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A Green Strategy to Synthesize Ag/Ag$_3$PO$_4$/Chitosan Composite Photocatalysts and Their Photocatalytic Degradation Performance under Visible Light Irradiation

Peng-Cheng Wu* | Hui-Li Peng* | Yu-Han Wu* | Lei Li | Xiao-Rui Hao | Bang-Hua Peng | Gui-Hua Meng | Jian-Ning Wu* | Zhi-Yong Liu*

Abstract—In this work, the plasmonic Ag/Ag$_3$PO$_4$/chitosan (Ag/Ag$_3$PO$_4$/CS) composite photocatalyst was prepared by a low-temperature strategy. Environmentally friendly CS plays triple vital roles in this composite. First, it was devoted to in situ reducing metallic silver from silver ions of Ag$_3$PO$_4$. Also, as the carrier of Ag/Ag$_3$PO$_4$ nanoparticles, CS can effectively prevent aggregation. Furthermore, benefitting from the settlement of hydrophilic CS, the prepared composite could be easily separated and recovered from the solution system. X-ray diffraction (XRD), the scanning electron microscope, energy-dispersive X-ray spectroscopy (EDS), ultraviolet-visible (UV-vis) diffused reflectance spectroscopy, and X-ray photoelectron spectroscopy (XPS) were employed to characterize the properties of materials. The results of photo-decomposition testing showed that the Ag/Ag$_3$PO$_4$/CS composite possessed good activity for the decomposition of Rhodamine B (RhB) under visible light.

Index Terms—Ag$_3$PO$_4$, chitosan (CS), in situ reduction, photocatalyst.

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1. Introduction

Nowadays, the environmental pollution and energy crisis are the main challenges for the future of humanity. How to address these two issues efficiently and simply has become the focus of research. Photocatalysis, a "green" technology, has been considered to be one of the most promising and effective methods for environmental treatment and energy conversion, including decomposition of organic pollution, reduction of high toxicity heavy metal ion, water splitting, and carbon dioxide reduction[11][12]. Among various photocatalysts, owing to its low cost, good stability, high activity, and non-toxicity, inorganic TiO$_2$ is the most studied[3][4]. However, it is worth noting that the photocatalytic activity of TiO$_2$ is extremely limited by its intrinsic property of the large bandgap ($E_g=3.2$ eV), indicating that it only responds to ultraviolet (UV) light (less than 5% of the solar spectrum). As is known, the visible light is about ten times abundant than UV light (~48%), hence the development of visible-light responsive photocatalysts arouses wide research interest in recent years[9].

In 2010, Ye’s group firstly explored and certificated the photooxidation property of silver phosphate ($E_g=2.36$ eV) under visible-light, after that this material has been widely studied in the field of oxidation of water and photodegradation of organic pollution[7]. When the wavelength of irradiation light is longer than 420 nm, Ag$_3$PO$_4$ has excellent quantum efficiency and photocatalytic activity[8]. Additionally, there are many experimental results showing that the surface plasmon resonance (SPR) effect of Ag$^0$ could drastically improve the activity and stability of Ag$_3$PO$_4$[9][10]. However, nano-sized Ag/Ag$_3$PO$_4$ is easy to aggregate into micrometer-sized particles in aqueous solutions, which would greatly decrease the photocatalytic activity. However, nano-photocatalysts are difficult to isolate and recover from the reaction medium in a simple way during the practical application that will lead to secondary contamination. To solve these issues, immobilizing nano-photocatalysts onto/into supporting materials is considered as one of the easiest and most effective ways. Until now, various kinds of materials have been developed as carriers, such as the inorganic clay[12][13], carbonaceous materials[17][18], metal oxide[22][23], and organics[24][25]. As mentioned above, the SPR effect could enhance the photocatalytic performance of Ag$_3$PO$_4$, but the progress of reducing Ag$^+$ to Ag$^0$ always meets some challenges. UV irradiation and high-temperature polyol reduction are the most general fabrication methods, but they are costly, tedious, and time-consuming. Therefore, the development of suitable carriers and facile reducing approaches is necessary and valuable.

Chitosan (CS), a hydrophilic and cationic polymer with lots of hydroxyl and amino groups, is the second most abundant natural biopolymer on the earth. CS is usually used as an adsorbent in environmental science. These functional groups could provide plentiful chelation sites for metal ions and organic contaminants[27][28]. In the domain of catalysis, CS has been selected as carriers to synthesize composite catalysts for the Suzuki coupling and catalase-like reaction[29], aerobic oxidation of alkyl arenes and alcohols[30], reduction of aromatic nitro compounds[31], advanced oxidation processes[32], and so on. Besides, previous work had demonstrated that CS could serve as an effective reduction agent and stabilizing agent to prepare noble metal nanoparticles. Qian and co-worker[33] employed CS as a mediator agent under the quiescent crystallization condition to synthesize Ag and Au nanoparticles. Wei et al.[34] took CS as a reductant and scaffold to prepare metal nanoparticles-CS bioconjugates. Taking advantage of these features of CS, our group synthesized three composite catalysts, Ag nanoparticles/N-doped carbon[35], Ag/AgCl/N-doped carbon[36], Ag/AgCl/CS[37], and applied them for the degradation of organic pollutants.

In light of the problems of Ag/Ag$_3$PO$_4$ nanoparticles and the characteristics of CS, in this work, we prepared an Ag/Ag$_3$PO$_4$/CS composite photocatalyst via a “green” strategy for photo-decomposition of organic dye. During the preparation process, Ag$^+$ is partially in situ reduced to Ag$^0$ by CS at low temperature; therefore, the reductant or UV light is no longer needed. As the carrier, CS could effectively prevent the agglomeration of Ag/Ag$_3$PO$_4$ nanoparticles. The photodegradation experiments indicated that the Ag/Ag$_3$PO$_4$/CS composites possess the superior photocatalytic activity in RhB degradation under visible light. Also, benefiting from the settlement of CS,
the Ag/Ag₃PO₄/CS composite photocatalysts could be easily separated from the medium solution and reused in the next experiment.

2. Experimental

2.1. Materials

CS powder was bought from Shanghai Lanji Technological Development Co., Ltd. The degree of deacetylated is >90.0% and the viscosity is <100 cps. Silver nitrate and sodium chloride were obtained from Xi’an Chemical Reagent Factory. RhB was purchased from Tianjin Guangfu Fine Chemical Research Institute. Anhydrous ethanol came from Tianjin Fuyu Fine Chemical Reagent Company. All the reagents are analytical grade and without further purification for use.

2.2. Synthesis

The preparation process of Ag/Ag₃PO₄/CS is illustrated in Fig. 1. All the samples were prepared by the green and facile water bath heating method. Briefly, 0.5 g CS was dispersed in a certain concentration of the silver nitrate aqueous solution (40 mL) with continuously magnetic stirring for 2 h at room temperature. This progress is to reach the adsorption-chelation balance between CS and Ag⁺. Then, the 1.1 fold stoichiometric Na₂HPO₄ aqueous solution was slowly impregnated. The prepared mixture was heated to 80 °C and kept 3 h. Finally, the production was collected and rinsed with the deionized water and anhydrous ethanol for several times, respectively. After drying in the vacuum overnight at 50 °C, the samples were obtained. They were designated as 40-Ag/Ag₃PO₄/CS, 60-Ag/Ag₃PO₄/CS, and 80-Ag/Ag₃PO₄/CS, and the corresponding mass ratios of Ag₃PO₄ to CS were 40%, 60%, and 80%, respectively. 80-Ag₃PO₄/CS was prepared in the same method and the difference is without heating. What needs special emphasis is that all the steps need to be covered with the tin foil to make sure Ag₃PO₄ would not be reduced by natural light.

2.3. Characterizations

Crystal structures of the samples were identified by X-ray diffraction (XRD) with Cu Kα radiation in a scanning range of 10° to 80° on an X-ray diffractometer (Bruker D8 Advance, Germany). Surface morphologies of the samples were observed by scanning electron microscope (SEM, Hitachi, S4800). X-ray photoelectron spectroscopy (XPS) was measured by using an axis ultra spectrometer with monochromatized Al Kα X-ray as the excitation source (225 W). The UV-visible diffuse reflection spectra (UV-vis DRS) were used to study the optical property by a UV-vis spectrophotometer (UV-2450, Corporation, Shimadzu).
2.4. Photocatalytic Activity

The photocatalytic experiments were carried out by the degradation of RhB \((C_0=10 \text{ mg/L})\) using a 150 W Xe lamp with a cutoff filter to filter the UV light \((\lambda<420 \text{ nm})\). Typically, 50 mg as-prepared photocatalysts were dispersed in a quartz vessel with the RhB aqueous solution of 50 mL, and the suspensions were magnetically stirred in the dark for 60 min to establish an adsorption-desorption equilibrium. The distance between the center of the quartz vessel and the lamp kept about 20 cm. Then turned on the cooling water and the light, about 2.5 mL aliquots were pipetted from the mixture every 10 min. To remove the residual photocatalyst thoroughly, the suspension was immediately centrifuged at 3000 rpm for 3 min. The RhB remnant concentration was recorded by a UV-vis spectrophotometer (TU-1901, Beijing) at its characteristic wavelength of 553 nm.

3. Results and Discussion

3.1. Photocatalyst Characterization

Figs. 2 (a) to (d) show the SEM images of CS and different mass ratio composite photocatalysts. In comparison with the other three samples, CS has a rather smooth surface. After the reaction, many particles can be clearly found on the surface of CS, and as the ratio increased, the more particles were loaded on the CS. It is worth noting that the particles on the CS surface do not have extensive agglomeration, which is attributed to the abundant hydroxyl and amino groups of the CS anchor silver ion before it reacts with sodium hydrogen phosphate. These well-dispersed nanoparticles will make the photocatalytic effect more efficient. The energy-dispersive X-ray spectroscopy (EDS) confirms the elemental composition of the composite (Fig. 2 (e)), including C, N, and O that come from CS, while Ag and P could be attributed to Ag\(_3\)PO\(_4\).

The XRD measurement was performed to confirm the crystal phase of the prepared sample (Fig. 2 (f)). The diffraction peaks at 21.17°, 29.82°, 33.49°, 42.66°, 47.99°, 52.92°, 55.27°, 57.57°, 61.93°, 70.18°, and 71.13° are well agreement with the crystal planes of (110), (200), (210), (211), (220), (310), (222), (320), (321), (400), (420), and (421) of the body-centered cubic phase Ag\(_3\)PO\(_4\) (Joint Committee on Powder Diffraction Standards (JCPDS), No. 06-0505). However, there are not any obvious diffraction peaks corresponding to metallic silver, a kind of reasonable explanation is that the content of silver is low or the size of Ag particles is too small. The similar situation had also been reported in the previous work of Ag\(_3\)X \((X=\text{Cl, Br, or I})\)\(^{[38],[39]}\).

XPS was used to study the elemental composition and chemical status of Ag\(_3\)PO\(_4\)/CS, and prove the existence of metallic silver. Fig. 3 (a) displays the fully scanned spectra of 80-Ag\(_3\)PO\(_4\)/CS from 0 to 1200 eV, it is clearly seen that the sample mainly composed of C, N, O, Ag, and P elements, indicating that there are no other impurities in the sample. The detailed spectra of Ag are shown in Fig. 3 (b). Two bands at 367.2 eV and 373.2 eV are clearly observed, ascribing to Ag 3d\(_{5/2}\) and Ag 3d\(_{3/2}\) binding energy, respectively. These two peaks can be further divided into two separate peaks. Based on the research result of Ferraria et al.\(^{[40]}\), the Ag\(^+\) photoelectron chemical shift is slightly lower than metallic silver. The predominant cause of this peculiar shift is due to initial state factors of the ionic charge and lattice potential\(^{[40]}\). Therefore, the peaks at 367.7 eV and 373.7 eV can be attributed to Ag\(^+\), indicating the existence of metallic silver in the prepared sample. And the peaks at 367.1 eV and 373.1 eV are assigned to Ag\(^+\) of Ag\(_3\)PO\(_4\). Fig. 3 (c) shows a broaden peak in the range of 130 eV to 135 eV of the P 2p spectrum that is corresponding to the phosphorous of Ag\(_3\)PO\(_4\).

The photo-absorption properties of 80-Ag\(_3\)PO\(_4\)/CS, 80-Ag\(_3\)PO\(_4\)/CS, and CS were measured by UV-vis DRS (Fig. 4). As a blank, the carrier CS has a very weak absorption. Compared with 80-Ag\(_3\)PO\(_4\)/CS, 80-Ag\(_3\)Ag\(_3\)PO\(_4\)/CS exhibits stronger absorbance in the whole region, especially, in the visible light region (at the range of 400 nm to 500 nm), which may arise from SPR of Ag nanoparticles. This is beneficial for making full use of solar light. In addition, the photographs of the sample before and after reaction would provide more intuitive evidence (insert images in Fig. 4) to verify the formation of metallic silver. The sample without water-bathing heating shows the
typical golden color of Ag$_3$PO$_4$, while the sample after reaction exhibits brownish yellow, indicating the production of metallic silver species during the reaction. Combining the optical properties with the XPS results, it could be confirmed that the existence of metallic silver in the composite.

3.2. Photocatalytic Activity and Mechanism

The photocatalytic activity of the CS, 80-Ag$_3$PO$_4$/CS, and 80-Ag$_3$PO$_4$/CS composites was firstly evaluated in terms of the degradation of RhB that is a kind of chemically stable and difficult degraded organic dye, under simulated visible light ($\lambda$>420 nm). As the blank test shown (Fig. 5 (a)), the self-degradation of RhB is negligible. CS possesses the absorption capacity of RhB to a certain extent, but lack of photodegradation activity. The
80-Ag/Ag₃PO₄/CS exhibited much higher photocatalytic efficiency than that of 80-Ag₃PO₄/CS during the same light irradiation time, which could be attributed to the SPR effect of nano-sized metallic Ag. Therefore, the better photocatalytic activity of 80-Ag/Ag₃PO₄/CS further confirms that the formation of Ag nanoparticles in the production.

Fig. 5 (b) shows the changes of the RhB aqueous solution in the absorption spectra at different times in the presence of 80-Ag/Ag₃PO₄/CS. At the beginning (0 min), the characteristic absorption peak of the conjugated chromophore structure at 554 nm is very obvious and sharp. After the light on, this peak decreases progressively and accompanied by a blue shift, indicating RhB was degraded to a series of N-demethylation intermediates.
Subsequently, the loading of the composite photocatalyst was investigated. The photodegradation curves of RhB as a function of time are displayed in Fig. 6 (a). During the dark absorption progress, the prepared samples

![Absorbance (a.u.)](image)

**Fig. 5.** Contrast and blank experiments: (a) photocatalytic efficiency for the degradation of RhB and (b) UV-vis spectra changes of RhB during the photocatalytic degradation by 80-Ag/Ag₃PO₄/CS.

Subsequently, the loading of the composite photocatalyst was investigated. The photodegradation curves of RhB as a function of time are displayed in Fig. 6 (a). During the dark absorption progress, the prepared samples

![Conversion (%)](image)

**Fig. 6.** Photocatalytic activity for the degradation of RhB: (a) photocatalytic efficiency curves of as-prepared samples with different mass ratios, (b) pseudo-first-order kinetics curves of different samples under visible light irradiation, (c) repeated photodegradation of RhB by 80-Ag/Ag₃PO₄/CS, and (d) comparison photographs of pure Ag₃PO₄ (left) and 80-Ag/Ag₃PO₄/CS (right) dispersed in water after standing for 1 min.
absorbed 5% to 10% RhB. All the samples had excellent photodegradation activity under visible light. When the reaction time reached 60 min, the degradation rates reach 66.48%, 79.17%, and 99.19% for 40-Ag/Ag₃PO₄/CS, 60-Ag/Ag₃PO₄/CS, and 80-Ag/Ag₃PO₄/CS, respectively. Fig. 6 (b) is the degradation kinetic constants of RhB over different photocatalysts; they were calculated based on a pseudo-first-order kinetic model as shown in (1):

$$\ln \left( \frac{C_t}{C_0} \right) = -kt$$  \hspace{1cm} (1)

where $C_t$ and $C_0$ are the time-dependent and initial concentration of RhB, and $k$ is the rate constant. The $k$ values of 40-Ag/Ag₃PO₄/CS, 60-Ag/Ag₃PO₄/CS, and 80-Ag/Ag₃PO₄/CS are 0.017 min⁻¹, 0.020 min⁻¹, and 0.049 min⁻¹, respectively. Table 1 summarizes the reported works on Ag₃PO₄-based photocatalysts for RhB degradation. The prepared-sample of this work has relatively good activity, although it is not the best. It must be emphasized that there are many factors could influence the result of the photocatalytic experiment, such as ambient temperature and light intensity. In addition, secondary pollution is one of the challenges in the field of photocatalyses; therefore, the separability of the nanoscale photocatalyst should be considered, it will be shown later.

Table 1: Comparative representation photocatalytic performance of Ag₃PO₄-based catalysts reported in other similar works

<table>
<thead>
<tr>
<th>Samples</th>
<th>Dosage (mg)</th>
<th>Concentration of RhB (mg·L⁻¹)</th>
<th>Time (min)</th>
<th>Efficiency (%)</th>
<th>$k$ (min⁻¹)</th>
<th>Ref.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ag/Ag₃PO₄/diatomite</td>
<td>20</td>
<td>20.0</td>
<td>60</td>
<td>90.00</td>
<td>0.0252</td>
<td>[16]</td>
</tr>
<tr>
<td>Ag₃PO₄/ZnO</td>
<td>50</td>
<td>4.5</td>
<td>40</td>
<td>70.00</td>
<td>0.0350</td>
<td>[43]</td>
</tr>
<tr>
<td>Ag/Ag₃PO₄</td>
<td>50</td>
<td>20.0</td>
<td>40</td>
<td>81.65</td>
<td>0.0440</td>
<td>[44]</td>
</tr>
<tr>
<td>Ag@Ag₃PO₄/graphene</td>
<td>60</td>
<td>12.0</td>
<td>18</td>
<td>97.00</td>
<td>0.1699</td>
<td>[45]</td>
</tr>
<tr>
<td>80-Ag/Ag₃PO₄/CS</td>
<td>50</td>
<td>10.0</td>
<td>60</td>
<td>99.19</td>
<td>0.0490</td>
<td>Our work</td>
</tr>
</tbody>
</table>

We chose 80-Ag/Ag₃PO₄/CS as an assessment object to test the cycling stability. As shown in Fig. 6 (c), after three times of successive cyclic experiments, the degradation rate was obviously decreased and still remained about 81.1%. There are three reasons may explain this phenomenon: 1) owing to the problem of photo-corrosion of Ag₃PO₄, a part of Ag⁺ was reduced to Ag⁰ during the photodegradation; 2) small part photocatalysts were lost during the cycling experiments; 3) the absorption ability of the residual hydroxyl and amino group in CS decreased[46]. Apart from the stability of the photocatalyst, the property of separation and recovery from the reaction medium is an important parameter that must be considered. As shown in Fig. 6, 80-Ag/Ag₃PO₄/CS (right in Fig. 6 (d) is very easy to separate from water, the majority of samples sink to the bottom of the bottle about 1 min, while the bottle with pure Ag₃PO₄ (left in Fig. 6 (d)) is still turbid. In the practical application of water purification, this property will drastically reduce the cost and avoid secondary pollution.

The photocatalytic mechanism of Ag/Ag₃PO₄/CS may be similar to previous related works (Fig. 7)[46][48]. Both Ag₃PO₄ and Ag nanoparticles can efficiently absorb visible light. Therefore, an amount of electron-hole pairs could be generated in Ag₃PO₄. The electrons in the valence band will jump up into the conduction band (CB), and then these electrons will immediately transfer to Ag nanoparticles, while the holes will stay in the valence band of Ag₃PO₄. After light absorption in the Ag/Ag₃PO₄/CS nanostructures and SPR excitation,
plasmons can decay, transferring the accumulated energy to electrons in CB of the $\text{Ag}_3\text{PO}_4$. The hot electrons produced by Ag nanoparticles and the photogenerated electrons would be trapped by $\text{O}_2$ on the surface of photocatalysts to create $\cdot\text{O}_2$. These reactive oxygen species could effectively oxidize the dye molecules into intermediates, $\text{CO}_2$ and $\text{H}_2\text{O}$. The photogenerated holes would react with the surrounding $\text{H}_2\text{O}$ or $\text{OH}^-$, and create reactive intermediate species ($\cdot\text{OH}$). There are two advantages of the existence of Ag nanoparticles. On the one hand, the excellent conductivity of silver nanoparticles enhances the interfacial charge transfer and stops the recombination of electron-hole pairs effectively. On the other hand, the hot electrons would be generated because of the SPR effect of Ag nanoparticles, which increases the performance of the composite catalyst.

4. Conclusions

In this work, we synthesized a plasmonic Ag/Ag$_3$PO$_4$/CS composite photocatalyst by a simple and green strategy. Ag$^+$ was in situ reduced to Ag$^0$ by renewable natural CS. In this case, additional reduction conditions were not needed. As the carrier, CS effectively prevented the agglomeration of Ag/Ag$_3$PO$_4$ nanoparticles. The results of photocatalytic experiments indicated that the Ag/Ag$_3$PO$_4$/CS photocatalysts have satisfying activity for organic contaminant degradation under visible light, but the stability needs further improvement. Therefore, the further work will focus on this issue. Fortunately, with the feature of easy separation, this kind of composite photocatalysts has a wide application prospect in water purification.

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