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Synthesis of Nanorod g-C_3N_4/Ag_3PO_4 Composites and Photocatalytic Activity for Removing Organic Dyes under Visible Light Condition

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Abstract: Nanorod graphitic carbon nitride (g-C_3N_4) was synthesized by reacting melamine ($C_3H_6N_6$) with trithiocyanuric acid ($C_3H_3N_3S_3$) in distilled water for 10 h at room temperature. The resulting mixture was calcined at 550°C for 2 h in an electric furnace under an air atmosphere. Nanorod g-C_3N_4/Ag_3PO_4 composites were prepared by adding nanorod graphitic carbon nitride (g-C_3N_4) powder, silver nitrate ($AgNO_3$), ammonia ($NH_3 \cdot H_2O$, 25.0-30.0%), and sodium hydrogen phosphate (Na_2HPO_4) to distilled water. The samples were characterized via X-ray diffraction, scanning electron microscopy, and Fourier-transform infrared spectroscopy. The photocatalytic activities of the nanorod g-C_3N_4/Ag_3PO_4 composites were demonstrated via the degradation of organic dyes, such as methylene blue and methyl orange, under blue light-emitting diode irradiation and evaluated using UV-vis spectrophotometry.

Keywords: Nanorod g-C_3N_4, Ag_3PO_4, photocatalytic activities, organic dyes, blue LED

Introduction

Wastewater from the textile industry contains large amounts of dyes that can be harmful to the environment.[1,2] Dyes are a significant water pollutant in the wastewater of textile, leather, food processing, dyeing, cosmetics, paper, and dye manufacturing industries.[3,4] Fortunately, the problem of water pollution from dyes has been solved with semiconductor photocatalytic technology.[5-7] The degradation of organic pollutants using photocatalysis is a very promising method that accessed as an economical and environmentally friendly solution.[8,9] Among the photocatalysts, titanium oxide (TiO_2)-based nanomaterials are considered among the most reliable photocatalytic materials for degrading toxic and hazardous organic pollutants.[10,11] However, TiO_2 is only responsive to wavelengths in the ultraviolet light, which can absorb only 4% of the solar spectrum.[12] Therefore, there is need to enhance the photocatalyst capability to absorb solar energy.[13,14]

Among semiconductor photocatalysts, graphite-like carbon nitride has attracted huge attention due to its unique properties, such as high chemical stability, high thermal and photochemical stability, strong mechanical properties, and nontoxicity.[15,16] Graphitic carbon nitride forms a 2D layer derived from the tri-s-triazine unit structures of carbon and nitrogen. These 2D layers are stacked together by strong van der Waals forces.[17,18]

Since the discovery by Wang's group in 2009, metal-free graphite nitride (g-C_3N_4) has received considerable attention from scientists.[19] The g-C_3N_4 has been reported as a metal-free polymer-like semiconductor photocatalyst with thermal and chemical stability as well as low cost.[20] The g-C_3N_4 photocatalyst has a bandgap of 2.7 eV and can absorb light up to 450 nm.[21] However, the application of single g-C_3N_4 photocatalyst has been limited by high electron-hole recombination and low specific surface area.[22] To overcome the limitation of photocatalyst, several strategies, such as combinations with metals and non-metals and heterojunction configurations, have been used for g-C_3N_4.[23,24]

Ag_3PO_4 photocatalyst has received great attention owing to its bandgap (2.45 eV) and excellent visible light-driven photocatalytic activity for the degradation of organic pollutants.[25,26] Ag_3PO_4 contains an electric field between the PO_4^{3-} and Ag^+ ions, resulting in a quantum efficiency of approximately 90% at 400-480 nm due to the separation of the

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photoelectron-hole pairs.[27,28]

In this study, the photocatalytic activities of the nanorod g-C_3N_4/Ag_3PO_4 were investigated to remove organic dyes, such as MB and MO, under blue LED (at 450 nm).

Experimental

1. Materials

Melamine ($C_3H_6N_6$), sodium hydrogen phosphate (Na_2HPO_4) and trithiocyanuric acid ($C_3H_3N_3S_3$) were purchased from Alfa Aesar. Ammonia solution ($NH_3 \cdot H_2O$ 25.0%~30.0%), ethyl alcohol (C_2H_5OH 99.9%), methylene blue (MB), methyl orange (MO) and silver nitrate ($AgNO_3$) were purchased from Samchun Chemicals.

2. Instruments

XRD pattern and crystallite size of the synthesized samples were obtained using X-ray diffraction (Bruker, D8 Advance) with a Cu Kα radiation source (λ=1.54178 Å). The surface morphology was investigated using SEM (JEOL Ltd, JSM-6510) at an acceleration voltage of 20 kV. FTIR analysis was performed using a Thermo Fisher Scientific instrument with sample powders diluted in KBr pellets. Photocatalytic degradation of the organic dyes was conducted using a UV-vis spectrophotometer (Perkin-Elmer) in the wavelength range of 200-800 nm with a Lambda 365. The organic dye solution was irradiated with visible light using blue LED (5W, 450 nm, T5 Jinsung Electronic., Ltd).

3. Synthesis of nanorod g-C_3N_4

Typically, to 100 mL of distilled water, 1.261 g of melamine ($C_3H_6N_6$) and 1.773 g of trithiocyanuric acid ($C_3H_3N_3S_3$) were added, followed by stirring at room temperature for 10 h. The resulting faint yellow precipitation was washed thrice with ethanol by centrifugation. The product was dried at 60°C for overnight, then calcined in an electric furnace at 550°C for 2 h at heating rate of 5°C/min in air atmosphere.

4. Synthesis of nanorod g-C_3N_4/Ag_3PO_4 composites

To synthesize the nanorod g-C_3N_4/Ag_3PO_4 composite, 0.3 g of the synthesized nanorod g-C_3N_4 powder was dispersed in 100 mL of distilled water under ultrasonic irradiation for 60 min. 0.17 g of silver nitrate was dissolved in 20 mL of distilled water. The silver nitrate solution was stirred into the suspended nanorod g-C_3N_4 solution using a magnetic stirrer for 30 min.

0.1 M of an aqueous ammonia solution was added dropwise to the solution at room temperature for 50 min. Then, 0.426 g of sodium hydrogen phosphate was dissolved in 15 mL of distilled water, added dropwise and mixed solution was stirred for 30 min. The product was washed thrice with ethanol and dried at 60°C for 12 h.

5. Photocatalytic degradation process of organic dyes

The concentration of MB and MO in the organic dye solution was 0.63×10^{-2} mM and 3.67×10^{-2} mM, respectively. The MB solution exhibited an absorption peak at 665 nm and the MO solution exhibited an absorption peak at 464 nm. The photocatalyst powder (5 mg) was added to a conical tube which containing 10 ml of an aqueous organic dye solution. To achieve adsorption-desorption equilibrium between the organic dye solution and photocatalysts, the conical tubes were kept in the dark environment for 15 min. The blue LED was used to provide a visible light source at a distance of 1 cm between the LED and the aqueous organic dye solution. The photocatalytic degradation of the organic dye was analyzed using a UV-vis spectrophotometer, monitoring at 15 min intervals.

Results and Discussion

1. XRD pattern

Figure 1 shows the XRD pattern of the nanorod g-C_3N_4/Ag_3PO_4 composites. Peaks at $2\theta = 13.1°$ and 27.6° were owing to the nanorod g-C_3N_4 and were assigned to the (100) and (002) planes, respectively. The main peak at $2\theta = 27.6°$ can be indexed to the (002) plane of graphite-like materials (JCPDS card no. 87-1526).[29-31] Peaks at $2\theta = 20.93°$, 29.75°, 33.35°, 36.64°, 42.56°, 47.87°, 52.74°, 55.08°, 57.33°, 61.73°, 66.09°, 69.98°, 71.97°, and 74.24° correspond to the (110), (200), (210), (211), (220), (310), (222), (320), (321), (400), (330), (420), (421), and (332) planes of Ag_3PO_4 (JCPDS file No. 06-0505).[32-34] No other diffraction peaks were observed for the nanorod g-C_3N_4/Ag_3PO_4 composites. The crystallite size of the Ag_3PO_4 was calculated using the Scherrer equation[35]:

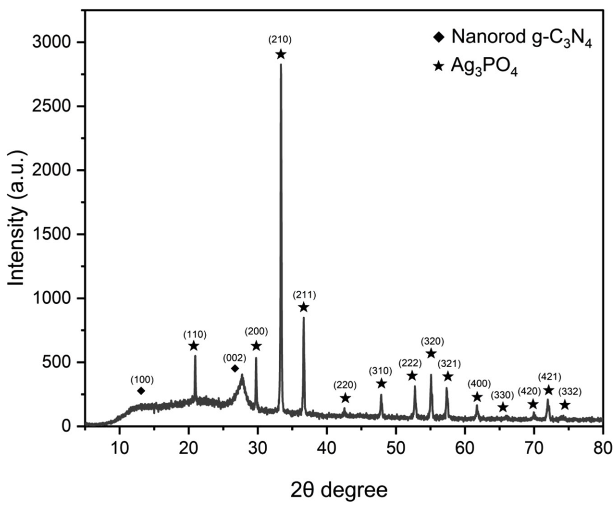

Figure 1. XRD pattern of synthesized nanorod g-C_3N_4/Ag_3PO_4 composites.

Table 1. Crystallite size of the Ag_3PO_4 in the g-C_3N_4/Ag_3PO_4 composites.

(hkl)	2θ	FWHM	Crystallite size (nm)
(200)	29.75°	0.144	59.64
(210)	33.35°	0.171	50.67
(211)	36.64°	0.194	45.07
Average			51.79

$$D = K\lambda / \beta\cos\theta.$$

where D is known as the crystallite size (nm), K is the Scherrer constant taken as 0.9, λ is the wavelength of the X-ray diffraction with Cu Kα, (λ = 1.5418 Å), β is the full width at half maximum (FWHM), and θ is the Bragg angle in degree. Table 1 shows the crystallite size of the Ag_3PO_4 at (200), (210), and (211) planes and the mean crystallite size of the Ag_3PO_4.

2. SEM image

Figure 2 shows the surface morphology of the nanorod g-C_3N_4/Ag_3PO_4 composites obtained using SEM. The SEM image shows two distinct structural morphologies in the synthesized nanorod g-C_3N_4/Ag_3PO_4 composites. Nanorod g-C_3N_4 exhibited an obvious one-dimensional rod shape, whereas Ag_3PO_4 had a cubic shape.

3. FT-IR spectra

Figure 3 shows the FTIR spectrum of the nanorod g-C_3N_4/

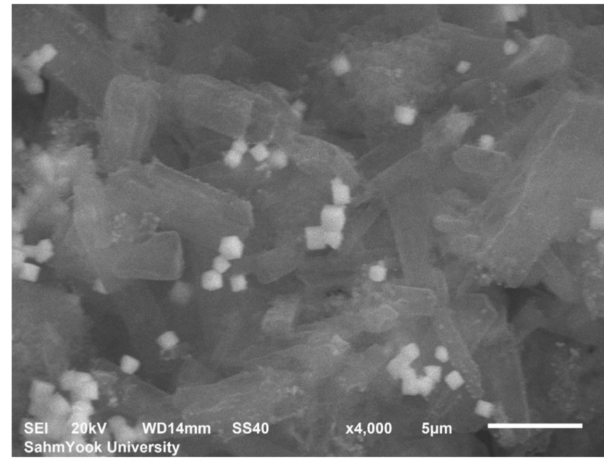

Figure 2. SEM image of synthesized nanorod g-C_3N_4/Ag_3PO_4 composites.

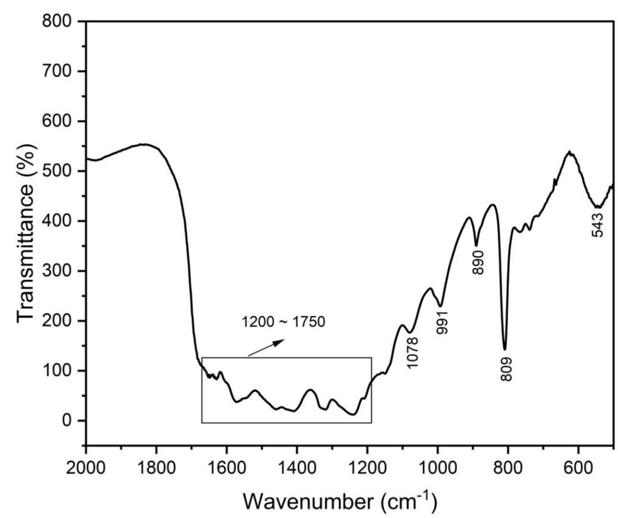

Figure 3. FT-IR spectrum of synthesized nanorod g-C_3N_4/Ag_3PO_4 composites.

Ag_3PO_4 composites used to characterize the various functional groups and interactions between the nanorod g-C_3N_4 and Ag_3PO_4. For nanorod g-C_3N_4, the sharp peaks at 809 and 890 cm^{-1} are attributed to the tri-s-triazine unit. The peaks in the range of 1200-1750 cm^{-1} are attributed to the aromatic CN heterocycles.[36-38] For Ag_3PO_4, the peaks at 543 cm^{-1} and 991 cm^{-1} are related to the P-O stretching vibration mode of PO_4^{3-}. The peak at 1078 cm^{-1} was attributed to the asymmetric stretching vibrations of the P-O-P group.[39-41]

4. Photocatalytic degradation efficiency of organic dyes

To study the photocatalytic degradation efficiency (PDE) of organic dyes such as MB and MO, the nanorod g-C_3N_4/

Ag_3PO_4 composites were irradiated using blue LED at 450 nm. The PDE was calculated using the following equation[42]:

$$PDE(\%) = \frac{C_0 - C_t}{C_0} \times 100 = \frac{I_0 - I_t}{I_0} \times 100$$

where C_0 represents the initial concentration of the organic dyes solution and C_t represents the concentration of the organic dyes solution at a certain time t. I_0 represents the intensity of the maximum absorbance peak in the UV-vis spectrum of the initial organic dye solution and I_t represents the intensity of the maximum absorbance peak in the UV-vis spectrum of the organic dyes solution at a certain time t.

Figure 4 shows the UV-vis spectra of photocatalytic degradation of MB and MO. The PDEs of MB and MO under blue LED irradiation were 93.24% and 61.94%, respectively. The photodegradation activity of the nanorod g-C_3N_4/Ag_3PO_4 composites was higher for MB than MO.

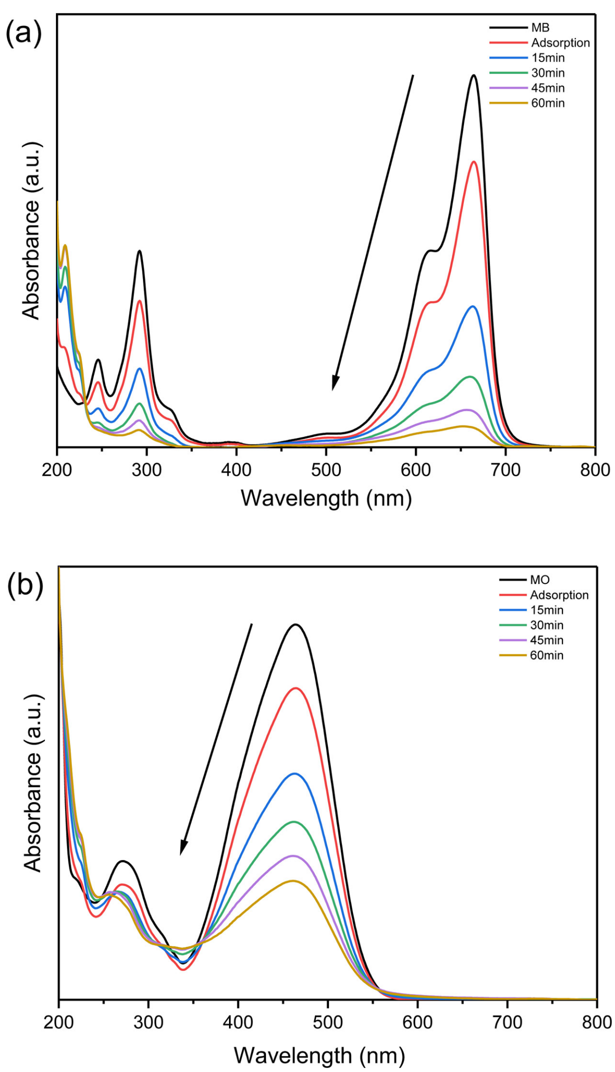

Figure 4. UV-vis spectra of photocatalytic degradation of (a) MB and (b) MO using nanorod g-C_3N_4/Ag_3PO_4 composites under blue LED (450 nm) irradiation.

5. Kinetics study

In the Langmuir-Hinshelwood kinetic model, the photocatalytic activity of MB and MO can be represented by the following apparent pseudo-first-order kinetic equations[43]:

$$\ln(C/C_0) = -K \cdot t$$

where C_0 represents the initial concentration of the organic dye solution, C represents the concentration at time t, and K is the rate constant of photocatalytic degradation.

MB has cationic properties that allow it to interact with anionic species, such as hydroxyl groups and superoxide anion radicals, on the photocatalytic surface. By contrast, MO tends to react with cationic species such as holes due to its anionic nature.[44] These difference can be attributed to the cationic and anionic properties of organic dye molecules. Figure 5 shows the results of kinetics study for MB and MO. Therefore, the MB had higher photocatalytic degradation efficiency and kinetics rate than for MO.

6. Photocatalytic degradation mechanism of organic dyes

Figure 6 illustrates the mechanism of the photocatalytic degradation of organic dyes using nanorod g-C_3N_4/Ag_3PO_4 composites. Under visible light, photogenerated electrons

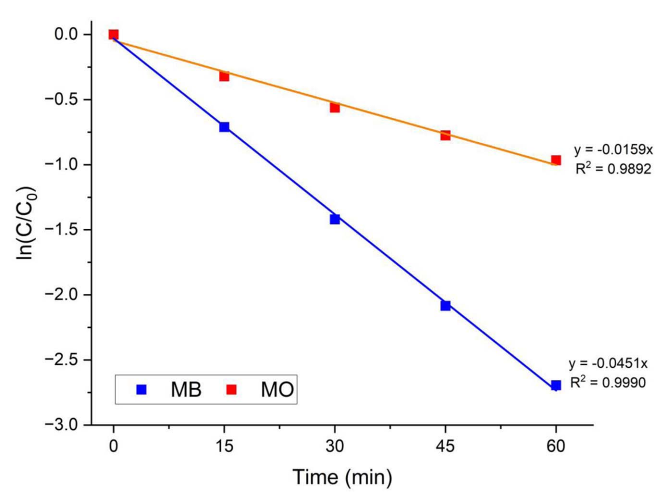

Figure 5. Kinetic studies of photocatalytic degradation of MB and MO using nanorod g-C_3N_4/Ag_3PO_4 composites under blue LED (450 nm) irradiation.

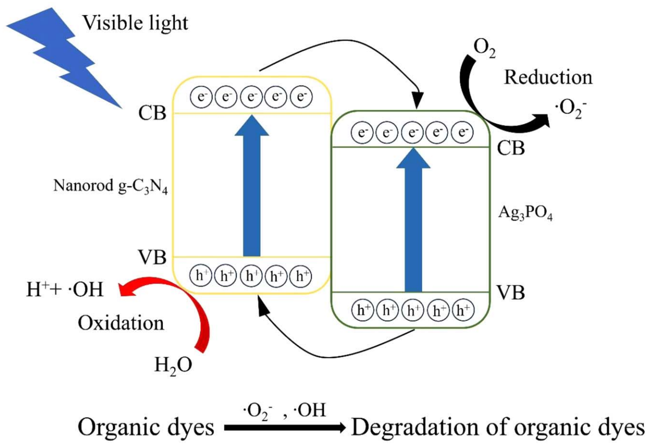

Figure 6. Mechanism of photocatalytic degradation for organic dyes using nanorod g-C_3N_4/Ag_3PO_4 composites under blue LED (450 nm) irradiation.

move from the conduction band of nanorod g-C_3N_4 to the Ag_3PO_4, while holes move from the valence band of Ag_3PO_4 to the nanorod g-C_3N_4, showing that the hybrid photocatalysts efficiently separate the photogenerated charges and reduce the recombination of electron-hole pairs. Consequently, water (H_2O) is oxidized through the holes (h^+) present in the valence band of the nanorods g-C_3N_4 to form hydroxyl radical (·OH), while oxygen (O_2) is reduced by electrons (e^-) from the conducting band of Ag_3PO_4 to form superoxide anion radical ($·O_2^-$). The electron and hole pairs can be generated the superoxide anion radicals ($•O_2^-$) and hydroxyl radicals (•OH) species to efficiently degrade organic pollutants.[45,46]

Conclusions

Rod-like g-C_3N_4 was obtained from precursors, such as melamine and trithiocyanuric acid. Nanorod g-C_3N_4/Ag_3PO_4 composites were synthesized by adding nanorod g-C_3N_4 product and silver nitrate ($AgNO_3$), ammonia ($NH_3·H_2O$ 25.0%-30.0%), and sodium hydrogen phosphate (Na_2HPO_4) to distilled water. The nanorod g-C_3N_4/Ag_3PO_4 composites were characterized using XRD, SEM and FT-IR. The synthesized hybrid photocatalyst was evaluated for the degradation of MB and MO under blue LED irradiation. The photocatalytic degradation processes were observed using a UV-vis spectrophotometer. The degradation of the organic dye process followed pseudo-first-order kinetics. Furthermore, the photocatalytic activity of the nanorod g-C_3N_4/Ag_3PO_4 composites for MB degradation under visible light was higher than for MO.

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Conflict of Interest: The authors declare that there is no conflict of interest.

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