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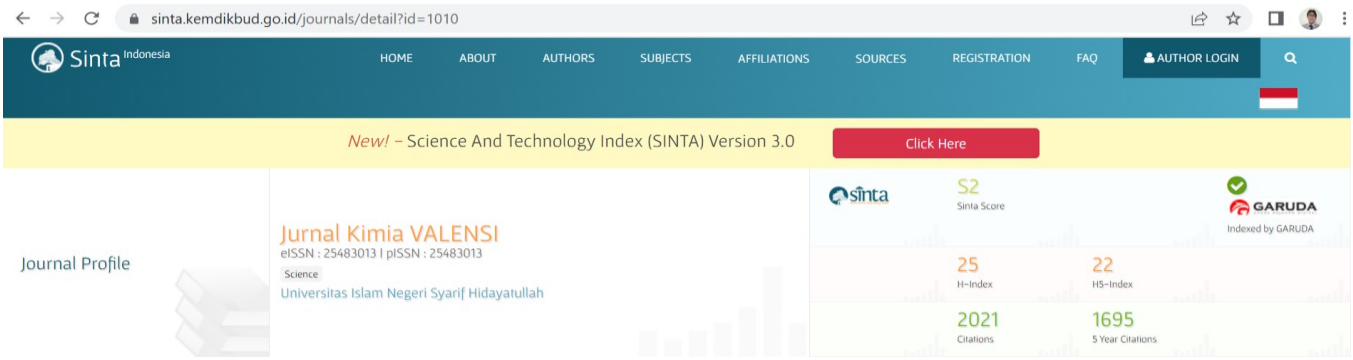

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The Starting Material Concentration Dependence of Ag_3PO_4 Synthesis for Rhodamine B Photodegradation under Visible Light Irradiation

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Abstract

Synthesis of Ag_3PO_4 photocatalyst under the varied concentrations of $AgNO_3$ and $Na_2HPO_4{\cdot}12H_2O$ as starting material has been successfully synthesized using the co-precipitation method. The concentration of $AgNO_3$ is 0.1; 0.5; 1.0; and 2.0 M, whereas $Na_2HPO_4{\cdot}12H_2O$ is 0.03; 0.17; 0.33; and 0.67 M, respectively. The co-precipitations were carried out under aqueous solution. As-synthesized photocatalysts were examined to degrade Rhodamine B (RhB) under blue light irradiation. The results showed that varying concentrations of starting materials affect the photocatalytic activities, the intensity ratio of [110]/[200] facet plane, and their bandgap energies of Ag_3PO_4 photocatalyst. The highest photocatalytic activity of the sample was obtained by synthesized using the 1.0 M of $AgNO_3$ and 0.33 M of $Na_2HPO_4{\cdot}12H_2O$ (AP-1.0). This is due to the high [110] facet plane and increased absorption along the visible region of AP-1.0 photocatalyst. Therefore, this result could be a consideration for the improvement of Ag_3PO_4 photocatalyst.

Keywords: Ag_3PO_4, co-precipitation method, photocatalytic activity, Rhodamine B.

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1. INTRODUCTION

To date, silver orthophosphate or Ag_3PO_4 photocatalyst has been promising visible light-activated photocatalyst material. This because of their small bandgap energy (~2.40 eV) and their quantum efficiency of nearly ~90% under visible light or above 520 nm (Yi *et al.*, 2010). In addition, it has greater photocatalytic efficiency than well-known traditional photocatalyst like doped-TiO_2 (Katsumata *et al.*, 2013), TiO_2 commercial (P25) (Liu *et al.*, 2012), and ZnO (Khan *et al.*, 2012).

To enhancing their photocatalytic activities, some modifications have been prepared and investigated. They were doping (Song *et al.*, 2017; Xie and Wang, 2014; Yu *et al.*, 2015), composite fabrication (Chen *et al.*, 2013; Zhankui *et al.*, 2013) and morphology design (Dong *et al.*, 2014; Guo *et al.*, 2015; Sulaeman *et al.*, 2016; Vu *et al.*, 2013). The modifications were also carried out by using the addition of complex solution (Wang *et al.*, 2012; Xu and Zhang, 2013), the hydrophilic polymer (Yang *et al.*, 2014), various reactant composition (Bozetine *et al.*, 2013), stirring time (Yan *et al.*, 2013) and temperature (Wang *et al.*, 2015). These modifications showed a significant effect on the photocatalytic activities of Ag_3PO_4 photocatalyst.

Recently, the different morphology, particle size, crystallinity, and absorption profiles could be resulted in the different concentrations of KH_2PO_4 as starting material in the Ag_3PO_4 synthesis. This might be due to the different concentration influenced the nucleation and crystal growth during photocatalyst synthesis. However, the high photocatalytic activity of Ag_3PO_4 photocatalyst can be found in the sample with smaller particle size, higher crystallinity, and UV-Vis absorption ability of Ag_3PO_4 (Afifah *et al.*, 2019). Besides KH_2PO_4, the reactant of $Na_2HPO_4{\cdot}12H_2O$ as a source of phosphate has been applied for Ag_3PO_4 design synthesis (Chen, Dai, Guo, Bu, & Wang, 2016; Wang et al., 2015; Wu et al., 2013). Nevertheless, the investigation using the variation of

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$Na_2HPO_4 \cdot 12H_2O$ concentration has not been reported. Based on these reports, it is a good idea to obtain high photocatalytic activity by using $Na_2HPO_4 \cdot 12H_2O$. Therefore, we have tried to observe the effect of the varied concentration of $AgNO_3$ and $Na_2HPO_4 \cdot 12H_2O$ as starting material for Ag_3PO_4 co-precipitation. The activities of the products were investigated using the Rhodamine B (RhB) photodecomposition under blue light irradiation.

2. MATERIALS AND METHODS

Materials

Materials were used without further purification, such as a commercial textile dye of RhB, distilled water (DW), $AgNO_3$ (Merck), and $Na_2HPO_4 \cdot 12H_2O$ (Merck).

Photocatalyst Characterization

Determination of crystallinity and purity of samples was done using X-ray diffraction (XRD) Bruker AXS D2 Phaser. UV and visible light absorption profiles were conducted using Diffuse Reflectance Spectroscopy (DRS) JASCO V-670. Photocatalyst samples were examined under blue light irradiation of LED Skyled (3W/220V) for RhB photodegradation. Lamp-suspension surface distance was adjusted at 10 cm. The absorbance of filtrate after the photodegradation process was analyzed using UV-Visible spectroscopy Shimadzu 1800.

Synthesis of Ag_3PO_4 Photocatalyst with The Varied Concentration of Reactants

Photocatalyst was synthesized using the co-precipitation method by reacting $AgNO_3$ and $Na_2HPO_4 \cdot 12H_2O$ with various concentrations of reactants. The concentration of $Na_2HPO_4 \cdot 12H_2O$ was 0.03, 0.17, 0.33, and 0.67 M were slowly mixed drop by drop in room temperature into 0.1, 0.5, 1.0, and 2.0 M of $AgNO_3$, respectively. The reactants of $AgNO_3$ and $Na_2HPO_4 \cdot 12H_2O$ were stoichiometrically synthesized following the reaction as shown in Eq. 1 (molar concentration ratio of Ag^+ to HPO_4^{2-} is 3:1) (Vogel, 1990) and separately dissolved in 25 mL volumetric flask using DW. Samples were stirred, and as-resulted yellow precipitates were collected, washed several times, and dried in the oven at 105 °C for 5-10 hours. Samples were labeled as AP-0.1, AP-0.5, AP-1.0, and AP-2.0, respectively.

$$3Ag^+ + HPO_4^{2-} \rightarrow Ag_3PO_4 + H^+ \quad (1)$$

Photocatalytic Activity Analysis

An amount of 0.2 g of Ag_3PO_4 photocatalyst was poured into 100 mL of 10 mg/L RhB under stirring condition. Initially, the reaction was carried out under the dark condition for 20 minutes to ensure the adsorption-desorption of dye and Ag_3PO_4 photocatalyst. After that, the suspension was irradiated under blue light, and the sample was drawn ±5 mL at a certain time. The suspension was centrifuged at 1.500 rpm, and the absorbance of the filtrate was measured (Febiyanto *et al.*, 2016).

3. RESULTS AND DISCUSSION

XRD Analysis of Ag_3PO_4 Photocatalyst

The XRD profile of as-synthesized Ag_3PO_4 was shown in Fig. 2. The diffraction of AP-0.1; AP-0.5; AP-1.0; and AP-2.0 are similar. As shown in Fig. 1, the photocatalysts synthesized under the varied concentration have a body-centered cubic of Ag_3PO_4. It is suitable with the JCPD #06-0505 (Huang *et al.*, 2019; Yan *et al.*, 2014). High intensities of peaks and no others peak except Ag_3PO_4, suggesting that the samples are highly crystalline and high purity (Ge, 2014).

The crystallite size of samples was calculated using the Debye-Scherrer equation as follow (Bozetine *et al.*, 2013):

$$D = \frac{K\lambda}{\beta cos\theta} \quad (2)$$

where D, K, λ, β, and θ were crystallite size (nm), Scherrer constant (~0.9), a wavelength of X-ray sources (Cu $K\alpha$=0.15406 nm), FWHM (radians), and peak position (radians). Relatively, the same average diameter of crystallite was observed in all samples. Further, the ratio intensity of [110]/[200] was also investigated and summarized in Table 1.

Table 1. Crystallite size and intensity ratio calculation of Ag_3PO_4 photocatalyst samples

Samples	$D_{average}$ (nm)	Intensity ratio of [110]/[200]
AP-0.1	52.02	0.93
AP-0.5	46.86	1.67
AP-1.0	46.96	2.02
AP-2.0	46.16	1.50

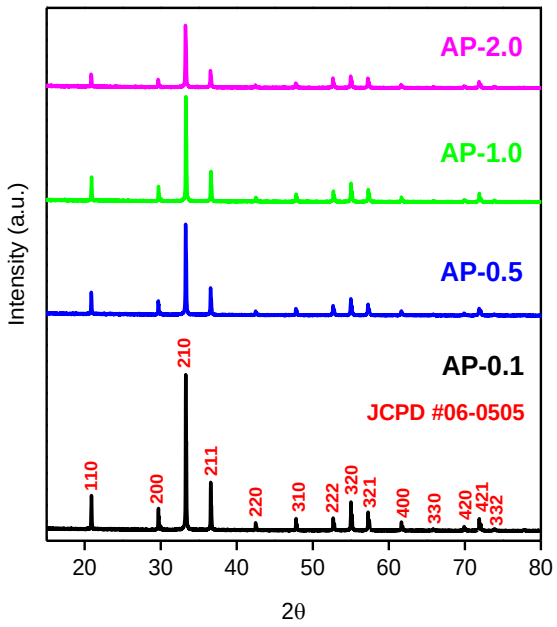

Figure 1. XRD diffraction of samples.

According to Table 1, the intensity ratio of facet planes was affected by the varied molar concentrations of reactants. The highest intensity ratio of [110]/[200] was observed in the sample of AP-1.0, suggesting that their surfaces are dominated by the [110] facet plane. This facet plane is similar to the tetrapod and rhombic dodecahedron morphology types (Martin *et al.*, 2013; Wang *et al.*, 2014). Moreover, the highest [110] dominated-facet surfaces could contribute to the improvement of the photocatalysis activity of Ag_3PO_4 photocatalyst (Bi *et al.*, 2012). Previously, the [110] facet plane of photocatalyst shows a high photocatalytic activity in the dye removal (Wang *et al.*, 2012; Zheng *et al.*, 2013).

Band gap energy analysis of Ag_3PO_4 photocatalyst

Analysis of UV-Vis absorption profiles of as-synthesized Ag_3PO_4 photocatalyst was analyzed using DRS and depicted in Fig. 2 (A-B). Based on Fig. 2 (A), samples have a good-absorption ability on the UV up to visible light (<600 nm). In addition, the difference in absorption ability could also be found in the visible wavelength at around 400-500 nm. The sample of AP-1.0 showed the highest absorption in the visible region (540-800 nm).

Fig. 2 (B) showed the band gap energy profiles of AP-0.1, AP-0.5, AP-1.0, and AP-2.0, respectively. The band energy of samples can be estimated using the formula as follows (Eq. 3) (Rawal *et al.*, 2012):

$$\alpha hv = A(hv - Eg)^{n/2} \quad (3)$$

where α, h, v, A, E_g, and n are absorption coefficient, Planck's constant, light frequency, a constant number, band gap energy, and value depend on the direct ($n=1$) and indirect ($n=4$) characteristics of nature semiconductor transition. According to Eq. 3, the $(\alpha hv)^2$ plot as a function of hv where the band gap energy of each sample was resulted using extrapolation of a straight line towards the axis (x)-direction. The direct band gap energy of samples was summarized in Table. 2. According to Table 2, the varied molar concentrations slightly affect the light absorption as same as the band gap energy of photocatalyst samples.

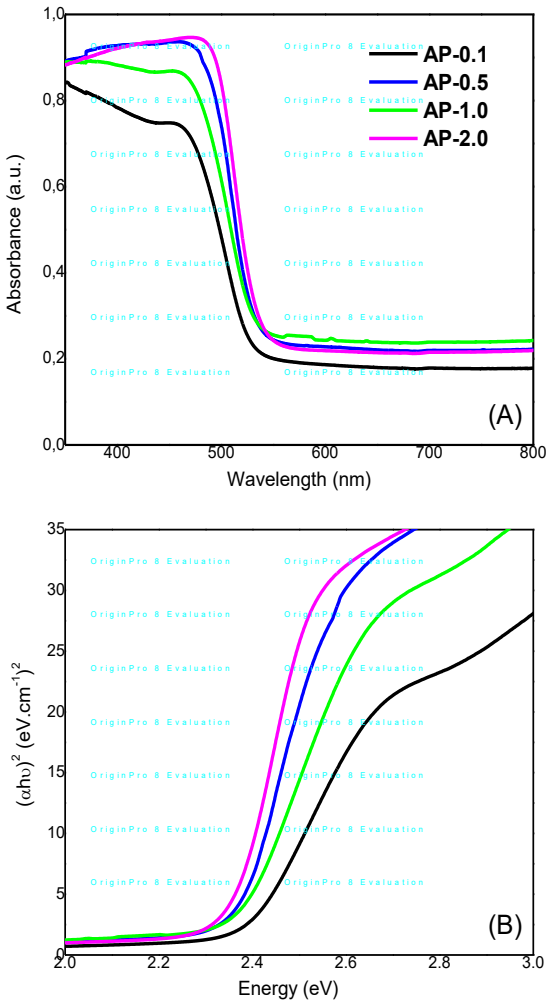

Figure 2. DRS spectra (A) and direct band gap energy characteristics (B) of samples.

Table 2. Sample characteristics of Ag_3PO_4 photocatalyst

Samples	Band gap energy (eV)	Linearity (R^2)
AP-0.1	2.38	0.987
AP-0.5	2.36	0.896
AP-1.0	2.36	0.960
AP-2.0	2.35	0.915

Photocatalytic activity examination of Ag_3PO_4 photocatalyst

Photocatalytic activity of as-synthesized Ag_3PO_4 photocatalyst under the varied molar concentration of reactants was performed in Fig. 3. Based on Fig. 3 (A), the control or RhB photolysis process without the addition of photocatalyst material showed a small decrease in RhB concentration under blue light irradiation. The percent RhB degradation using photolysis reaction over 80 minutes of light exposure was less than 6% (yellow color). The slight decrease in RhB photodegradation without catalyst addition could be explained by Wilhelm and Stephan (Wilhelm and Stephan, 2007) as follows: the excitation of RhB compounds (Eq. 4) is followed by O_2 reduction to be O^{2-}• and photogenerated RhB^{+}• (Eq. 5) under the light irradiation. The O^{2-}• radicals react with H^{+} from H_2O autoprotolysis, and then OOH• was resulted (Eq. 6). Subsequently, RhB cationic was degraded, resulting in CO_2, H_2O, and acid minerals (Eq. 7). Qu and Zhao reported that these reaction mechanisms are a very slow reaction, and the photocatalysis reaction using a catalyst is the best reason to explain a significant decrease in RhB photodecomposition (Qu and Zhao, 1998).

$$RhB + h\nu \rightarrow RhB^* \quad (4)$$
$$RhB^* + O_2 \rightarrow RhB^{+}\bullet + O_2^{-}\bullet \quad (5)$$
$$O_2^{-}\bullet + H^{+} \rightarrow OOH\bullet \quad (6)$$
$$RhB^{+}\bullet + O_2 \rightarrow \text{products} \quad (7)$$

In addition, the same trends were also observed in the adsorption ability of photocatalysts under the dark condition, with a percent decrease of less than 10%, as shown in Fig. 3 (A) (green color). It concluded that RhB photodecomposition under blue light irradiation or photolysis and adsorption process at least could be neglected since there is no apparent concentration changing compared to the RhB photodecomposition with the addition of Ag_3PO_4 photocatalyst. However, the dye photodegradation using radical species that is resulted from light-activated photocatalyst was a plausible mechanism to decrease the absorbance or dye concentration than photolysis reaction (Febiyanto *et al.*, 2019).

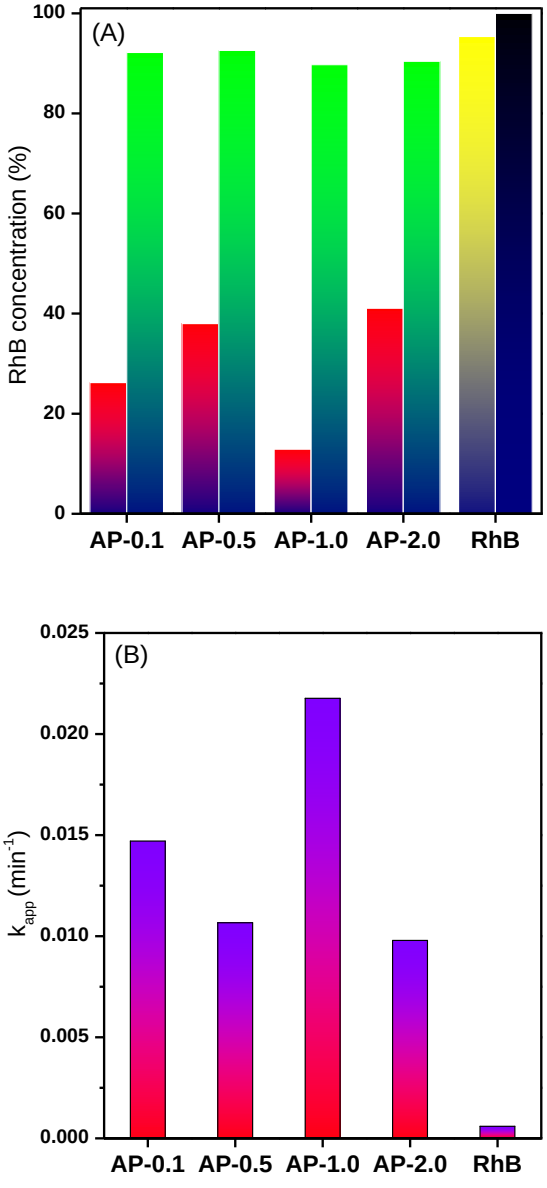

Figure 3. Photocatalytic activities of RhB over 80 minutes of light exposure under blue light (red color) and adsorption ability (green color) over 20 min in the dark condition (A), and their pseudo-first-order rate constant (B). Black and yellow colors (A) are percent initial concentration and photolysis reaction of RhB, respectively.

A significant decrease in RhB concentration was found when the RhB sample was mixed with Ag_3PO_4 photocatalyst, as shown in Fig. 3 (A). Fig. 3 (A) (red color) showed that all samples could effectively degrade the RhB dyes. The high photocatalytic activity can be found in the AP-1.0 sample, with the percent degradation of 82.5%. The high photocatalytic activity might be induced by the highest intensity of [110] facet plane and different properties obtained by co-precipitation under the starting material of AP-1.0 photocatalyst. This preparation generates high absorption along the visible region, as shown in Fig. 2 (A). The AP-1.0 is the high absorption in the visible region. This phenomenon increases the photogenerated electron and holes that lead to improving the photocatalytic activity. The percent degradation of 69.2, 57.4, and 54.3% could be observed in AP-0.1, AP-0.5, and AP-2.0, respectively. These photocatalytic activities were relatively similar and highly different from the AP-1.0 sample.

Photocatalytic kinetic analysis of Ag_3PO_4 photocatalyst with varied molar concentrations was performed in Fig. 3 (B). Rate constant can be determined from a slope that is resulted from linear regression. These can be determined using a formula as follows (Eq. 8):

$$\ln(C_o/C) = kt \quad (8)$$

where C_o, C, t, and k are initial concentration, the concentration at a certain time, irradiation time, and a rate constant of pseudo-first-order kinetic (Yan *et al.*, 2014). Based on Fig. 3 (B) and Table 2, the rate constant of samples shows a good-linearity and following the pseudo-first-order rate kinetic (Pradhan *et al.*, 2014). However, the sample with AP-1.0 showed a high photocatalytic rate kinetic of 0.0218 min^{-1}, whereas samples of AP-0.1, AP-0.5, and AP-2.0 were 0.0147, 0.0107, and 0.0098 min^{-1}, respectively. The results showed that varying molar concentrations affect the rate constant, as same as photocatalytic activities of Ag_3PO_4 photocatalyst.

The RhB oxidation and reduction mechanism chain of photocatalyst generally might be happened on the photocatalyst surfaces of Ag_3PO_4 under visible light irradiation. This following the photodegradation mechanisms below *(Guo et al.*, 2015):

$$Ag_3PO_4 + h\nu \rightarrow Ag_3PO_4 (e^-_{CB} + h^+_{VB}) \quad (9)$$
$$Ag_3PO_4(h^+_{VB}) + H_2O \rightarrow Ag_3PO_4 + H^+ + \bullet OH \quad (10)$$
$$Ag_3PO_4(h^+_{VB}) + OH^- \rightarrow Ag_3PO_4 + \bullet OH \quad (11)$$
$$Ag_3PO_4(e^-_{CB}) + O_2 \rightarrow Ag_3PO_4 + \bullet O_2^- \quad (12)$$
$$\text{Organic compounds} + \bullet O_2^-/\bullet OH \rightarrow CO_2 \text{ and } H_2O \text{ (acid minerals)} \quad (13)$$

Photocatalyst under blue light excites electron (e^-) from valence band (VB) to the conduction band (CB) resulted in electron (e^-_{CB}) and hole (h^+_{VB}) (Eq. 9). The electron-hole pairs transferred to the Ag_3PO_4 surfaces and oxidation/reduction of water molecules (H_2O) (Eq. 10) and OH^- ions or dissolved oxygen (O_2) (Eq. 11-12) can take place. Subsequently, this can produce a radical species of hydroxyl (•OH) and superoxide ($\bullet O_2^-$). The benefits of this chemical radical decompose the RhB dye structures to be small molecules such as H_2O, CO_2, and acid minerals (Eq. 13).

4. CONCLUSION

The photocatalyst of Ag_3PO_4 under the varied concentration of starting materials have been successfully synthesized using the co-precipitation method. The results showed that the varied concentrations of starting materials affect the photocatalytic activities, the intensity ratio of [110]/[200] facet plane, and their bandgap energies of Ag_3PO_4 photocatalyst. Photocatalyst synthesized using 1.0 M of $AgNO_3$, and 0.33 M of $Na_2HPO_4 \cdot 12H_2O$ (AP-1.0) showed the highest photocatalytic activity with the percent photodegradation of 82.5%. This is due to the high [110] facet plane and the absorption along a visible region of AP-1.0 photocatalyst. However, the [110] dominated-facet plane which is similar to the tetrapod or dodecahedron morphology structure could contribute to the improvement of photocatalysis reaction of Ag_3PO_4 photocatalyst in the RhB photodegradation.

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