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Research Article

Facile Synthesis of Ag_3PO_4 Photocatalyst with Varied Ammonia Concentration and Its Photocatalytic Activities For Dye Removal

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Abstract

The highly active photocatalyst of Ag_3PO_4 could be synthesized under ammonia solution using the facile co-precipitation method with the starting material of $AgNO_3$ and $Na_2HPO_4.12H_2O$. The variation of ammonia concentration was designed at 0.00, 0.05, 0.15, and 0.30 M. The products were characterized using X-ray diffraction, UV-diffuse reflectance spectroscopy, and scanning electron microscopy. The photocatalytic activities were evaluated using the Rhodamine B degradation under blue light irradiation. The effect of calcination, pH condition, and visible light source irradiation was carried out in the experiment. The highest photocatalytic activity was found in the sample prepared using the addition of ammonia solution at the concentration of 0.05 M. This photocatalytic activity was 4.13 times higher compared to the Ag_3PO_4 prepared without the ammonia. The effective condition of photocatalytic activity was achieved at the sample prepared without calcination, degradation at pH of 7 and under blue light irradiation.

Keywords: Ag_3PO_4 photocatalyst; photocatalytic activity; Rhodamine B; varied ammonia concentration

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1. Introduction

Recently, the silver orthophosphate or Ag_3PO_4 semiconductor has been attracting the attention of many researchers because of its promising catalyst properties which have higher photo-oxidation ability for organic pollutants or dye removal. Silver orthophosphate has the band gap energy of 2.36 eV, that can absorb solar energy shorter than ~530 nm [1-6], possess stronger photo-oxidative capability than the previous photocatalyst [7-9], generating O_2 from water splitting with a quantum efficiency nearly 90% in visible light irradiation [10]. In order to improve its photocatalytic activity in the visible light region, many approaches have been performed especially in catalyst fabrication design

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for a large-scale application. Among them, the synthesis of Ag_3PO_4 assisted by ammonia solution [11-12] was reported. The use of ammonia solution as the additional component in the synthesis produces the varied-morphology of Ag_3PO_4, such as: the cubic-like structure, that exhibits a good photocatalytic activity than the irregular structure. Yan and co-workers [13] reported that the synthesis of Ag_3PO_4 microcubes can improve their visible light driven photocatalytic degradation for Rhodamine B, methylene blue, and methylene orange. Xingyuan and co-workers [14] also reported that the synthesis of uniform and well defined Ag_3PO_4 microcrystal showed high photocatalytic activity, three times higher compared to the Ag_3PO_4 micro-particle.

Based on the above-mentioned information, the synthesis of Ag_3PO_4 assisted by ammonia solution needs more deep investigation. There is no sufficient information of Ag_3PO_4 preparation especially the effect of varied ammonia concentration, the temperature of calcination, pH degrees, and their photocatalytic properties under variation of visible light lamps. These samples were evaluated using the decomposition of Rhodamine B under visible light irradiation and characterized using the X-ray diffraction, UV-diffuse reflectance spectroscopy, and scanning electron microscopy. Catalyst with the highest photocatalytic activity was also treated with the variation of calcination temperature, pH, and visible light lamps to determine the effect of calcination, acid-base condition and light sources in photocatalytic reaction. In this research, the varied ammonia concentration, calcination treatment, pH and light sources significantly affected the photocatalytic activity. All information about our research has been discussed and compared in detail.

2. Materials and Methods

2.1 Materials

All the reagents are pro analysis and provided from Merck Darmstadt, F.R. Germany. Silver nitrate ($AgNO_3$), disodium hydrogen phosphate 12-hydrate ($Na_2HPO_4.12H_2O$), and 25% ammonia were used without further purification. The LED lamp of blue, red, yellow, and green light (3W/220V) were used as the visible light source irradiation.

2.2 Preparation of Catalyst

The solutions of $AgNO_3$, $Na_2HPO_4.12H_2O$, and 25% ammonia are prepared under deionized water separately as solution-1, 2, and 3, respectively. Solution-1 was prepared by dissolving the 2.5485 g of $AgNO_3$ to 50 mL of deionized water and solution-2 was prepared by adding the 1.7895 g of $Na_2HPO_4.12H_2O$ to 50 mL of deionized water. These solutions were created under stirring to dissolve the precursors quickly. Solution-3 was created by adding the 25% of ammonia solution to the deionized water with the variations of ammonia concentration at 0.00 M, 0.05 M, 0.15 M, and 0.30 M in 100 mL of solution. The photocatalyst was synthesized by dripping solution-3 to the solution-1 under stirring until the color of the solution changed into blackish-brown due to the formation of Ag complexes with ammonia. Then, the solution-2 was added drop by drop to this mixture solution until the solution changed to a yellow-light suspension. The yellow-light precipitates were filtered and washed 3 times with deionized water. The resulting yellow-light precipitate was dried in an oven for 7 hours at 105 °C. The samples, with the concentration of 0.00 M, 0.05 M, 0.15 M, and 0.30 M, were named as Ag_3PO_4, Ag_3PO_4-0.05, Ag_3PO_4-0.15, and Ag_3PO_4-0.30, respectively. To know the effect of calcination temperatures, the catalyst with the highest photocatalytic activity was calcined at various temperatures of 300, 400, 500, 600, and 700 °C for 5 hours.

Chemical oxygen demand (COD) of photocatalytic activity results was evaluated using simple technique by APHA [15] with modification, typically a 10 mL of deionized water as control and Rhodamine B solution (before and after photocatalysis treatment) separately poured into 100 mL Erlenmeyer and then mixed with 0.1 g of $HgCl_2$ and 2 mL of $KMnO_4$ 0.01 M, respectively. Control and Rhodamine B samples were heated under hotplate stirrer until boiled up for 5-10 minutes, then cooled until room temperature condition for a while. The aqueous solution of 2 mL of H_2SO_4 4 N and 1 mL of KI 10% were poured into control and Rhodamine B samples, respectively. Subsequently, they were titrated using $Na_2S_2O_3$ 0.05 N aqueous solution and it can be stopped for a minute when the solution turned into a yellow light and thus added some droplets of amylum (solution then changed to be a blue dark color). The titration could be continued until the blue dark color of the solution was disappeared exactly. Noted that all samples were repeated by duplet examination. The concentration of samples can be calculated by the formula as follows: $COD\ dose = ((A–B) \times N \times 8000)/S$; where A and B are volume (mL) of $Na_2S_2O_3$ for control (deionized water) and sample (Rhodamine B before and after photocatalysis treatment), re-

spectively; N is the concentration of $Na_2S_2O_3$ (0.05 N) and S is the volume (mL) sample which is used in this experiment (10 mL).

2.3 Characterization

The prepared samples were characterized by Scanning Electron Microscopy (SEM), UV-Vis Diffuse Reflectance (DRS), and X-ray Diffraction (XRD).

2.4 Photocatalytic Activity Test

Typically, an amount of 0.1 gram catalysts mixed with 80 mL of 10 mg/L Rhodamine B under blue light irradiation. The distance between the surface of the suspension and lamp was set at 20 cm. The photodecomposition reaction initially set in the dark condition for 10 minutes to ensure the adsorption-desorption equilibrium between catalyst and dyes in suspension. When the lamp was turned on, the sample was drawn every 1 minute until a certain time for absorbance measurement. Afterward, the sample was centrifuged at 2000 rpm for 30 minutes and the concentration of filtrate was measured by UV-visible spectroscopy. The sample of highest photocatalytic activity was also evaluated under the red, green and yellow of LED lamps.

3. Results and Discussion

Figure 1(a) and (b) showed the X-ray diffraction and UV-Vis diffuse reflectance spectra of Ag_3PO_4, Ag_3PO_4-0.05, Ag_3PO_4-0.15, and Ag_3PO_4-0.30 photocatalysts, respectively. Based on Figure 1 (a), the diffraction peaks of all samples could be identified as body-centered cubic structure (JCPDS no. 06-0505) [16], with small impurity indicating that the Ag_3PO_4 photocatalysts with or without the addition of 25% ammonia solution were successfully synthesized by facile co-precipitation method. Moreover, the sharp peaks could be distinctly observed on the samples with the addition of 25% ammonia solution, indicating that the ammonia-treated photocatalyst has a highly crystallized structure compared to the pristine Ag_3PO_4 [18].

The diffuse reflectance spectra were shown in Figure 1 (b). The edge absorption of ~530 nm was observed in all samples, indicating that the photocatalyst has a good photocatalytic ability in visible light irradiation. The addition of ammonia in the synthesis of Ag_3PO_4 decreased the absorbance. These decreased absorptions were attributed to the differences in morphology or particle size of catalyst [2,19]. Xingyuan *et al* [14] explained that the decrease in particle size of photocatalyst could shift absorption to the lower wavelength as well as increased its band gap energy. Conversely, a higher particle size can absorb the light of the higher wavelength and then decrease their band gap energy of photocatalyst. Using the calculation of direct transition, the band gap energy of 2.39, 2.41, 2.46, 2.45 eV were found at the samples of Ag_3PO_4, Ag_3PO_4-0.05, Ag_3PO_4-0.15, and Ag_3PO_4-0.30, respectively (Table 1). It was also similar to the previous re-

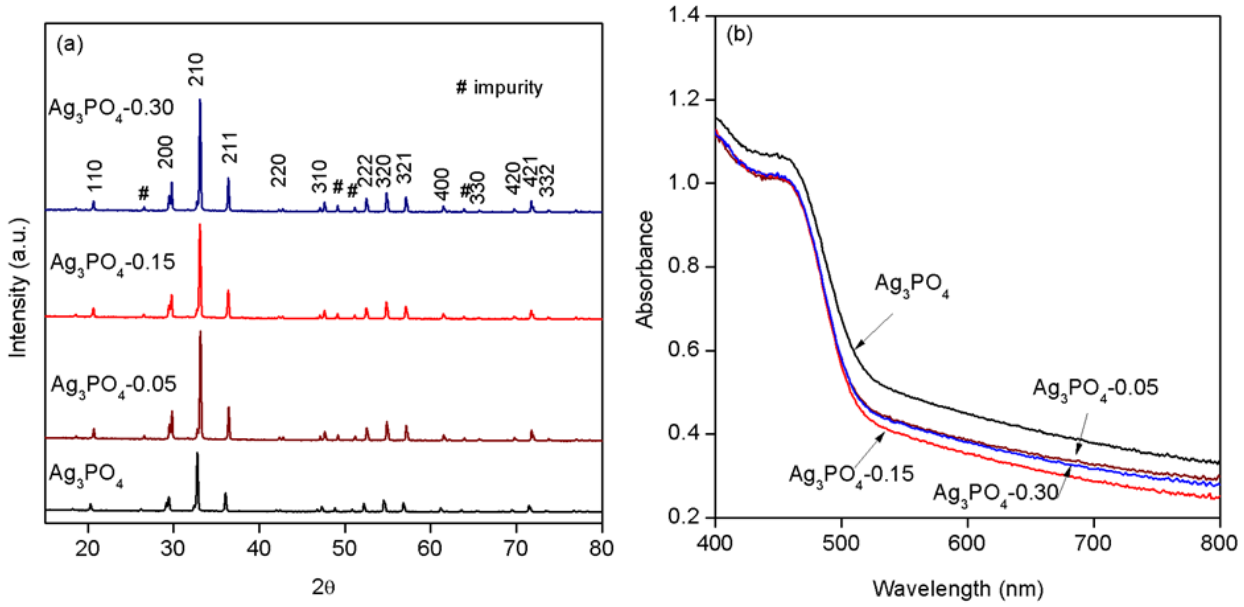

Figure 1. XRD (a) and DRS (b) spectra of Ag_3PO_4 and Ag_3PO_4 photocatalyst assisted by various ammonia concentration

ports [20-21]. Therefore, these the band gap energies might not be the main reason for the enhanced photocatalytic activity.

The morphology of Ag_3PO_4 and Ag_3PO_4-0.05 can be seen in Figure 2. Sample without the addition of 25% ammonia solution exhibited a mixed morphology of spherical and irregular (Figure 2 (b)) with the particle size of ~0.5–2 µm. In contrast, the addition of 25% ammonia in the synthesis of Ag_3PO_4 has successfully decreased the particle size to be ~0.2–1 µm ((Figure 2 (d)). The agglomeration of the particle also could be decreased and the particles were more uniform compared to the pristine Ag_3PO_4. The decreased agglomeration and enhanced homogeneity of particles might be caused by a formation of silver-amino ($[Ag(NH_3)_2]^+$) complex. It was also similar to another report [11]. The $[Ag(NH_3)_2]^+$ complex was created by the interaction of Ag^+ and ammonia in the solution which was favorable to decrease the reaction rate and thus easily produced more well-structured particle or hindered the agglomeration. Moreover, these properties could increase the surface area and enhance the photocatalytic activity of the catalyst [22-25]. The high surface area could provide the more active site for photodecomposition of Rhodamine B.

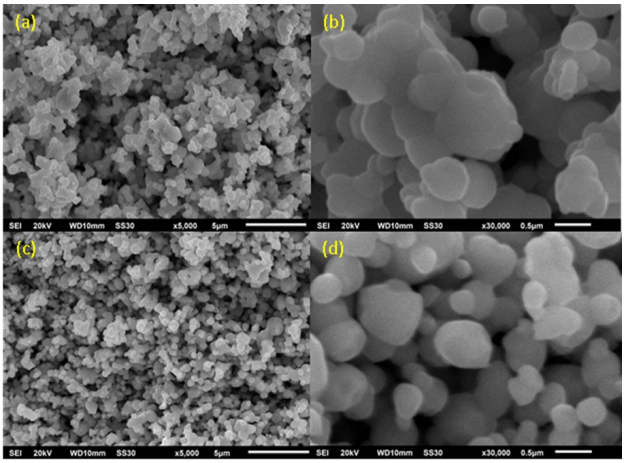

Figure 2. SEM images of Ag_3PO_4 (a), Ag_3PO_4-0.05 (c) and their magnifications of Ag_3PO_4 (b) and Ag_3PO_4-0.05 (d)

Figure 3 (a) shows the photocatalytic activity of photocatalyst with and without the addition of a 25% ammonia solution. All samples have a good visible light driven-photocatalytic activity. The highest photocatalytic activity could be found in the sample of Ag_3PO_4-0.05, indicating that the addition of ammonia significantly affected the photocatalytic activity. However, the increase of ammonia concentration decreases the photocatalytic activities. A high photocatalytic activity of Ag_3PO_4-0.05 photocatalyst is attributed to the smaller particle size and more uniform distribution that was obtained at the addition of 0.05 M ammonia solution.

The rate constant calculation in Rhodamine B photodegradation under blue light irradiation was also investigated using the pseudo-first-order kinetics model [26-27], as follows the formula: $\ln (C_0/C) = kt$; where k is the rate

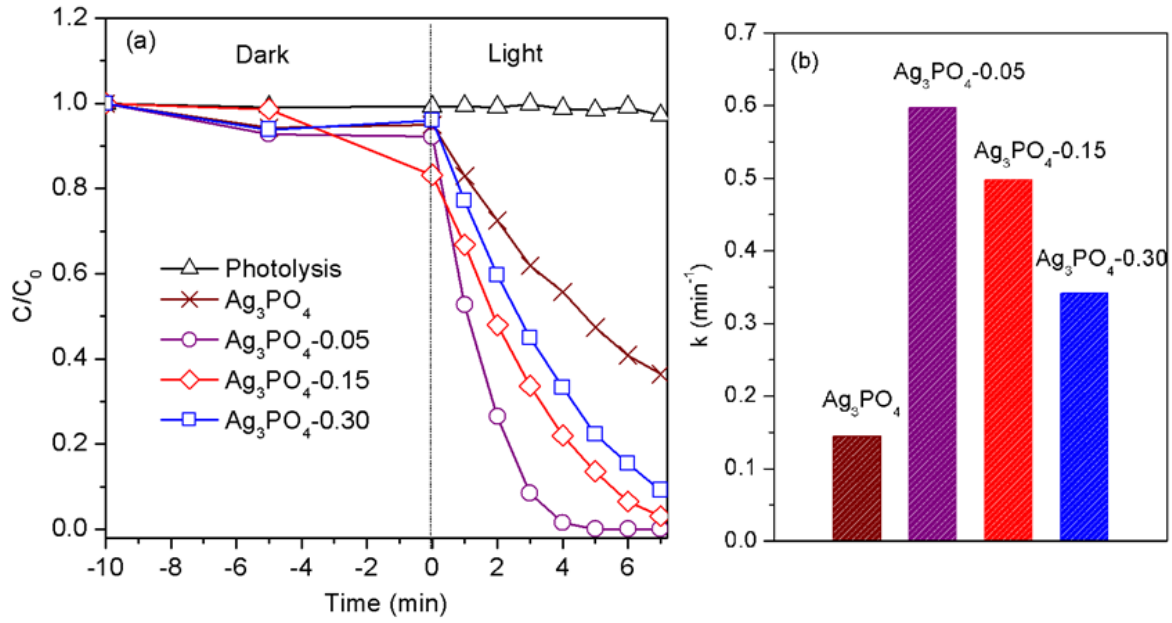

Figure 3. Photocatalytic activity (a) and rate constant (b) of Ag_3PO_4 and Ag_3PO_4 photocatalyst assisted by various ammonia concentration

constant, t is reaction time, C_0 is initial concentration, and C is the concentration of dye in reaction time. According to the Figure 3(b) and Table 1, the photocatalytic reaction of Rhodamine B followed the pseudo-first-order-kinetics model. The rate constant of Ag_3PO_4, Ag_3PO_4-0.05, Ag_3PO_4-0.15, and Ag_3PO_4-0.30 photocatalyst are 0.144, 0.597, 0.498, and 0.342 min^{-1}, respectively. The rate constant of Ag_3PO_4-0.05 photocatalyst shows 4.13 times higher than the Ag_3PO_4 photocatalyst.

The highest photocatalyst (Ag_3PO_4-0.05) was calcined at the various temperature and the results were shown in Figure 4(a). It showed that the calcination temperature had significant effects on photodegradation activities. The calcination at 300 °C and 400 °C showed that their photocatalytic activities were similar with the sample without calcination. The percent degradations of 98.71%, 95.80%, and 99.24% could be found in the sample calcined at 300 °C, 400 °C, and without calcination, respectively. The calcination at the higher temperature of 500 °C, 600 °C, and 700 °C showed the percent degradation of 49.94%, 7.93%, and 5.20%, respectively. The increase of temperature calcination more than 400 °C significantly decreased their photocatalytic activity. Some previous works reported that the calcination can influence the photocatalytic activity [28-29]. The calcination could also affect the active site, specific surface area, pore size

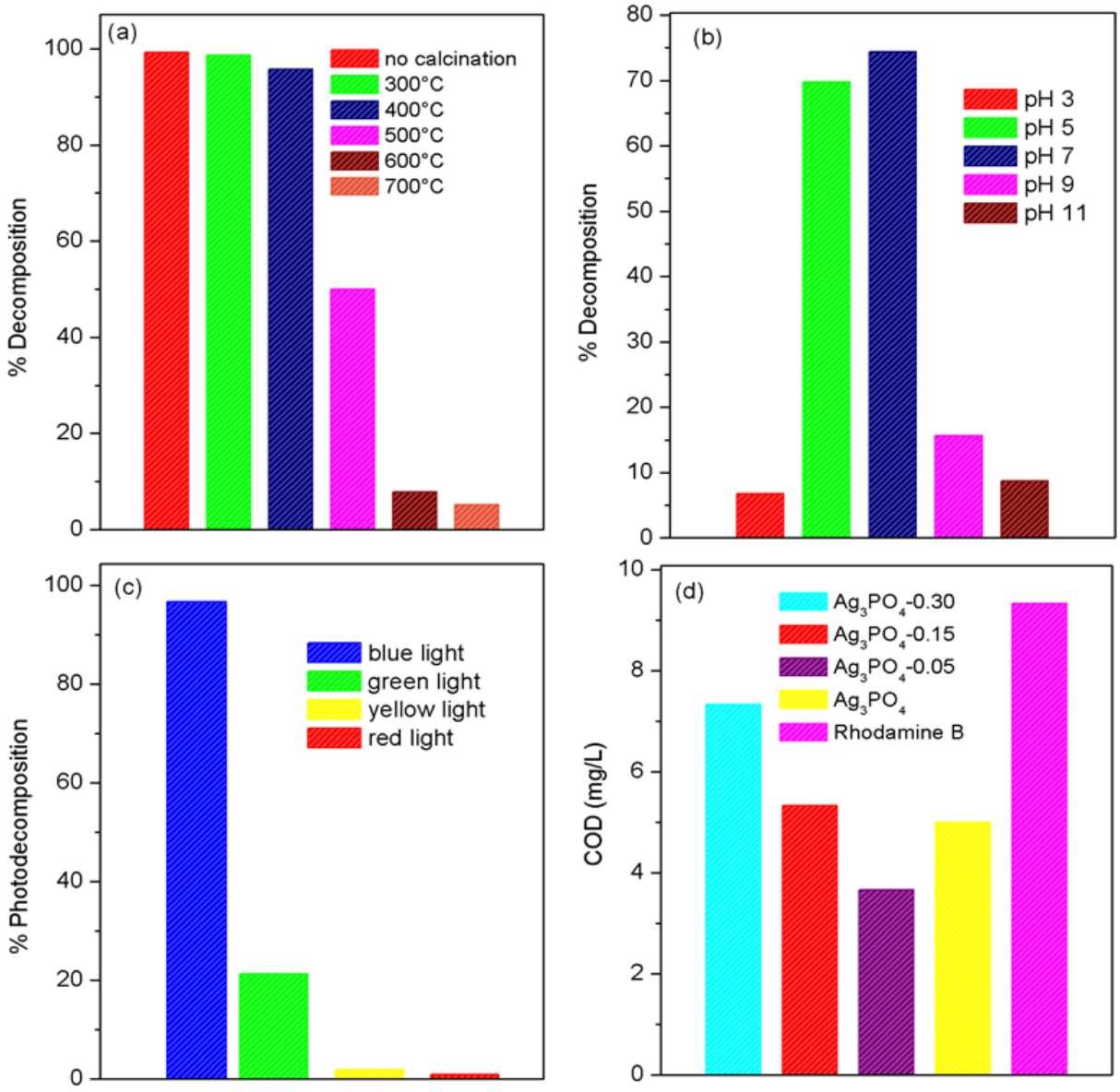

Figure 4. The effect of calcination temperature (a), pH condition (b), light sources (c) and COD test (d) of the photocatalyst in Rhodamine B photodegradation.

and pore volume of photocatalyst [30-32]. Dong and his co-workers [17] reported that the appropriate calcination treatment was beneficial to enhance the oxygen vacancies and create a defect on the Ag_3PO_4 surfaces. These merits could promote the charge transfer and then increase the photocatalytic activities of the photocatalyst. Sulaeman and co-workers [33] also explained that the increase in calcination temperature increases the photocatalytic activity as well as the crystallinity of catalyst. However, calcination temperature in higher temperature could decrease the activity due to decreasing the active site or surface area of the catalyst.

The effect of pH on the photocatalytic reaction was also investigated and the results were shown in Figure 4(b). The higher photocatalytic activity could be observed in the neutral solution (pH=7) with the percent degradation of 74.39%, whereas the pH of 3, 5, 9, and 11 have the percent degradations of 6.79%, 69.77%, 15.66%, and 8.74%, respectively. In pH of 7, the surface of Ag_3PO_4 has negative charge [34]. This surface can effectively attract the Rhodamine B dyes which have positive charges [34-35]. The adsorbed dyes onto Ag_3PO_4 surfaces subsequently can be degraded directly under blue light irradiation.

The acid condition of the appropriate H^+ ions (pH=5) enhanced the catalysis reaction through photogenerated OH• radicals that was generated by H_2O_2 reacted with electron [36-37], but in the lowest pH condition (pH=3), the photocatalytic activity of catalyst could be poor. These could be explained by two plausible reasons: (1) catalyst like silver orthophosphate can be degraded in the lowest or extreme pH degrees (pH < 5). In the extreme acid situation, the Ag_3PO_4 can be oxidized into Ag^+ ions which dissolved clearly in Rhodamine B solution and subsequently decreasing their photocatalytic activity. In our experiment, the Ag_3PO_4-0.05 photocatalyst (without calcination) disappeared easily when the reaction time either less than or reached to ~17 minutes of reaction time in pH 3, or (2) the low photocatalytic activity of catalyst could be caused by a successive H^+ ions in suspension where anchored along the Ag_3PO_4 surfaces thus creates a positive charge on the Ag_3PO_4 surfaces. These can suppress the dyes adsorption due to the repulsion of catalyst and dyes [38]. Conversely, although in the appropriate base condition where OH^- ions are able to produce the photo-generated OH• radicals through hole and electron reaction mechanism [39-40], in the higher or extreme base condition, the photocatalytic activity decreases due to the reduction of Ag^+ ions to Ag^0 [23]. It could be considered that the optimum pH was achieved at a neutral condition or a pH of 7. The pH treatment (acid or base condition) decreases the photocatalytic activity due to disturbing the interaction of dye and the surface of the catalyst.

The effect of visible light sources was presented in Figure 4(c). Based on Figure 4(c), the photodegradation ability of Ag_3PO_4-0.05 photocatalyst under visible light sources were subsequently determined by light exposure test of red, yellow and green light of LED lamps. Compared to the other visible-light sources, the Ag_3PO_4-0.05 photocatalyst has higher photocatalytic activity under blue light irradiation with the percentage of Rhodamine B photodegradation was 96.67% whereas the visible-light sources of red, yellow and green were 0.93%, 1.92%, and 21.27%, respectively. It can be concluded that the optimum photocatalytic activity of Ag_3PO_4-0.05 photocatalyst is achieved under blue light irradiation (λ = ~445 nm). It is because that the blue light has a lower wavelength with the energy of ~2.79 eV. This energy could promote the excitation of the electron in the valence band (VB) to the conduction band (CB) of Ag_3PO_4 photocatalyst [33].

The COD (chemical oxygen demand) investigation was performed to find out the demineralization ability of catalysts (Figure 4(d)). Based on the experiment, it was found that

Table 1. Summary data of pure Ag_3PO_4 and Ag_3PO_4 photocatalyst assisted by various ammonia concentration

Photocatalysts	Band gap energy (eV)	K_{app} (min^{-1})	R^2
Ag_3PO_4	2.39	0.1442	0.9951
Ag_3PO_4–0.05	2.41	0.5972	0.9384
Ag_3PO_4–0.15	2.46	0.4980	0.9649
Ag_3PO_4–0.30	2.45	0.3416	0.9791

Ag_3PO_4-0.05 photocatalyst showed the highest demineralization activity than those of all samples. The COD value of 9.33, 5.00, 3.66, 5.33 and 7.33 mg/L was observed at the samples of before photodecomposition, after photodecomposition using the Ag_3PO_4, Ag_3PO_4-0.05, Ag_3PO_4-0.15, and Ag_3PO_4-0.30, respectively. It was suggested that Rhodamine B was not only decolorized but also degraded by photocatalyst. However, the results ascribed that all catalysts could not completely degrade the dyes into small molecules such as CO_2, H_2O or non-toxic minerals due to the complexity of cyclohexane or bulky structure of Rhodamine B. We had supposed that the left complex compound of Rhodamine B could be completely degraded by prolonging reaction time. Wilhelm and Stephan [41] had also assumed that the complete demineralization of Rhodamine B needs a longer reaction time.

4. Conclusions

The addition of 25% ammonia in the synthesis of Ag_3PO_4 could affect the photocatalytic activity. The highest photocatalytic activity could be achieved at the addition of 0.5 M ammonia solution. This treatment could successfully decrease the particle size and increase the homogeneity of catalyst that improves the photocatalytic activity up to 4.13 times higher compared to the Ag_3PO_4 synthesized without ammonia solution. The excellent condition of photocatalytic activity was achieved at the sample prepared without calcination, degradation at pH of 7 and under blue light irradiation.

Acknowledgments

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