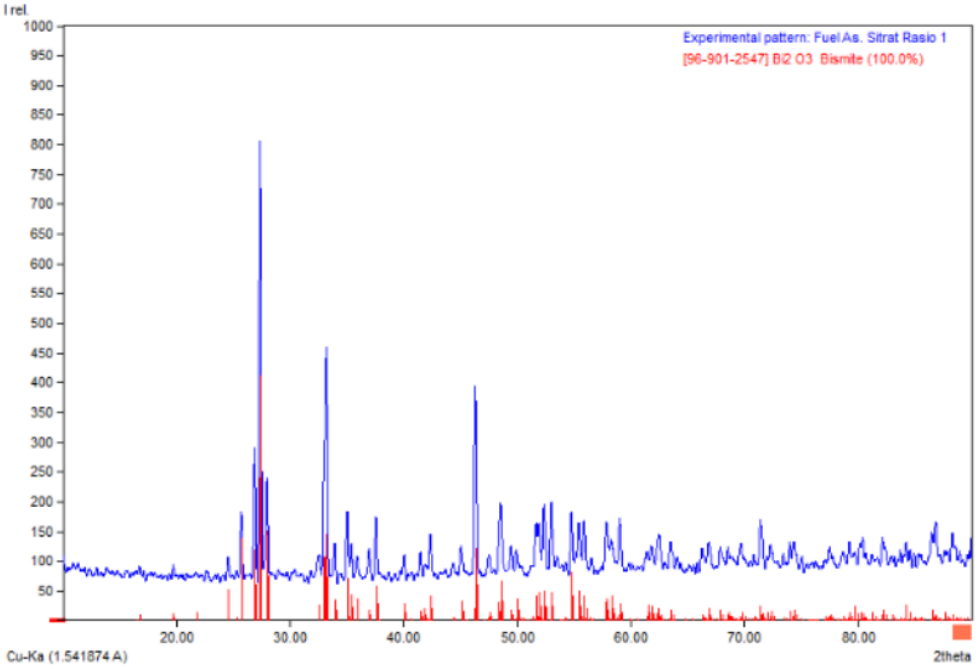

Figure 3. Diffractogram of bismuth oxide analysis results match.

The crystal structure of product after calcination was analyzed using XRD. The diffractogram was then analyzed using match software to determine the content of the product. As seen in Figure 3, the product has 95.38% similarity with reference Malmros indicating the presence of bismuth oxide

(Bi2O3) [14]. This result confirmed that bismuth oxide was formed after calcination process and the impurity was almost 4.62%.

3.2. Photocatalytic Activity

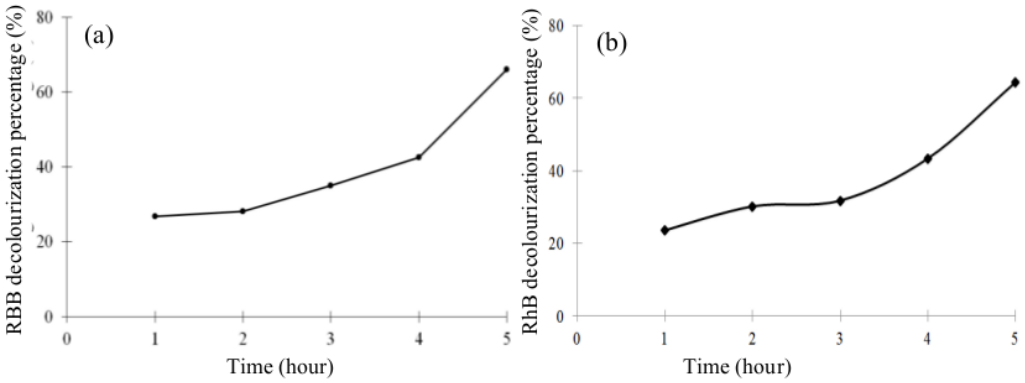

Figure 4. Photocatalytic activity of prepared bismuth oxide on decolorization of (a) RBB, and (b) RhB.

The photocatalytic activity of bismuth oxide was then investigated by applying in decolorization of RBB and RhB dye molecules. Figure 4 showed the decolorization percentage of dye molecules by bismuth oxide. It shows that after 5 hours irradiation, decolorization of RBB was around 66 % while RhB was 64 %. The difference of decolorization percentage may be due to the differences in dye molecules stability. Meanwhile, decolorization of RBB and RhB by commercial bismuth oxide as presented in Figure 5 showed that after 3 hours irradiation, decolorization percentages of RBB and RhB were 66 and 79%, respectively. This result indicated that resulting products content impurity.

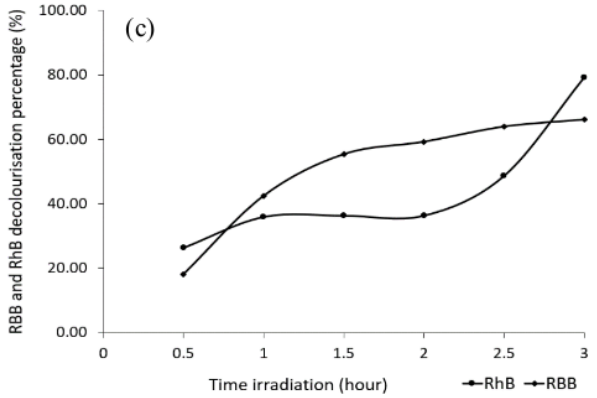

Figure 5. Photocatalytic activity of commercial bismuth oxide on decolorization of RBB and RhB.

Furthermore, in order to clarify that prepared bismuth oxide enabled to decolorized RBB and RhB, these dyes were irradiated using UV-A without presence of bismuth oxide. The result showed that after irradiation for 5 hours, only 1.4% and 9% of RBB and RhB were decolorized.

Decolorization of dyes by bismuth oxide occurs when the light source illuminates the surface of the semiconductor bismuth oxide so that resulting in electrons (e−) to be excited from the valence band to the conduction band, and forms a hole (h+) in the valence band. Holes in the valence band react with hydroxyl anions (OH−) to form hydroxyl radicals (HO•). The electrons in the conduction band react with O2 from the atmosphere and form superoxide radical anions (O2•-) which then react with H+ form HO2•. After that, HO2• reacts with electrons to produce HO2−which further reacts with H+ to form H2O2 oxidants. H2O2 reacts with O2•− to produce •OH, OH− and O2. Strong oxidizing agents such as OH−, HO •, and H2O2 oxidize organic dye molecules such as RBB and RhB into simpler molecules [15-17].

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Acknowledgement

The authors would like to thank the Ministry of Research, Technology and Higher Education, Republic of Indonesia for financial support through scheme of "Penelitian Dasar" in 2018 (No. 101-71 / UN7.P4.3 / PP / 2018) and 2019 (No. 101-71 / UN7.P4.3 / PP / 2019).

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