Facile one-step synthesis of PhC₂Cu nanowires with enhanced photocatalytic performance

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Abstract

In recent years, the research of phenylethynylcopper (PhC₂Cu) in photocatalysis has attracted immense attention. However, its synthesis requires two steps extending over 9 days. In this paper, the successful preparation of PhC₂Cu nanowires is reported using a highly rapid and facile one-step method directly using copper acetate and phenylacetylene as raw materials and ascorbic acid as reducing agent. The kinetic studies indicated that the synthetic reaction follows a pseudo-second-order equation through electrical conductivity. Comparative studies of the crystal structures, morphologies, and optical properties of PhC₂Cu prepared by the traditional two-step and the current one-step methods were conducted using XRD, SEM, UV–Vis Drs, FT-IR, Raman spectra, and photocurrent. Meanwhile, the PhC₂Cu nanowires exhibited excellent photocatalytic activity to degrade methyl violet (MV) and glyphosate. This facile and rapid method dramatically improves the preparation efficiency of PhC₂Cu, and the obtained PhC₂Cu also shows higher photocatalytic activity. This remarkable progress enables the possibility of large-scale and efficient preparation of PhC₂Cu with high photocatalytic activity, indicating their excellent application prospect in photocatalysis.

Keywords PhC₂Cu · One-step synthesis · Kinetic studies · Photodegradation

Introduction

With the continuous growth of textile, paper, and plastic industries in recent decades, hard-dealt organic wastewater emissions have significantly impacted the ecological environment and human health (Sun et al. 2017). Therefore, more and more attention has been paid to treating organic pollutants, such as dyes (Sansotera et al. 2019; Chen et al. 2020). Conventional wastewater treatment includes physical (Wang et al. 2011) and biological methods (He et al. 2016), ozone (Paucar et al. 2019), Fenton oxidation (Tan et al. 2020), green nano-membranes (Rabiee et al. 2022), magnetic high absorbers (Meidanchi and Akhavan 2014), and super- and/or sub-critical water (Jouyandeh et al. 2022), widely used to exclude organic pollutants from wastewater. Semiconductor photocatalysis has received extensive attention as one of the most appealing and attractive technologies (Reza et al. 2017; Prasad et al. 2020; Huang et al. 2017; Ye et al. 2018). Notably, photocatalysis can mineralize organic pollutants into CO₂ and H₂O under light irradiation, making it an ideal technology for treating organic pollutants.

As a fascinating organometallic polymer, PhC₂Cu (phenylethynylcopper) with a narrow bandgap (~2.48 eV) has been investigated as a visible light photocatalyst for the degradation of organic pollutants. It was reported that its photocatalytic activity was better than that of g-C₃N₄ under visible light by degradation of 2,4-DCP and phenol (Wei et al. 2018). Furthermore, the composites NiFe₂O₄/PhC₂Cu and Ag/PhC₂Cu had better photodegradation activities to tetracycline hydrochloride than that of PhC₂Cu (Zhao et al. 2020; Zhao et al. 2019). However, the traditional synthetic process of PhC₂Cu takes 9 days (Wei et al. 2018; Buckley et al. 2010), thus limiting the applications of this relatively low-efficiency method (Scheme 1). Although our previous
work improved the second step reaction (Zhao et al. 2020),
the preparation of PhC2Cu still needs two steps, and it is
of great significance to complete the one-step synthesis of
PhC2Cu.

Herein, a facile one-step method for fabrication of
PhC2Cu nanowires is demonstrated in Scheme 2. Cupric
acetate monohydrate was directly used as raw material
and dissolved in the mixed solution of methanol and water
(Vmethanol:Vwater = 48:2). Then, ascorbic acid was added to
reduce Cu2+ to Cu+, and PhC2Cu was formed rapidly within
1.5 h by adding phenylacetylene. This method can signifi-
cantly reduce the synthesis time and improve the synthe-
sis efficiency of PhC2Cu. It is worth noting that if absolute
methanol were used here, Cu2+ would be thoroughly reduced
to Cu0 metal nanoparticles when ascorbic acid was added.
Thus, PhC2Cu cannot be obtained.

**Experimental section**

**One-step synthesis of PhC2Cu**

A 0.200 g Cu(OAc)2 ·H2O powder was fully dissolved in
50 mL methanol (containing 4 v% of water) under sonica-
tion, then 0.176 g ascorbic acid and 0.12 mL phenylacety-
lene were added into the solution, and stirred at room tem-
perature for 1.5 h, PhC2Cu was obtained as a golden yellow
powder, which was centrifuged and washed three times with
purified water and ethanol, and then dried under vacuum at
60 °C for 4 h to obtain PhC2Cu.

**Determination of chemical kinetics for one-step
synthesis reaction of PhC2Cu**

A 0.200 g (1.0 mmol) Cu(OAc)2 ·H2O was fully dissolved in
50 mL methanol (containing 4 v% of water) under sonica-
tion at a constant temperature of 298 K (303 and 308 K), then
0.176 g (1.0 mmol) ascorbic acid was added into the solu-
tion. The conductivity change of the solution is monitored
by a conductivity meter during the whole process. When the
conductivity of the solution reached the minimum value,
0.12 mL (1.0 mmol) phenylacetylene was injected. The first
value was measured immediately, and other values were
recorded after a specified time interval (15 s). The rate con-
stants k at different temperatures were calculated by plotting
1/Lt ~ t. Then the activation energy E_a was calculated
by the Arrhenius formula.

**Photocatalytic experiments**

For convenience, P represents the PhC2Cu prepared by the
new one-step method, and P* represents that prepared by
the traditional two-step method in the following discussion.
The performances of P and P* were evaluated by the pho-
todegradation of MV and glyphosate under the irradiation
of visible light at room temperature. To eliminate thermal
effects, the experiments were conducted at 20 °C with a cir-
culating water system. Typically, 0.030 g of the powdered
P or P* were placed in a quartz vessel, then 2 mL CH3OH
was added dropwise to soak the powders, and 100 mL of MV
(10 mg/L) or glyphosate (16 mg/L) aqueous solution was
added to form a suspension. The suspensions were stirred
in the dark for 50 min to establish an adsorption–desorption
equilibrium. A 300-W Xe lamp was used as the light source
(wavelength ≥ 420 nm; light intensity was 0.024 W·cm−2).
At certain time intervals (MV, 10 min; glyphosate, 30 min),
4 mL of the suspension was sampled and filtrated by a filter
membrane to remove particulates. The filtrates from MV
degradation were analyzed by UV–Vis spectroscopy at
579 nm. The concentration of glyphosate was determined
by monitoring the absorbance at 243 nm on a UV–vis spec-
trophotometer with the nitrosylation method.

**Characterizations**

The crystal forms of the as-prepared photocatalysts were
tested by X-ray diffraction (Haoyuan, DX-2700BH, X-ray
Diffractometer). The morphology and texture of the

![Scheme 1](image1.png) **Scheme 1** The traditional synthetic process of PhC2Cu

![Scheme 2](image2.png) **Scheme 2** The process of one-step synthesis of PhC2Cu

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as-prepared photocatalysts were observed by field emission scanning electron microscopy (FE-SEM, Hitachi S4800). The diffuse reflectance spectra (DRS) of the as-prepared photocatalysts were recorded by a UV–vis spectrophotometer (Shimadzu UV-2700, Japan) in the 200–800 nm range using BaSO₄ as a reference sample. Raman spectroscopy was recorded in a microscopic confocal Raman spectrometer HORIBA HR 800 with an excitation of the Ar⁺ laser (514.5 nm). FT-IR was recorded on Thermo Fisher Nicolet iS50 using KBr as the background. Electrical conductivity values were measured using a conductivity meter (Conductivity Meter, DDS-100).

Results and discussion

The reaction of Cu⁺ and PhC≡CH is illustrated in Scheme 3. Before and after the reaction, phenylacetylene and PhC₂Cu have little effect on the conductivity of the solution (other substances, such as excess OAc⁻, remain unchanged, their effects on conductivity can be ignored). For a dilute solution, the conductivity L of a strong electrolyte is proportional to its concentration, and the total conductivity of the solution is equal to the sum of the conductivity of the electrolyte constituting the solution. With increasing reaction time, Cu⁺ and phenylacetylene formed PhC₂Cu precipitate rapidly, and the conductivity of the solution decreased gradually. Therefore, the conductivity meter was used to measure the conductivity change with time in this reaction process to track the change of reactant concentration with time.

The following kinetic model is often used to describe the second-order reaction rate equation of a solid–liquid system (Ho 2006).

\[ \frac{1}{C_t} = kt + \frac{1}{C_0} \]

where \( C_t \) is the concentration at the time of \( t \), \( C_0 \) is the initial concentration, and \( k \) is the rate constant. Furthermore, \( L_t \propto C_t \), so, plot with \( 1/L_t \sim t \), if a straight line is obtained, it can be determined that the reaction is a second-order reaction. Plots of \( 1/L_t \) vs. \( t \) at different temperatures are shown in Fig. 1.

These lines (1a–1c) have an excellent linear relationship. Relationship coefficients are more than 0.98 (\( r > 0.97 \)). Now, it can be determined that this is a pseudo-second-order reaction. The reaction rate constant \( k \) of the reaction at different temperatures can be obtained from the slope of the straight line (Table 1).

According to Arrhenius formula, plot with ln(\( k/[k] \)) ∼ 1/T (1d), the slope \( s \) of the line is ∼ 7.30×10³ K, and the activation energy \( E_a = s \times R = 7.30 \times 8.315 \times 10^3 = 60.7 \text{ kJ} \cdot \text{mol}^{-1} \). It is worth noting that due to the poor stability of Cu⁺ in solution, if the reaction is carried out at a temperature higher than 308 K, the reaction characteristics will no longer conform to the above-mentioned second-order reaction model due to the occurrence of side reactions.

The possible reaction mechanism between Cu⁺ and phenylacetylene is illustrated in Scheme 4. SE2 reaction occurred between Cu⁺ and phenylacetylene, transition state (I) was formed firstly; then protons left to form phenylethynylcopper.

The X-ray diffraction (XRD) patterns of P and P* samples are shown in Fig. 2. PhC₂Cu was the monoclinic phase. Their XRD peaks are not very smooth due to their poor crystallinity. However, their prominent diffraction peaks do not show significant differences. Therefore, it can be determined that P and P* have the same composition. The XRD patterns show several main diffraction peaks that were indexed to (100), (001), (200), (101), and (−102) crystal faces of PhC₂Cu.

Figure 3 shows typical SEM images of P and P* on the different scales. It clearly shows that both materials are nanowire structures. Their widths are about 30 nm, and the difference is not significant. However, the length of P is much smaller than that of P*. In other words, P has a smaller particle size, which means that P may have better catalytic activity in heterogeneous reactions (Becker et al. 2011). The specific surface areas of P and P* were accessed from the N₂-absorption isotherm (Fig. S1). The BET specific surface area determined for P and P* is 61.8 m² g⁻¹ and 45.4 m² g⁻¹, respectively (Supporting information).

The optical properties of the as-prepared P and P* samples were measured by UV–Vis DRS. Both P and P* nanowires exhibit strong absorption in the range of 250–480 nm, while in the range of 480–800 nm, their light-harvesting ability to visible light decreases rapidly, as shown in Fig. 4a. This means that enhancing their absorption capacity to visible light may be one of the focuses of future research. The band gap values of P and P* nanowires are approximately 2.20 eV in Fig. 4b, and their absorption edge is 563 nm. In previous research, the optical band gap of the PhC₂Cu (phenylethynylcopper) was ∼2.48 eV (Wei et al. 2018). The possible reason for such difference is that they have different particle sizes. The quantum size effect changes band gap...
width (Furukawa and Miyasato 1988; Samavati et al. 2021; Frame and Osterloh 2010).

Figure 5 represents the FT–IR spectrum of P recorded in the 3500–500 cm$^{-1}$ range. The peaks at 1928 cm$^{-1}$ are

Table 1 Reaction rate constant at different temperatures

| T/K  | 298  | 303  | 308  |
|------|------|------|------|
| k    | 3.00×10$^{-5}$ | 4.91×10$^{-5}$ | 6.64×10$^{-5}$ |

Scheme 4 The possible reaction mechanism of Cu$^{+}$ and phenylacetylene

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Fig. 2 XRD patterns of P and P* samples

Fig. 3 SEM images of a, b P and c, d P* samples
Fig. 4  a UV–Vis Drs of the as-prepared P and P* samples. b Plots of (αhν)^1/2 vs. E (eV)

Fig. 5 FT–IR spectrum of P and P* samples
attributed to the C≡C bridging modes (Chui et al. 2005), as shown in Fig. 5. The peaks at 1595, 1480, and 1442 cm\(^{-1}\) are attributed to the stretching vibration of the C≡C band of phenyl groups. At the same time, 748 and 686 cm\(^{-1}\) are the characteristic absorption peaks of bending vibration of the benzene ring. Furthermore, P nanowire has no absorption peaks at 650 cm\(^{-1}\) (ascribed to -C≡C-H), proving that hydrogen was entirely substituted by copper (Krikor et al. 1987). Compared with P*, the characteristic infrared absorptions of P show a particular red shift. This phenomenon may be attributed to the surface effects of the material. The proportion of surface atoms increases with the decrease of particle size. Compared with bulk atoms, the distance between surface atoms is larger, and the force constant of corresponding surface atoms is smaller, resulting in decreased surface phonon frequency. In this way, the relaxation effect of surface atoms may lead to the redshift of the infrared spectrum.

The Raman spectra of P nanowire at about 1006, 1186, and 1604 cm\(^{-1}\) are the characteristic lines of the benzene ring (Fig. 6). The Raman band at 1936 cm\(^{-1}\) may be

Fig. 6 Raman spectrum of P and P* nanowires
attributed to a second-order transition enhanced by Fermi resonance with v(C≡C) (Garbusova et al. 1973). Other lines (536, 790 cm\(^{-1}\)) may be ascribed to vibrations originating from the coordination interaction between copper and phenylacetylene. It is worth noting that the positions of these lines show a “redshift” compared with the previous reports. This may be due to the smaller size of P nanowires.

Transient photocurrent responses of P and P* nanowires were measured under intermittent visible light irradiation, as shown in Fig. 7. Notably, the photocurrent density of P nanowire was about 1.10 μA/cm\(^2\), which is 14 times higher than that of P* at the same condition, which can be attributed to the quantum size effect (Frame and Osterloh 2010; Yu et al. 2006). This could mean that P nanowire has better application potential in photoelectric conversion.

The photocatalytic activities of P and P* nanowires were examined by degrading MV and glyphosate under visible light irradiation (a 300-W xenon lamp was used as the visible light source). Before the irradiation, adsorption–desorption equilibrium was established by stirring in the dark for 50 min. In the photodegradation process, P nanowires exhibit a better catalytic activity than P*, as shown in Fig. 8. After 30 min of illumination, the degradation rates of MV and glyphosate were 97% and 75%, respectively, as seen in

Fig. 7 Photocurrent density of P and P* samples

Fig. 8 Photocatalytic degradation curves of MV and glyphosate over P and P* nanowires

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Table 2 Photodegradation rates normalized by the surface area

| Photodegradation rates | P-MV     | P*-MV    | P-glyphosate | P*-glyphosate |
|------------------------|----------|----------|--------------|---------------|
| Photodegradation rates  | −0.1109  | −0.0405  | −0.0094      | −0.0082       |
| Photodegradation rates normalized by the surface area | −1.82*10⁻³ | −0.99*10⁻³ | −1.52*10⁻⁴ | −1.80*10⁻⁴ |

Fig. 8. Glyphosate is more difficult to degrade than methyl violet. After 150 min of illumination, their degradation rates were 62% and 58%, respectively. The possible reasons why P has better photocatalytic performance are as follows: (1) P has a larger specific surface area, so it has higher surface energy and stronger photocatalytic activity. (2) P exhibits quantum size effects due to the smaller particle size and has better photoelectric response to visible light. The photogenerated charge carriers in P are separated more efficiently than that in P* (Tian et al. 2015).

The pre-sorption experiments were conducted in dark to determine the contribution of the adsorption of MV and glyphosate. For P, the initial absorbance of MV and glyphosate is 0.198 and 0.610, respectively, when the adsorption–desorption equilibrium reached, the absorbances became 0.093 and 0.354, so the contribution of adsorption is 53% and 42% respectively.

According to the previous studies, when the initial concentration (C₀) of MV or glyphosate solution is small, the Langmuire-Hinshelwood model could be employed to investigate the kinetics of the MV degradation, which was ascribed to a pseudo-first-order reaction (Wang et al. 2015):

\[ \ln\left(\frac{C}{C_0}\right) = kt \]

where \( C \) is the concentration of MV or glyphosate at time \( t \), \( C_0 \) is the initial concentration of MV or glyphosate, the slope \( k \) is the apparent reaction rate constant, and higher \( k \) value indicates faster degradation rate. Figure 8b displays the pseudo-first-order kinetics curves for the photodegradation of MV and glyphosate. The photodegradation rates normalized by the surface area are listed in the Table 2. It can be seen from the results in Table 2 that the specific surface area has a certain impact on the photodegradation of glyphosate.

**Conclusions**

In summary, this paper reports a new and facile one-step process for rapidly synthesizing PhC2Cu nanowires. The kinetic studies indicated that the synthetic reaction process follows a pseudo-second-order equation through electrical conductivity. The PhC2Cu nanowires prepared by the new method have smaller particle sizes than those prepared by the traditional method. Moreover, the PhC2Cu nanowires have intense absorption in the visible light range. It exhibits a better activity for MV degradation under visible-light irradiation. MV and glyphosate degradation rates were 97% and 70%, respectively. This one-step method can significantly improve the synthesis efficiency of PhC2Cu. Furthermore, the obtained PhC2Cu shows higher photocatalytic activity than that of the PhC2Cu prepared by the traditional two-step method. This strategy proposed a new avenue for designing and constructing novel metal–organic polymer photocatalysts.

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**Author contribution** Gao-yu Zhao: overall article, experimental method, result analysis, and writing—original draft. Hai-yan Li: result analysis, experimental operations and data analysis. Yu Yang: experimental operations. Qing-hua Zhao: experimental operations. Ting-ting Su: experimental operations, Jiang-li Ma: experimental operations. All authors have read and agreed to the published version of the manuscript.

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**Declarations**

**Ethics approval and consent to participate** Not applicable.

**Consent for publication** Not applicable.

**Conflict of interest** The authors declare no competing interests.

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