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Molten Salt Synthesis of Photocatalyst Material $SrBi_4Ti_4O_{15}$ for Methylene Blue Degradation

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Abstract

The four-layered Aurivillius $SrBi_4Ti_4O_{15}$ compound has been reported to be potentially used as a photocatalyst material. In this research, $SrBi_4Ti_4O_{15}$ was prepared by the molten salt method using NaCl/KCl and then used to degrade methylene blue. The analysis of sample diffractogram indicated that the $SrBi_4Ti_4O_{15}$ was obtained but there was still the impurities of $Bi_4Ti_3O_{12}$. A micrograph showed that particle shape of $SrBi_4Ti_4O_{15}$ is plate-like (sheets) with a lot of agglomeration. The band gap energy of $SrBi_4Ti_4O_{15}$ is 3.14 eV (394.85 nm), according to the Kubelka-Munk calculation from the spectrum of reflectance. The photocatalytic test results showed that $SrBi_4Ti_4O_{15}$ degraded methylene blue to 47.8% in 120 minutes.

Keywords: Aurivillius, degradation, methylene blue, photocatalyst material, $SrBi_4Ti_4O_{15}$

1. Introduction

Dye pollutants caused by industrial activities have become a global concern because of their serious environmental impact, such as carcinogenicity and inability to be decomposed by biological processes (Schwarzenbach et al., 2006; Cripps et al., 1990). Various methods have been developed to addres this issue, and one of the potential methods is photocatalyst because of its low cost, chemical stability, low toxicity, and no waste generated (Fujishima et al., 2000). Methylene blue is a dye commonly used in the textile industry. Because of its complex aromatic structure, methylene blue is chemically stable and difficult to degrade via biological processes (Fadillah et al., 2018). In addition, methylene blue is toxic to humans and capable of harming aquatic ecosystems by inhibiting biota growth and chelating metal ions, making them toxic to aquatic organisms (Sheng et al., 2009). Therefore, special handling methods are required, such as the use of semiconductor photocatalysts.

One of crystal structure compound reported to be potentially used in photocatalyst technology is the Aurivillius structure family (Wang et al., 2010; Lu et al., 2015). Aurivillius compound family is a metal oxide compound consisting of a pseudo-perovskite layer $(A_{n-1}B_nO_{3n+1})^{2-}$ and a bismuth $(Bi_2O_2)^{2+}$ layer alternately arranged along the *c* axis with *A* is the larger cation (1.34-1.61 Å) with dodecahedral coordination, such as Na^+, K^+, Ba^{2+}, Pb^{2+}, Bi^{3+}, Sr^{2+}, rare earth metals, or their mixtures, and *B* is a smaller cation (0.59-0.65 Å) with octahedral coordination, such as Mo^{6+}, Ti^{4+}, Ta^{5+}, Nb^{5+}, W^{6+}, and *n* represent the number of pseudo perovskite layers (Borg et al., 2002). Many Aurivillius compounds can be applied as photocatalyst material, such as $Bi_4Ti_3O_{12}$, $CaBi_2Ta_2O_9$, $SrBi_2Ta_2O_9$, $BaBi_2Ta_2O_9$, and $SrBi_4Ti_4O_{15}$ (Li et al., 2018; Li et al., 2008; Tu et al., 2019). The photocatalytic properties of Aurivillius compounds are related to electrons in the Bi 6*s* and O 2*p* orbitals (valence band) (Wang et al., 2020). $SrBi_4Ti_4O_{15}$ is a four-layer Aurivillius compound with a band gap energy of 3.0 eV that has better photocatalytic activity in reducing CO_2 gas than $Bi_4Ti_3O_{12}$, TiO_2, and BiOBr (Tu et al., 2019). In addition, Haikal and Prasetyo (2021) also reported that $SrBi_4Ti_4O_{15}$ can degrade rhodamine B by 41.10% in 120 minutes. However, the photocatalyst properties of four-layer Aurivillius compound report is still limited. In addition, the activity photocatalyst of $SrBi_4Ti_4O_{15}$ on dye compounds degradation, such as methylene blue is still not widely reported.

One of the factors that affected the Aurivillius compound's photocatalytic activity is its particle morphology (Cheng et al., 2021). Previous studies have discovered that plate-like Aurivillius compounds are an effective photocatalysts (Liu et al., 2019; Zhao et al., 2018). Chen et al., (2016) suggested that $Bi_4Ti_3O_{12}$ nanosheet has a good ability to degrade rhodamine B because of the high number of active sites on its surface particle, and can inhibit the rate of recombinant electrons (e^-)-hole (h^+) (Chen et al., 2016).

Many researchers have synthesized the four-layer Aurivillius compound family using various methods, such as (a) solid state reaction (Ding et al 2020) (b) molten salt synthesis (Zhao et al., 2014) (c) hydrothermal (Sardar, 2012), and sol-gel (Mamidi et al, 2018). The molten salt method has been used by many researchers to synthesize the compound $SrBi_4Ti_4O_{15}$, and to deliver typical particle morphology (Garcia-Guederrama et al., 2005; Zulhadjri et al., 2011; Kimura, 2011). In addition, the molten salt method has several advantages, such as (a) a low reaction temperature, (b) control over particle size and shape, (c) increased homogeneity, and (d) green synthesis (Kimura, 2011; Rahman, 2003).

The crystal growth process in the molten salt method is divided into two stages: (a) nucleation and (b) crystal growth. During the nucleation step, a crystal nucleus is formed and expanded through the crystal growth process (Kimura, 2011; Marela et al., 2021). In addition, the morphology of the particles obtained by the molten salt method was influenced by several factors, including the time and temperature of synthesis, the salt/product ratio, and the type of salt flux. Controlling these parameters would result in particles with distinct morphology (Akdogan et al*.*, 2006).

Mixed salts (such as NaCl/KCl, and Na_2SO_4/K_2SO_4) are often used in the molten salt synthesis which aims to get lower the melting point temperature. Zhao et al (2014) synthesized plate-like $Bi_4Ti_3O_{12}$ using mixed Na_2SO_4/K_2SO_4 salt at temperature range 850-950 ^oC. It indicates that mixed salt can be used in molten salt synthesis of Aurivillius compound to produce the plate-like (sheet) particle. Chang et al. (2014) reported that they successfully synthesized $SrBi_4Ti_4O_{15}$ compound using the molten salt method, and obtained particles in the form of microplatelets (microsheets). However, studies on the use of the plate-like $SrBi_4Ti_4O_{15}$ obtained a molten salt method to degrade dye compound is still limited thus further studies are needed to reveal its photocatalyst properties. Therefore, in this research, we synthesized plate-like $SrBi_4Ti_4O_{15}$ via the molten salt method using mixed salt NaCl/KCl and investigated its photocatalytic activity in degrading methylene blue.

2. Methodology

2.1. Materials

The chemicals used in this research were Bi_2O_3 (Himedia, 99% powder), TiO_2 (Sigma-Aldrich, 99% powder), $SrCO_3$ (Sigma-Aldrich, 99.9% powder), NaCl (Merck, 99%), KCl (Merck, 99%), $AgNO_3$ (Merck, 99%), and acetone (Merck).

2.2. Synthesis of $SrBi_4Ti_4O_{15}$

The molten salt method was used to synthesize the Aurivillius $SrBi_4Ti_4O_{15}$ compound target, with a salt mixture of NaCl/KCl (1:1) and a mole ratio of the target compound and salt of 1:7. The precursors Bi_2O_3, TiO_2, and $SrCO_3$ were mixed as the first step in the synthesis. Stoichiometrically, the precursor mass requirement was calculated with a target mass of three grams. The mixture was then ground until homogeneous with an agate mortar for one hour, with acetone added during the grinding process. The mixture was calcined for eight hours at 700°C. The mixture was then homogenized for one hour with an agate mortar using NaCl/KCl salt (salt requirement is based on stoichiometric calculations). The mixture was then re-calcined in a furnace for eight hours at 750 and 800°C. The product samples were washed with hot distilled water to remove the NaCl and KCl salts. The $AgNO_3$ test was used to determine the salt content of NaCl/KCl. Finally, the product was dried for three hours in a 110°C oven.

2.3. Sample Characterization

Characterization techniques used in this study included *X*-ray diffraction (XRD), scanning electron microscopy-energy dispersive *X*-ray spectroscopy (SEM-EDS) and ultraviolet-visible diffuse reflectancy spectroscopy (UV-Vis DRS). The XRD instrument was used to determine the type of compound in a sample. The measurements were taken at 2θ = 3–90° using a Rigaku Miniflex diffractometer with a Cu K radiation source (λ = 1.540593). SEM-EDS characterization was used to analyze particle morphology, and to determine the constituent elements of a compound. This measurement was taken with a JEOL JSM-6360LA and a magnification of 15.000x. The UV-Vis DRS instrument was used to study the reflectance pattern of light, and the band gap energy would be calculated using the Kubelka-Munk equation. A Thermo Scientific Evolution

220 spectrometer with a wavelength range of 200–800 nm was used for the measurements.

2.4. Methylene Blue Degradation (Photocatalytic Activity Test)

At a concentration of 4 ppm methylene blue, 0.1 gram of $SrBi_4Ti_4O_{15}$ was added to 100 mL of methylene blue. The mixture was then stirred in the dark for 30 minutes with a magnetic stirrer to achieve adsorption-desorption equilibrium. Then, it was exposed to 8 Gaxindo T5 N093 8 Watt ultraviolet lamps in a photoreactor for 30, 60, 90, and 120 minutes. UV-Vis spectroscopy (Thermo Scientific Evolution 220 spectrometer) with a maximum wavelength of 664 nm was used to measure the concentration of methylene blue as a result of the degradation process.

3. Results and Discussion

3.1. Characterization Sample Product

Figure 1 shows the sample diffractogram, and a $SrBi_4Ti_4O_{15}$ compound was obtained based on the conformity of the peaks with the Joint Committee on Powder Diffraction Standard (JCPDS) data No.43-0973, with typical peaks at position 2θ = 21.76, 23.1, 30.3, 32.9, 37.2, 38.7, 39.7, 47.2, 52.5, 57.2, 64.57, and 69.2°. Figure 1 also shows the peak diffraction shape is sharp that indicates the crystallinity of the sample is good. However, the diffractogram shows additional peaks indicating the presence of $Bi_4Ti_3O_{12}$ impurities at 2θ = 16.3, 26.93, 48.09, 51.39, and 62.52°. This impurity phase is the result of a reaction between Bi_2O_3 and TiO_2 precursors that does not react with $SrCO_3$.

One of the stages in the molten salt synthesis method is the rearrangement and diffusion of precursor species in the molten salt. At this stage, the reaction between the precursors has occurred in the flux salt media at above melting point of NaCl/KCl salt. As know well that the salt type influenced to the synthesis mechanism in molten salt synthesis. The presence of impurities ($SrCO_3$) indicates that the flux of NaCl/KCl salt (1:1) is still not suitable for making a reaction. Therefore it may $SrCO_3$ precursor does not diffuse well in the NaCl/KCl flux (Qiu et al., 2017).

Furthermore, the average crystallyte size was determined using the Debye-Scherrer equation ($D = \frac{K\lambda}{\beta \cos\theta}$ with D is the crystallite size (nm), λ is the wavelength of X-ray source with Cu k-α (1.5406 nm), K is the Scherer constant (0.9), β is full width half maximum peak (radians), and θ is diffraction angle) (Benali et al., 2020). According to the calculations, the average crystal size of the sample is ~80 nm.

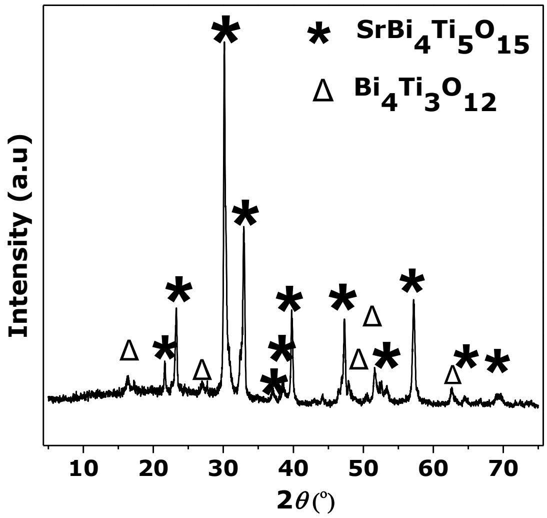

Figure 1. The diffractogram of $SrBi_4Ti_4O_{15}$.

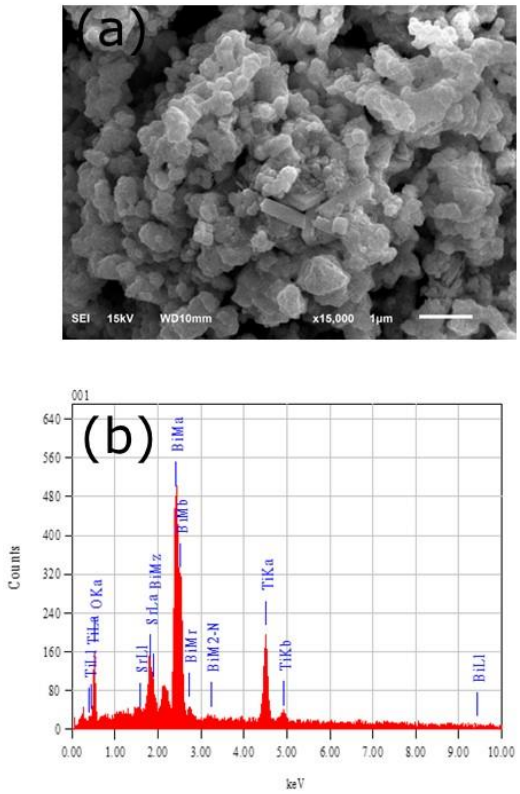

Figure 2. Micrograph and EDS spectrum of $SrBi_4Ti_4O_{15}$.

Figure 2 shows a micrograph of the $SrBi_4Ti_4O_{15}$ compound with particle size about 250-500 nm. The morphology has a plate-like shape and agglomerated particles. Figure 2 also shows that the particle size is small and uniform relatively. It indicates that the nucleation rate is higher than the growth particle rate. The crystal seed is formed at nucleation stage therefore the higher nucleation rate produce large amounts of small-sized particles. Meanwhile the particle agglomeration due to high synthesis temperatures condition (Wang and Chen, 2012). In this research used 750 and 850 °C, meanwhile the melting point of NaCl/KCl is 657 °C. The EDS spectra (Figure (2(b)) confirms constituent elements (Sr, Bi, Ti, and O) present in the sample and there is no other element that indicates impurities. The results of percent elemental weight are summarized in Table 1.

Table 1. The EDS results.

Sample	Elemental weight (%)			
	Sr	Bi	Ti	O
$SrBi_4Ti_4O_{15}$	6.75	65.62	16.49	11.14

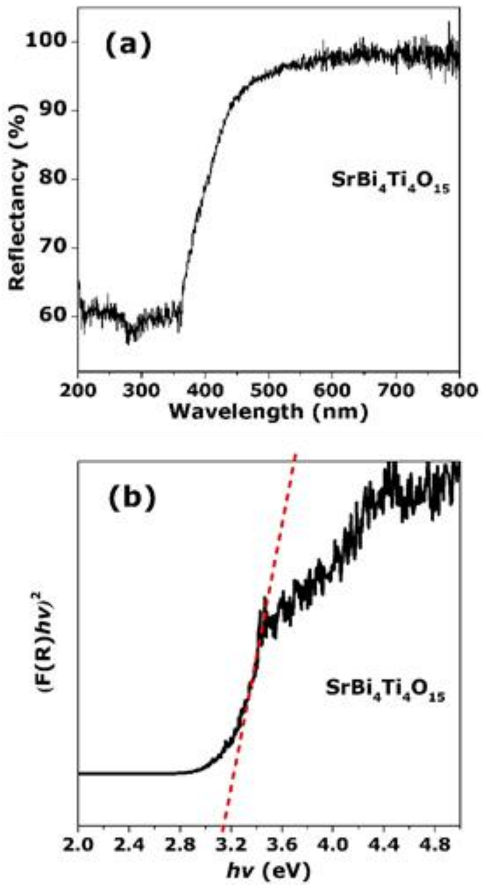

Figure 3. (a) The UV-Vis DRS spectrum, and (b) Tauc plot of $SrBi_4Ti_4O_{15}$.

Figure 3 shows the Kubelka-Munk equation's UV-Vis DRS spectrum and Tauc plot. According to the Kubelka-Munk calculation, the band gap energy of $SrBi_4Ti_4O_{15}$ is 3.14 eV, which corresponds to a wavelength of 394.85 nm. It relates to the amount of energy required to excite electrons from the valence band (VB) in the O 2*p* and Bi 6*s* orbitals to the conduction band (CB) in the Ti 3*d* orbitals (Naresh and Mandal, 2014). The sample's band gap energy is higher than that reported previously by Tu et al., (2019). Impurities in the sample may influence the bandgap energy. The band gap energy obtained also shows that the $SrBi_4Ti_4O_{15}$ sample still works in the UV light region, and it gives a disadvantage for utilization as photocatalyst material.

3.2. Activity Photocatalyst Test

3.2.1. Adsorption-desorption test

Methylene blue degradation occurs on the surface of materials, so dye adsorption by $SrBi_4Ti_4O_{15}$ is a possibility. Figure 4 depicts the UV-Vis spectrum of the adsorption-desorption test, demonstrating that absorbance slighty decreases as methylene blue concentration decreases. It corelates to interaction between $SrBi_4Ti_4O_{15}$ surface and methylene blue. It proves that the $SrBi_4Ti_4O_{15}$ compound is capable of adsorbing methylene blue. $SrBi_4Ti_4O_{15}$ adsorption properties are comparable to those reported by Ziyaadini and Ghashang (2021), who reported the ability of Co doped $SrBi_4Ti_4O_{15}$ compounds to absorb Rhodamine B. They also reported that the adsorption mechanism between Rhodamine B and Co-doped $SrBi_4Ti_4O_{15}$ is chemical adsorption (Ziyaadini and Ghashang, 2021).

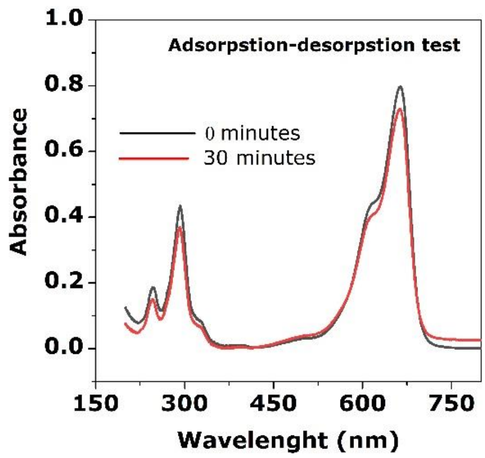

Figure 4. The spectra of adsorption-desorption test.

3.2.2 Metylene blue degradation test

The absorption spectra of methylene blue degradation by $SrBi_4Ti_4O_{15}$ are shown in Figure 5, and it can be seen that the absorption intensity decreases as a function of time. It indicates that methylene blue concentration decrease as a result degradation process by

$SrBi_4Ti_4O_{15}$. The calculation results of methylene blue concentration after degradation process were presented in Figure 6. According to this result, increasing the photocatalytic process time can enhance the percentage of degradation, with methylene blue being reduced up to 47.8% in 120 minutes. These results imply that the $SrBi_4Ti_4O_{15}$ photocatalyst exhibits good photocatalytic activity.

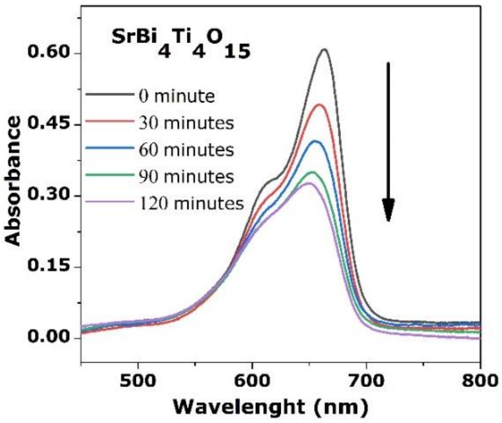

Figure 5. The absorption spectra of methylene blue degradation by $SrBi_4Ti_4O_{15}$.

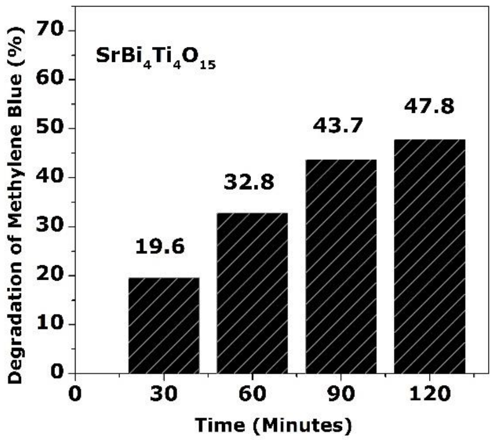

Figure 6. Percentage degradation of methylene blue by $SrBi_4Ti_4O_{15}$.

Figures 4 and 5 also suggest that the photocatalyst process plays a significant role in reducing the concentration of methylene blue because the contribution of the adsorption process to reducing the concentration of methylene blue is small. Using the dye degradation mechanism by another photocatalyst semiconductor compounds thus the degradation mechanism of methylene blue by $SrBi_4Ti_4O_{15}$ can be describe as follows: the light hit the surface of $SrBi_4Ti_4O_{15}$ as a result the electrons (e^-) move from valence band (*VB*) to the conduction band (*CB*) and cause holes (h^+) formation in the VB. Furthermore, e^- (*VB*) react with O_2 species to form O_2 superoxide anion ($O_2^{-}\cdot$), while h^+ (*CB*) react with OH/H_2O to form OH radical (OH·). Then, the existence of radicals reacts with dyes compound and produced new smaller molecule (methlene blue degradation) (Cheng et al., 2021 and Mamidi et al., 2019)

When compared to $Bi_4V_2O_{11}$ (some other Aurivillius compound), the degradation ability of $SrBi_4Ti_4O_{15}$ compounds is lower, which is possible because of (a) the lower bandgap energy of $Bi_4V_2O_{11}$ (2.08 eV), (b) the smaller particle size of $Bi_4V_2O_{11}$, it can be explained because the dye degradation process occurs on the surface of photocatalyst compound thus the larger of surface area give the higher degradation ability. (Lu et al., 2015), and (c) the $SrBi_4Ti_4O_{15}$ particle in agglomerated form. Meanwhile, Pellegrino et al., (2017) suggested that the agglomeration particles can interfere with the absorption process of photon radiation, then causing the photocatalytic activity to decrease (Pellegrino et al., 2017). The another factor affected to photocatalyts activity is e^--h^+ recombinanation rate that correlates the life time of e^--h^+ (Chen et al., 2016). Meanwhile the existence of e^--h^+ is very important factor in dyes compound degradation, but in this research did not conduct the photoluminescence (*PL*) spectroscopy measurement. Thus there is no information about the influence of e^--h^+ recombination rate to methylene blue degradation result.

4. Conclusion

The compound $SrBi_4Ti_4O_{15}$ was successfully synthesized, but an impurity phase of $Bi_4Ti_3O_{12}$ was discovered that indicates the synthesis condition did not enough to reaction accomplish. The morphology of the $SrBi_4Ti_4O_{15}$ compound that was synthesized was plate-like with many agglomerations. The higher synthesis temperature may induce the agglomeration formed. The $SrBi_4Ti_4O_{15}$ compound has a bandgap energy of 3.14 eV (394.85 nm). The difference band gap energy obtained sample with previous report band gap energy of $SrBi_4Ti_4O_{15}$ due to the existence of impurities phase in the sample. The photocatalytic activity test in degrading methylene blue up to 47.8 % in 120 minutes. Despite the $SrBi_4Ti_4O_{15}$ compound can act as an adsorbent but the role of the photocatalyst mechanism is more powerful.

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