

Effect of H₂O₂ Addition on the Photocatalyst Properties of Ag₃PO₄ for Methylene Blue Photodegradation

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Abstract

In this work, a facile enhanced property of Ag₃PO₄ photocatalyst using H₂O₂ for methylene blue photodegradation has been conducted. Ag₃PO₄ photocatalyst was synthesized using the coprecipitation method. Then, the photocatalytic activity and stability of Ag₃PO₄ were tested in the methylene blue degradation under the addition of an H₂O₂ 30% solution. According to the results, the prolonged photocatalysis reaction led to a decrease in the catalysis ability of the Ag₃PO₄ photocatalyst. Meanwhile, the addition of 5 mL of H₂O₂ (AH-10% sample) tends to enhance the stability of Ag₃PO₄ photocatalyst with the dye removal percentage and recycling test were up to 90.3% and three recycle runs, respectively. The low stability of Ag₃PO₄ might be due to the catalyst photo-corrosion through Ag⁺ reduction. This research suggests that H₂O₂ is beneficial to inhibit the photo-corrosion of Ag₃PO₄ photocatalyst, even though released Ag ions that were caused by the excess addition of H₂O₂ oxidizing agent should be considered.

Keywords: Ag₃PO₄; H₂O₂ compound; methylene blue; photocatalysis; photocatalyst stability

Introduction

Recently, silver phosphate or Ag₃PO₄ has been intensively investigated for degrading organic and inorganic contaminants, and microorganism deactivations. This is caused by its high quantum yield (the amount of reactant consumed to the amount of photon absorbed using photocatalyst) of more than 90%. It can absorb visible light lower than 530 nm with band gap energy of 2.2-2.5 eV, and high photooxidation ability under visible light illumination (Joo, 2006; Yi et al., 2010; Bai et al., 2013; Hewer et al., 2014; Luo et al., 2014; Sulaeman et al., 2016).

Based on the previous literature, we summarize that there are three major studies or explorations in the development of Ag₃PO₄ photocatalyst for dye and wastewater treatments (Chen et al., 2014; Ji et al., 2015; Diao et al., 2019; Chen et al., 2020). First, photocatalysis ability enhancement. Second, material stability. The last is precursor's usage efficiency by considering the use wisely of the costly silver compound during the Ag₃PO₄ synthesis. To fulfill the three studies mentioned above, researchers devote themselves to designing an efficient and effective pathway of Ag₃PO₄ fabrication. It is composite materials, morphology design, defect engineering, support material

combinations, doping strategy, reaction system, and environment adjustments (Ma et al., 2014; Xie and Wang, 2014; Song et al., 2017; Zheng et al., 2019; Febiyanto and Sulaeman, 2020). For example, adding some sacrificial agents such as silver sources (AgNO_3) into suspension could significantly increase the stability and photocatalysis reaction of Ag_3PO_4 as well (Yi et al., 2010). This might be due to the Ag_3PO_4 structure destruction being much lower than that without a sacrificial agent. The structure destruction may be caused by the phase changing through Ag ions alteration to be its metal (Ag^0) species so restricts their wide application in the dye and wastewater concern.

Given the photo-corrosion processes of Ag_3PO_4 photocatalyst; under visible light irradiation, electron (e^-) excites to the conduction band (CB) level and a hole (h^+) can be produced in the valence band (VB) energy level. In the system without a sacrificial agent, the electron can reduce the Ag ions from the Ag_3PO_4 structure to be Ag^0 when exposed to the visible light ($\text{Ag}_3\text{PO}_4 + 3e^- \rightarrow 3\text{Ag}^0 + \text{PO}_4^{3-}$, $E^\circ=0.45$ eV vs. NHE). Prolonging the catalysis reaction, the photo-corrosion proceeded continuously, and then it was given even worse. Thereby, structure destruction and/or phase changing are inevitable. Finally, the low-photocatalysis and stability of Ag_3PO_4 catalyst have resulted.

The addition of sacrificial agents is considered an effective way to enhance the stabilities as well as the photocatalytic performances. Nevertheless, a silver precursor is commonly known as an expensive material in inhibiting the photo-corrosion of Ag_3PO_4 . Therefore, the other sacrificial agents are necessary to explore and research.

Hydrogen peroxide (H_2O_2) is one of the chemical compounds that can be used as an additive solvent. The potential redox (vs. NHE) of H_2O_2 is lower than CB of Ag_3PO_4 and may act as an electron acceptor. Jia et al., (2019) suggested that the photogenerated electron is easier to combine with H_2O_2 than Ag_3PO_4 due to more negative reduction potential of $\bullet\text{OH}/\text{H}_2\text{O}_2$ than that $\text{Ag}/\text{Ag}_3\text{PO}_4$

($\text{H}_2\text{O}_2 + e^- \rightarrow \bullet\text{OH} + \bullet\text{OH}^-$, $E^\circ=0.87$ eV vs. NHE). By this, the excited electron from CB of Ag_3PO_4 can be transferred to H_2O_2 . Subsequently, it can hinder the severe Ag ions reduction well. Here, the photocatalytic and stability properties will be increased.

In the dye wastewater removal studies, a synergistic reaction named Fenton's mechanism could have proceeded through a combination of Fe ions and H_2O_2 in enhancing the photocatalyst performances (Riapanitra et al., 2015; Tama et al., 2020). Wang et al., (2014^a) use of the sacrificial agent of H_2O_2 30% can slow down the oxidative destruction and thus extend the working life of Ag_3PO_4 photocatalyst on the methylene blue degradation. Agbe et al. (2017) synthesized H_2O_2 -rejuvenated Ag_3PO_4 . During synthesis, as-prepared Ag_3PO_4 containing Ag^0 is rejuvenated by an appropriate H_2O_2 and shows a higher photocatalytic activity for methyl orange, methylene blue, and rhodamine B photodegradation than fresh Ag_3PO_4 . Despite them, the use of H_2O_2 in the dye photodegradation using Ag_3PO_4 catalyst directly over various concentration additions on the photocatalytic and photostability properties of Ag_3PO_4 has been not explored, especially on the methylene blue dye. At least, the H_2O_2 is relatively cheap than the well-known electron acceptor of the AgNO_3 precursor. Therefore, in this work, we investigated a direct way for increasing Ag_3PO_4 properties using the H_2O_2 compound. Then, the results were discussed and presented in this paper.

Research Methodology

Materials

Materials that are used were silver nitrate (AgNO_3), disodium hydrogen phosphate dihydrate ($\text{Na}_2\text{HPO}_4 \cdot 2\text{H}_2\text{O}$), hydrogen peroxide (H_2O_2) 30%, methylene blue (MB) dye that was purchased from MERCK, and commercial aquadest. Materials were used without further purification before. A visible light source that is used was LED Philips Daylight 12 W.

Photocatalyst synthesis

Photocatalyst synthesized by using co-precipitation method. Herein, the solutions were divided into two precursors that were solution-A and solution-B. Solution-A: 5 mmol of AgNO₃ was poured into 20 mL of aquadest and stirred until all powders were dissolved. Meanwhile, solution-B consisted of 20 mL of Na₂HPO₄·2H₂O (3 mmol). During stirring, solution-B was poured drop by drop into solution-A until a yellow light precipitate has resulted. Subsequently, the Ag₃PO₄ precipitate was stirred for 20 minutes. The precipitate was rinsed using aquadest to remove unreacted precursors and to adjust the pH at neutral (~7) conditions has been achieved. The as-synthesized sample was dried at 100 °C for 2 h and stored in a sealed bottle containing silica gel to restrict the water and contaminant adsorption from the atmosphere.

Photocatalysis test

Catalyst with 50 mg was dispersed into 50 mL of methylene blue (MB) 10 mg/L. Lamp and suspension were left for 10 minutes in the dark condition to reach an equilibrium state between catalyst and dye molecules. The distance of the suspension surface and lamp was set at 15 cm away. Then, the lamp was switched on and a sample was mixed for 50 minutes. To know the effect of H₂O₂ on the MB photodegradation, the experiment was carried out by adding 0, 1, 3, and 5 mL (0, 2, 6, and 10% (v/v)) of H₂O₂ 30%. After degradation, samples were centrifuged and measured using a UV-Visible spectrophotometer. Samples were labeled as Ag₃PO₄, AH-2%, AH-6%, and AH-10% respectively.

Recycling ability of Ag₃PO₄ photocatalyst

To know the stability of the catalyst, the Ag₃PO₄ photocatalyst was recycled for degrading MB 10 mg/L under identical conditions three times. The photocatalysis system was set at the optimum or maximum condition of H₂O₂ addition.

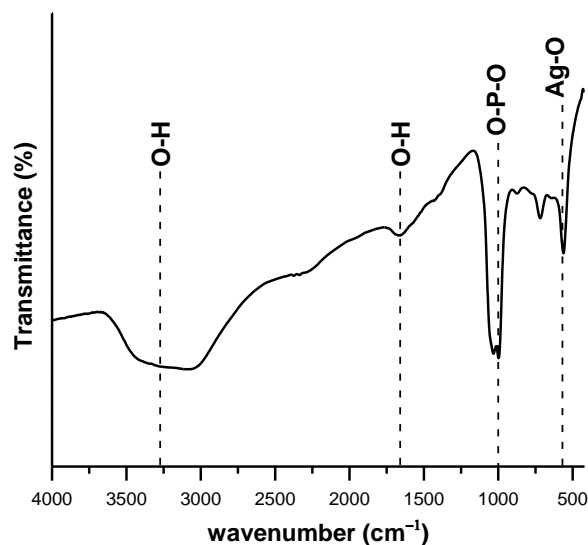


Figure 1. Fourier Transform IR of Ag₃PO₄ sample.

Photocatalyst mechanism

The radical species test on the MB photodegradation can be determined by using radical trappers such as p-benzoquinone (BQ), isopropanol alcohol (IPA), and ammonium oxalate (AO) for superoxide (\bullet O₂), hydroxyl (\bullet OH), and hole (h⁺) radicals (Febiyanto et al., 2016), respectively.

Characterizations

Photocatalyst was characterized using Fourier Transform IR Prestige-21 at a wavenumber range of 400-4000 cm⁻¹. Filtrate after photodegradation was measured using UV Vis Spectrophotometer Genesys 10S.

Results and Discussion

Functional group analysis of Ag₃PO₄ photocatalyst

Functional groups of Ag₃PO₄ were identified using FTIR and shown in Fig. 1. The Ag₃PO₄ characteristics were observed at a wavenumber of 930 and 550 cm⁻¹ that assigned as O-P-O bending vibration and Ag-O group (Qian et al., 2019; Wu et al., 2013; Piccirillo et al., 2015), respectively. Moreover, some previous reports confirmed O=P-O bending and P-O stretching vibrations could be found near 550 cm⁻¹ (Chai et al., 2014; Anwer and Park, 2019). The stretching and

bending vibrations of the O–H group may strictly be found at a broad spectrum of 3400 and 1650 cm^{-1} , respectively. Further, nitrogen impurity was not detected at 1384 cm^{-1} (Ma et al., 2014), indicating that the Ag_3PO_4 was free of unreacted precursors and successfully synthesized using the facile co-precipitation method.

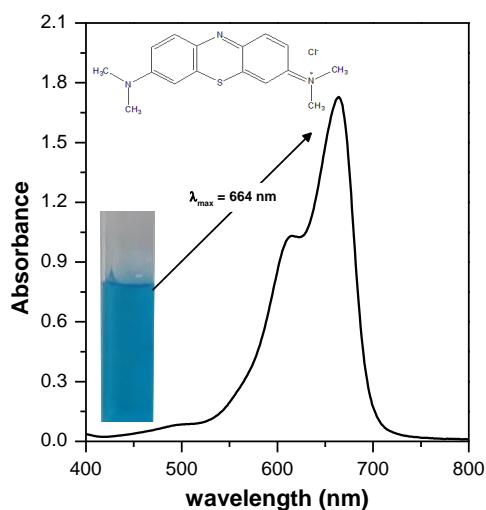


Figure 2. Wavelength scanning of MB maximum absorption. The inset picture was MB structure and solution.

Methylene blue (MB) characterization

Methylene blue (MB) dye was characterized by the maximum wavelength absorption (λ_{max}) over the visible region of 400 up to 800 nm. The λ_{max} was presented in Fig. 2. It was concluded that MB has λ_{max} at around 664 nm. Besides, the pH of the tested solution was 6 similar to previous research (Zhang et al., 2020).

Photocatalysis experiment of Ag_3PO_4 photocatalyst

The photodecomposition of MB was presented in Fig. 3 and Table 1. It could be seen from Fig. 3 that the addition of H_2O_2 generally restricts the photocatalysis reaction. In particular, although the activity was lower than pristine Ag_3PO_4 , the addition of 5 mL (AH-10% sample) into the Ag_3PO_4 -MB suspension system shows a higher catalysis activity than the other additions. Here, to know the effect of MB dissolution, photolysis,

and H_2O_2 -catalyzed MB degradation, the system was also tested for MB+ H_2O_2 +light, MB+ H_2O_2 (dissolution effect), and photolysis reaction under identical conditions.

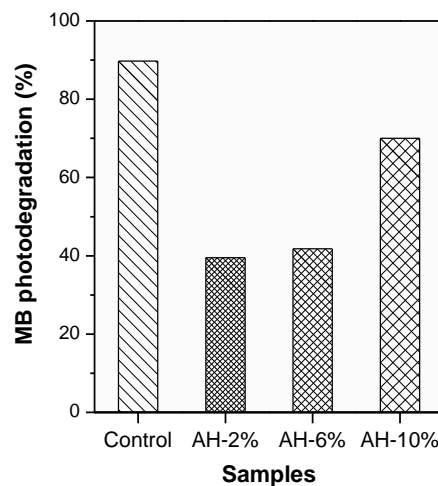


Figure 3. Photocatalytic activities of samples with and without the addition of H_2O_2 compound.

Table 1. Photocatalyst activities of samples

Sample	Degradation (%)
Photolysis	6.6
MB+ H_2O_2 (5 mL) + light	15.6
MB+ H_2O_2 (5 mL)	3.3
Ag_3PO_4	89.7
AH-2%	39.5
AH-6%	41.8
AH-10%	70.0

Based on Table 1, shows that the MB+ H_2O_2 +light system as a percentage of MB photodecomposition was about 15.6%. However, by considering other things, the high of this percentage could be also caused by the photolysis reaction of 6.6% and H_2O_2 -activated catalysis reaction by following the reaction in Eq. 1 (Deng and Zhao, 2015).



The H_2O_2 compound is one of the strong oxidizing agents with a reduction potential value is 1.776 V and is beneficial in degrading the dye pollutants (Setyaningtyas et al., 2019). Further, under visible light irradiation, the H_2O_2 compound can be

broken down into the highest reactive agent of hydroxyl radical ($\bullet\text{OH}$) (Eq. 1) with redox potential 2.720 V (Jiang and Lloyd, 2002; Li et al., 2011). In addition, at least, the dissolution and H₂O₂-activated catalysis effects can be counted by $\sim 9\%$. Thereby, we assumed the dissolution effect caused by the excessive H₂O₂ addition was negligible ($\sim 3\%$) and the catalysis percentage of the AH-10% sample was relatively comparable with single Ag₃PO₄ catalysis activity. Noted that the addition of 10% of H₂O₂ was further used in the following experiments.

Recycling test of Ag₃PO₄ photocatalyst

The stability of Ag₃PO₄ with and without the addition of H₂O₂ for the degradation of MB (10 mg/L) was observed through a recycling test under visible light illumination and the results were shown in Fig. 4 and Table 2. After four times running, the photocatalytic ability of the Ag₃PO₄ catalyst was significantly reduced from 89.7% to 29.6%, confirming the poor catalyst stability of single Ag₃PO₄ or without H₂O₂ addition. In contrast, the catalysis percentage of 50 up to 90% could be achieved by using the AH-10% sample, indicating an excellent stability performance of the sample. However, the slight reduction of catalysis ability on the 2nd recycle runs of AH-10% sample might be caused by MB molecule deposition on the catalyst's surface so which inhibits the dye and photocatalyst interactions. This caused a little photocatalysis reduction in their catalysis performance.

The stability test of the Ag₃PO₄ and AH-10% system was visually displayed in Fig. 5. For 1st run, Ag₃PO₄ powder becomes grey or dark attributing to the silver metallic generation as compared to yellow powder of the AH-10% system. Ag⁰ can be observed by the visual appearances of samples and was indicated through catalyst's color from yellow to grey or dark brown (Khan et al., 2012; Trench et al., 2018), as shown in Fig. 5 (above). Moreover, the Ag₃PO₄ sample became even darker for long recycling up to the 4th run. In contrast, the AH-10% remained stable with the visual appearance of yellow

powder. Interestingly, the quantity of powder is relatively low than the used Ag₃PO₄ (3rd and 4th runs). An excess of H₂O₂ may slightly increase the stability of Ag₃PO₄, through the electron acceptor mechanism of H₂O₂ owing to the more negative reduction potential of $\bullet\text{OH}/\text{H}_2\text{O}_2$ than that Ag/Ag₃PO₄ ($\text{H}_2\text{O}_2 + \text{e}^- \rightarrow \bullet\text{OH} + \bullet\text{OH}^-$, $E^\circ = 0.87 \text{ eV vs. NHE}$). This process caused the yellow light Ag₃PO₄ powder to remain in the MB suspension.

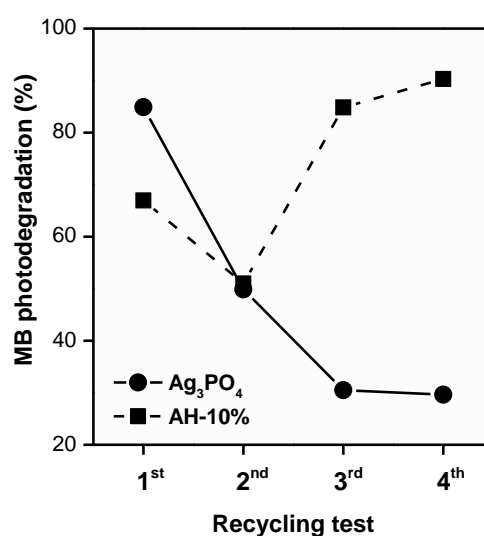


Figure 4. Recycling test of Ag₃PO₄ with and without the addition of H₂O₂ solution.

At the same time, nevertheless, in a long catalysis reaction, the Ag⁰ metallic may be produced higher. In the presence of excess dissolved H₂O₂ and without the precipitate precursor addition like phosphate sources, the photoinduced Ag⁰ will be regenerated into Ag⁺ ions. Nonetheless, those will release into solution and reduce the catalyst quantity of Ag₃PO₄ photocatalyst because of Ag⁺ dissolution. The regeneration mechanism can be followed by the reaction in Eq. 2 (Wang et al., 2014^b). Although the photocatalytic stability was better than single Ag₃PO₄, the photocatalysis reaction was slightly reduced in the 2nd time and increased up to 4th recycling runs sacrificed the powdered Ag₃PO₄ led to dissolve rapidly. The dissolution of Ag₃PO₄ powder is shown in the 4th recycle run of the AH-10% sample. In this research, the AH-10% powder shows a little quantity, whereas the Ag₃PO₄ becomes darker over the long photocatalysis reactions.

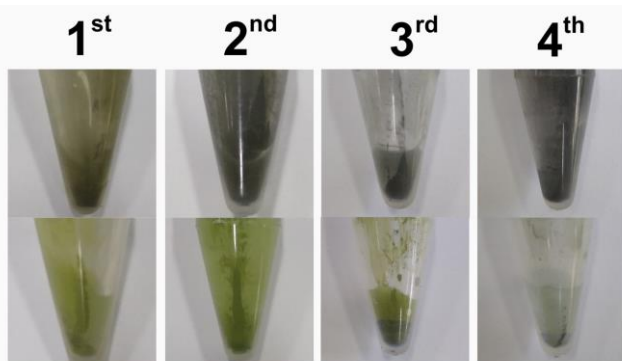


Figure 5. Visual appearances of recycling test for Ag_3PO_4 (above) and AH-10% systems (below), respectively.

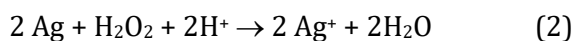


Table 2. Recycle experiment of samples

Recycle	Degradation (%)	
	Ag_3PO_4	AH-10%
1 st	84.9	70.0
2 nd	49.8	51.0
3 rd	30.5	84.2
4 th	29.6	90.3

Role of reactive oxygen radical species

It is well known that the catalysis reaction of photocatalyst-based materials has occurred on their surface's photocatalyst. Additionally, the photogenerated radical species of photocatalyst are useful for degrading the pollutant structures into harmless small molecules and minerals. Therefore, a series of trapping experiments have been carried out to determine the main active radical on the MB photodegradation in more detail. In this study, radical scavenger such as isopropyl alcohol (IPA), ammonium oxalate (AO), and p-benzoquinone (BQ) was employed as hydroxyl ($\bullet\text{OH}$), hole (h^+), and superoxide ($\bullet\text{O}_2^-$) quenchers, respectively. The results were shown in Fig. 6 and Table 3.

Without scavenger additions, Ag_3PO_4 has a photocatalytic activity was 89.7%. With the addition of IPA, the photocatalysis reaction was 92.6% and slightly similar to the Ag_3PO_4 , confirming the $\bullet\text{OH}$ was not the main radical species in the MB photodegradation.

Conversely, the addition of AO and BQ scavenger inhibited the catalyst's ability with the percentage of 51.8 and 44.3%, respectively. This result concluded that the $\bullet\text{O}_2^-$ and h^+ were the main and secondary photogenerated radicals in the MB removal than $\bullet\text{OH}$ species. Moreover, to observe the effect of H_2O_2 in the BQ system as main radical was also done and showed the percentage of 50.4% indicating H_2O_2 addition did not disturb the radical determination.

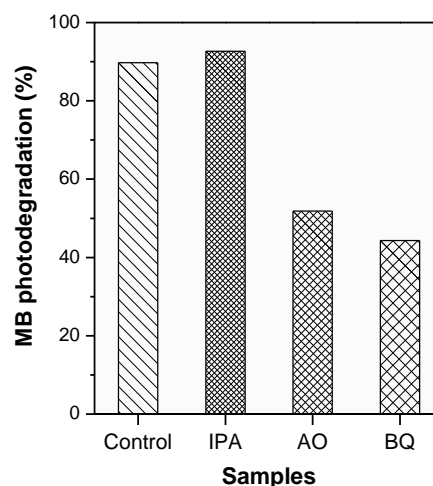


Figure 6. Photocatalytic mechanism of Ag_3PO_4 sample (without H_2O_2 addition).

Table 3. Mechanism test of Ag_3PO_4 sample

Sample	Degradation (%)
Control	89.7
IPA	92.6
AO	51.8
BQ	44.3
BQ+ H_2O_2	50.4

The $\bullet\text{OH}$ species was not observed based on the mechanical test results. Insufficient energy of Valence Band (VB) (+2.45 V vs. NHE) of Ag_3PO_4 to oxidize the H_2O to be $\bullet\text{OH}$ ($E^\circ(\bullet\text{OH}/\text{H}_2\text{O}) = +2.68 \text{ V vs. NHE}$) causing the $\bullet\text{OH}$ radical has been not produced in MB decomposition. On other hand, the $\bullet\text{O}_2^-$ radical could be the main radical species, even though the CB level of Ag_3PO_4 is more positive (+0.45 V vs. NHE) than a single-reduction reaction of O_2 ($E^\circ(\text{O}_2/\text{O}_2^{\bullet-}) = -0.33 \text{ V vs. NHE}$) (Katsumata et al., 2013). However, the $\bullet\text{O}_2^-$ and h^+ as main and secondary radical species of Ag_3PO_4 for

degrading the dye wastewater have been reported before (Xu et al., 2017; Pavanello et al., 2020). We assumed the O₂ reduction mechanism might be happened by the O₂ dissolution that has been resulted under bubbling processes through stirring treatment.

Possible mechanism of Ag₃PO₄ photocatalyst

Based on the results above, the photocatalytic process of MB dye is proposed as follows. The possible mechanism illustration is shown in Fig. 7. When Ag₃PO₄ is irradiated using visible light, e⁻ can be excited to the CB and left h⁺ in the VB. An excited e⁻ can be transferred to the O₂ producing the •O₂⁻ radical species. Radicals of photogenerated •O₂⁻ and h⁺ can break down the MB structures into small molecules and minerals, properly.

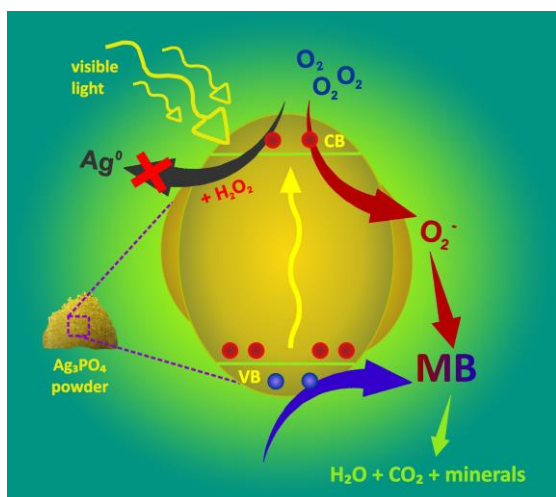


Figure 7. Dye photodegradation and H₂O₂-inhibited Ag metal production of Ag₃PO₄ powder. Blue and red circles were hole (h⁺) and electron (e⁻), whereas VB and CB were Valence Band and Conduction Band levels, respectively.

However, under simultaneous catalysis reaction, e⁻ can also be easily captured by Ag⁺ and changed into Ag⁰ (Wang et al., 2014; Mu et al., 2019). As the prolonging reaction time, more silver was likely to undergo photoreduction. It was indicated by the yellow precipitate of Ag₃PO₄ photocatalyst became grey or dark color as illustrated in Fig. 5 for pristine Ag₃PO₄. It

concluded that the photo-corrosion of Ag₃PO₄ had occurred and led to the decrease in the catalysis efficiency after every cycle (Fig. 4 and 5).

In contrast, the addition of H₂O₂ compound can inhibit directly the photo-corrosion of Ag₃PO₄ into Ag⁰, due to the more negative reduction potential of •OH/H₂O₂ compared to Ag/Ag₃PO₄ (H₂O₂ + e⁻ → •OH + •OH⁻, E°=0.87 eV vs. NHE). Notwithstanding, the excessive time reaction and more H₂O₂ solution will oxidize the Ag⁰ into Ag⁺ ions (Eq. 2). Besides, without the precipitation agents, Ag⁺ ions will release to the suspension, causing severe Ag₃PO₄ structure destruction. It can be seen in Fig. 5 for the AH-10% sample. Therefore, a proper consideration in the H₂O₂ quantity and principal aim to create the high photocatalytic and stability properties of Ag₃PO₄ should be selectively selected.

Conclusions

The direct addition of H₂O₂ into the Ag₃PO₄ suspension system has enhanced the stability property of its catalysis reaction for MB photodegradation under visible light. The maximum condition of H₂O₂ was 5 mL (AH-10% sample) with the percentage of MB removal being 70%. This study found that the excessive addition of H₂O₂ could enhance the stability of catalyst up to three-cycle runs, indicating that the photo-corrosion of Ag₃PO₄ photocatalyst had been restricted simultaneously. Although, the Ag⁰ oxidation into released-Ag⁺ ions caused by H₂O₂ addition, however, should be considered for an efficient and effective way of photocatalysis reaction system of Ag₃PO₄ photocatalyst in the future.

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