

Synthesis of g-C₃N₄ Supported Au and Pt Nanoparticles for CO₂ Reduction

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ABSTRACT

The continuous increase in atmospheric CO₂ concentration, which has currently exceeded 400 ppm, has drawn significant attention to CO₂ capture and reduction technologies. In addition, the photocatalytic conversion of CO₂ into hydrocarbon fuels using semiconductor photocatalysts has attracted considerable interest as a promising approach to address global energy shortages. In this study, g-C₃N₄ was first synthesized via a thermal polymerization method using urea as the precursor. Subsequently, gold and platinum nanoparticles were deposited onto the surface of the synthesized g-C₃N₄ through a photodeposition method assisted by ethanolamine. The as-prepared photocatalyst was thoroughly characterized using various techniques, including Brunauer–Emmett–Teller (BET) surface area analysis, X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), transmission electron microscopy (TEM), X-ray photoelectron spectroscopy (XPS), and UV–visible spectroscopy (UV–vis). Microstructural analyses confirmed that the metallic nanoparticles were uniformly formed and well dispersed on the surface of g-C₃N₄ nanosheets. The presence of gold nanoparticles enhanced visible-light absorption due to the localized surface plasmon resonance effect. Moreover, the interaction between the metallic nanoparticles and the semiconductor matrix significantly improved the photocatalytic performance by suppressing electron–hole recombination. The synthesized nanocomposite exhibited a CO₂ reduction yield of approximately 33 μmol, which was nearly three times higher than that of pristine g-C₃N₄. The possible mechanisms involved in the CO₂ reduction process over the prepared nanocomposite were also discussed.

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1. Introduction

In recent years, human societies have been confronted with two major global challenges: the rapid depletion of fossil fuel resources and the continuous increase in atmospheric CO₂ concentration. Several strategies have been proposed to address these issues, including CO₂ capture and storage technologies, solar photovoltaic cells, and clean energy carriers such as hydrogen production via water electrolysis, as well as carbon-based fuels obtained from CO₂ reduction. Among these approaches, the conversion of CO₂ into value-added chemicals and clean fuels (e.g., CO, CH₄, or CH₃OH) is considered a highly promising strategy, as it has the potential to simultaneously mitigate environmental concerns and energy shortages [1,2]. Photocatalytic CO₂ reduction was first reported in the late 1970s, and since then, extensive research efforts have been devoted to the photocatalytic conversion of CO₂. Although TiO₂ is the most widely studied photocatalyst due to its strong reactivity, commercial availability, and excellent chemical stability, it can absorb only ultraviolet (UV) light, which accounts for approximately 4% of the solar spectrum. This limitation arises from the wide band gap of TiO₂ ($E_g = 3.2$ eV), significantly restricting its practical application as a solar-driven photocatalyst [3,4]. Therefore, the development of highly efficient photocatalysts with strong activity under solar irradiation remains an urgent research priority.

Graphitic carbon nitride (g-C₃N₄), owing to its moderate band gap ($E_g \approx 2.7$ eV), high chemical stability, and low cost, has recently emerged as a promising metal-free semiconductor photocatalyst. Since the first reports on photocatalytic hydrogen evolution over g-C₃N₄, substantial research efforts have focused on enhancing the photocatalytic performance of g-C₃N₄-based systems. g-C₃N₄ can be readily synthesized via thermal polymerization of various low-cost nitrogen-rich precursors, such as urea, thiourea, melamine, cyanamide, dicyandiamide, and guanidine hydrochloride [5,6]. Moreover, owing to its simple preparation route, g-C₃N₄ is generally more cost-effective than TiO₂.

Importantly, g-C₃N₄ possesses a much more negative conduction band potential ($E_{cb} \approx -1.20$ V) compared to TiO₂ ($E_{cb} \approx -0.29$ V), indicating that photoexcited electrons in g-C₃N₄ exhibit stronger reduction ability. This property enables the reduction of CO₂ into CH₃OH or other hydrocarbon fuels that are difficult to produce on TiO₂-based photocatalysts. However, pristine g-C₃N₄ still suffers from several inherent drawbacks, including low specific surface area, insufficient visible-light utilization, and particularly the rapid recombination of photogenerated charge carriers [7,8]. Consequently, enhancing the generation, separation, and lifetime of

photogenerated electron–hole pairs has become a major research focus in the development of efficient photocatalytic systems.

Among recent advances in g-C₃N₄-based photocatalysts, the construction of g-C₃N₄-based composites has attracted considerable attention due to its effectiveness in promoting charge separation and suppressing electron–hole recombination. Various materials have been coupled with g-C₃N₄ to improve its photocatalytic performance.

In recent years, it has been demonstrated that certain noble metals, such as gold, exhibit plasmonic properties at the nanoscale. Plasmonic nanoparticles are characterized by the collective oscillation of conduction electrons in response to incident electromagnetic radiation with wavelengths much larger than the particle size. Owing to their high surface-to-volume ratio, particularly at sizes below 20 nm, metallic nanoparticles exhibit pronounced localized surface plasmon resonance (LSPR) at specific frequencies, which for noble metals such as gold and silver typically fall within the visible-light region. This unique optical property enables plasmonic metals to serve as effective co-catalysts when coupled with semiconductor photocatalysts, thereby enhancing light absorption and charge transfer processes [9–11].

In this study, a g-C₃N₄-based composite photocatalyst loaded with gold and platinum nanoparticles was synthesized, and its photocatalytic activity was evaluated through the photocatalytic reduction of CO₂. g-C₃N₄ was first prepared via a thermal polymerization method and used as the support material. Subsequently, Au and Pt nanoparticles were deposited onto the surface of g-C₃N₄ through a photodeposition process. The results demonstrate that noble metal loading significantly enhances the photocatalytic performance of g-C₃N₄ for CO₂ reduction. Furthermore, the synthesized composite was comprehensively characterized using various analytical techniques.

2. Materials and Methods

The raw materials used in this study included dicyandiamide and HAuCl₄, which were purchased from Sigma-Aldrich. These materials were used directly in the experiments without any further pretreatment. Triethanolamine (TEOA) and platinum chloride were obtained from Merck. Double-distilled water was used as the solvent throughout the process. To synthesize graphitic carbon nitride sheets, 50 mL of water was first poured into a beaker, and then 20 g of dicyandiamide was added. The mixture was heated and stirred on a magnetic stirrer until complete dissolution was achieved. Subsequently, the sample was placed in a drying oven and dried at 80 °C. The obtained product was then heated at 550 °C for 3 h.

The resulting material consisted of yellow bulk graphitic carbon nitride.

The product was then ground, and in order to increase the specific surface area and induce exfoliation, it was subjected again to thermal treatment using a surface oxidation method. In this step, the furnace temperature was set to 460 °C, and the treatment time was 2 h. The synthesis steps are illustrated in Fig. 1. After the synthesis of graphitic carbon nitride sheets, gold and platinum nanoparticles were deposited on

their surface via a photodeposition process, as described below. First, gold and platinum salts were dissolved in water, and then a certain amount of graphitic carbon nitride powder was added to the solution. Subsequently, 1 wt% of TEOA was added to the mixture, and the system was evacuated. The mixture was then exposed to simulated solar light to allow the photodeposition process to take place.

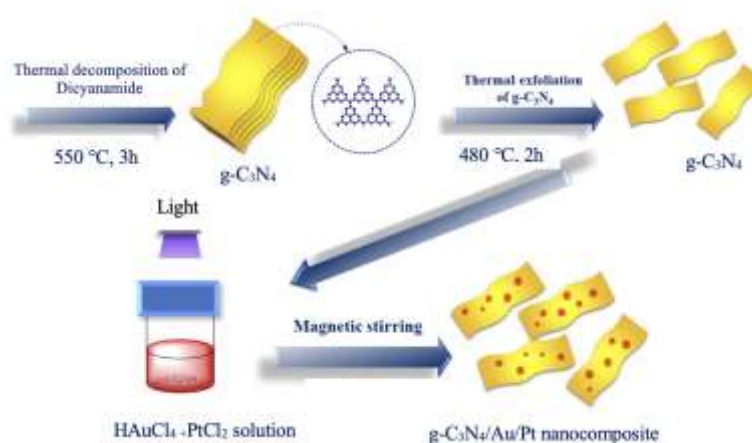


Fig. 1. Synthesis steps of the g-C₃N₄/Au/Pt nanocomposite

To investigate the crystalline structure of the synthesized phases, X-ray diffraction (XRD) analysis was performed using a Philips X'Pert diffractometer. A copper cathode lamp with a wavelength of 1.54059 Å was employed. The crystallite size was calculated from the XRD patterns using the Scherrer equation (Eq. 1).

$$d = \frac{0.9\lambda}{\beta \cdot \cos\theta} \quad (1)$$

In this equation, θ is the diffraction angle, d is the crystallite size, λ is the wavelength of the X-ray source, and β is the full width at half maximum (FWHM) of the diffraction peak. The microstructure of the samples was examined using a transmission electron microscope (TEM, JEOL JEM 1230 operated at 120 kV). Diffuse reflectance UV–Vis spectroscopy (Shimadzu UV-3600 UV–Vis–NIR) was employed to investigate the optical properties of

the synthesized materials. The specific surface area (BET) and pore size (BJH) was measured using a NOVA 2000 nitrogen adsorption analyzer. FTIR spectra of the catalysts were recorded using a Nicolet iS50 FTIR spectrometer. XPS measurements were carried out using a Physical Electronics ESCA 5800 spectrometer. Carbon was used for peak calibration to compensate for surface charging effects.

Photocatalytic CO₂ reduction experiments were performed in a quartz reactor (Fig. 2). In the photocatalytic CO₂ reduction system, 5 mg of catalyst was dispersed in 4 mL of deionized water. Prior to light irradiation, the system was thoroughly purged with CO₂ to remove air from the reactor. During the reaction, the CO₂ pressure was maintained at 0.4 MPa, and the reaction temperature was controlled at 80 °C. After 3 h of light irradiation, the gaseous products were analyzed using a SRI 8610C gas chromatograph.

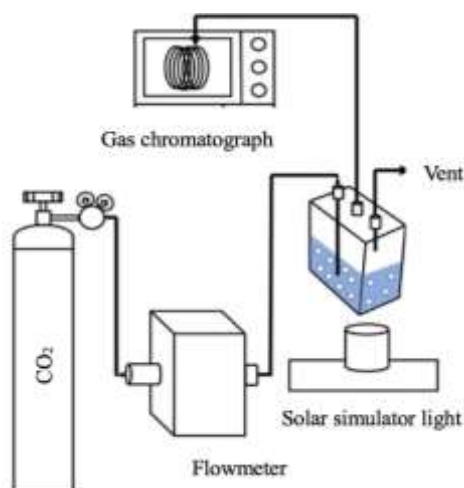


Fig. 2. Photocatalytic CO₂ reduction reactor

3. Results and Discussion

The surface chemical composition of the synthesized sample was determined by XPS analysis, as shown in Fig. 3. The results confirm the presence of gold, platinum, carbon, and nitrogen in the samples. Figure 3a shows the C 1s spectrum. As can be seen, two peaks located at 288.1 eV and 284.6 eV are observed, which originate from sp²-hybridized carbon (N–C=N) and C–C bonds in the g-C₃N₄ structure, respectively.

The N 1s spectrum is presented in Fig. 3b. Peaks at approximately 398.5 eV, 399.4 eV, and 401.0 eV correspond to sp²-hybridized nitrogen (C=N–C) in tri-s-triazine rings, sp³-hybridized nitrogen (C₃–N) associated with the edge sites of the carbon nitride structure, and amino nitrogen (C–N–H), respectively [12,13].

Figure 3c displays the Au-related peaks. As observed, gold exists in the metallic state in the samples, as indicated by the Au 4f_{7/2} (83.2 eV) and

Au 4f_{5/2} (86.9 eV) peaks [14]. Figure 3d shows the Pt 4f spectrum of the sample. The binding energy peaks at 71.1 eV and 74.5 eV can be assigned to Pt 4f_{7/2} and Pt 4f_{5/2} of metallic platinum, respectively, while the minor peaks at 72.7 eV and 76.1 eV originate from Pt 4f_{7/2} and Pt 4f_{5/2} of oxidized platinum species [15,16]. The presence of oxidized platinum species is likely due to the oxidation of metallic platinum upon exposure to air. Nevertheless, metallic platinum is the dominant species in the samples. It has been reported that the coexistence of platinum oxide and metallic platinum can enhance photocatalytic activity. Compared with XPS data of pure gold and platinum, the Au and Pt peaks in the composite slightly shifted toward lower binding energies, indicating a strong interaction between these metals and g-C₃N₄. This phenomenon suggests that gold and platinum metals possess a higher negative charge in the composite structure.

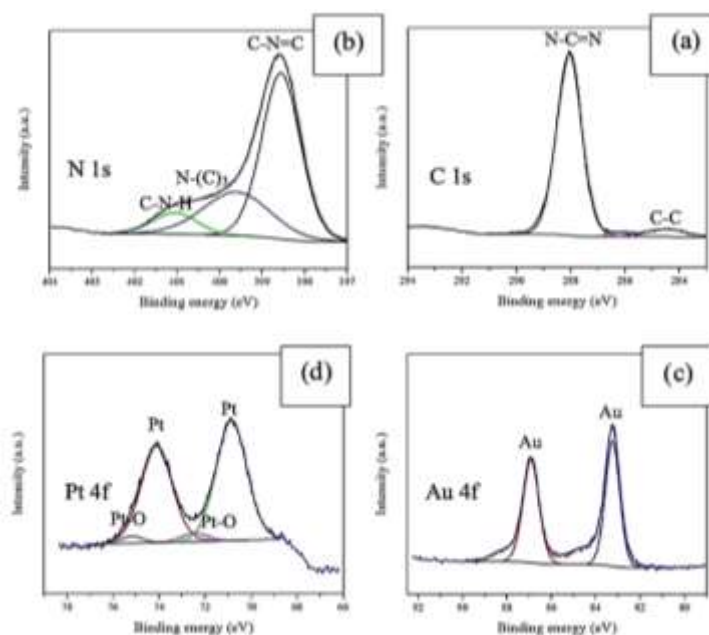


Fig. 3. XPS spectra of the g-C₃N₄/Au/Pt sample

The XRD patterns of the samples are shown in Fig. 4a. As observed, pristine $g\text{-C}_3\text{N}_4$ exhibits two diffraction peaks at approximately 13.1° and 27.2° , confirming the formation of this phase. These peaks are characteristic of the typical graphitic carbon nitride structure. The crystallite size was estimated to be about 6 nm using the Scherrer method. Figure 4b illustrates the chemical reaction steps involved in the formation of this phase. Dicyandiamide is first condensed into melamine, followed by pyrolysis and polymerization to form carbon nitride. After loading the metal nanoparticles, diffraction peaks corresponding to gold appear, whereas no

platinum-related peaks are observed, which is likely due to the amorphous nature of platinum. In addition, the intensity of the carbon nitride peaks decreases after metal loading. This reduction in peak intensity is attributed to the formation of a surface coating, which diminishes the diffraction contribution from the underlying phase. Although the presence of platinum was not confirmed by XRD analysis, XPS results verified the existence of this phase. Furthermore, no peak shift was observed after nanoparticle loading, indicating that the secondary phases did not incorporate into the carbon nitride lattice.

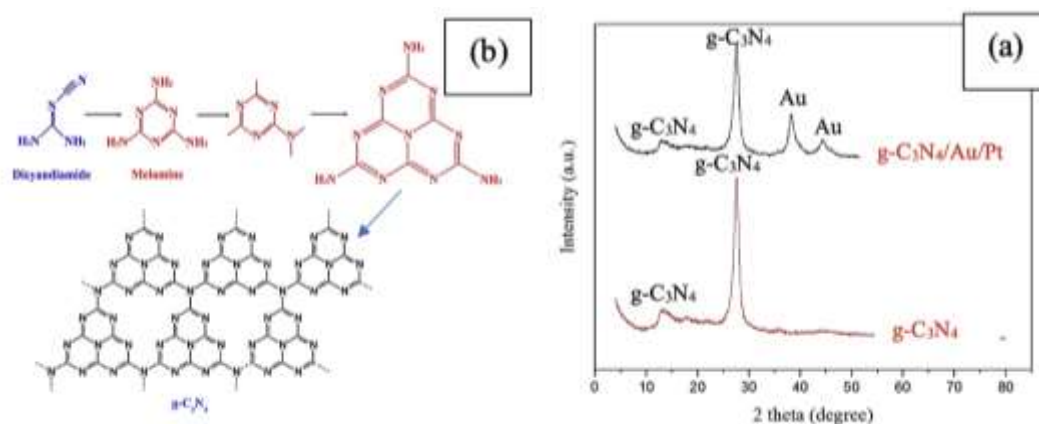


Fig. 4. (a) XRD patterns of $g\text{-C}_3\text{N}_4$ and $g\text{-C}_3\text{N}_4/\text{Au/Pt}$ samples, and (b) formation mechanism of $g\text{-C}_3\text{N}_4$ from the cyanamide precursor

FTIR analyses were performed on the pristine and composite samples to identify the chemical bonds and functional groups (Fig. 5). As observed, a broad absorption band in the range of $3000\text{--}3500\text{ cm}^{-1}$ corresponds to N–H stretching vibrations. The peaks located in the wavenumber range of $1200\text{--}1700\text{ cm}^{-1}$ are attributed to C=N and C–N stretching vibrations in the heterocyclic rings, as well as C–N stretching vibrations outside the ring structure. The peak appearing at 1460 cm^{-1} is assigned to the triazine ring

system associated with terminal NH groups. The absorption band at 812 cm^{-1} is characteristic of the s-triazine ring vibration [17,18].

The FTIR spectrum of the composite sample is similar to that of pristine carbon nitride, since metals generally do not exhibit distinct features in FTIR analysis. Moreover, the absence of noticeable peak shifts indicates that the deposition of metal nanoparticles did not adversely affect the carbon nitride structure.

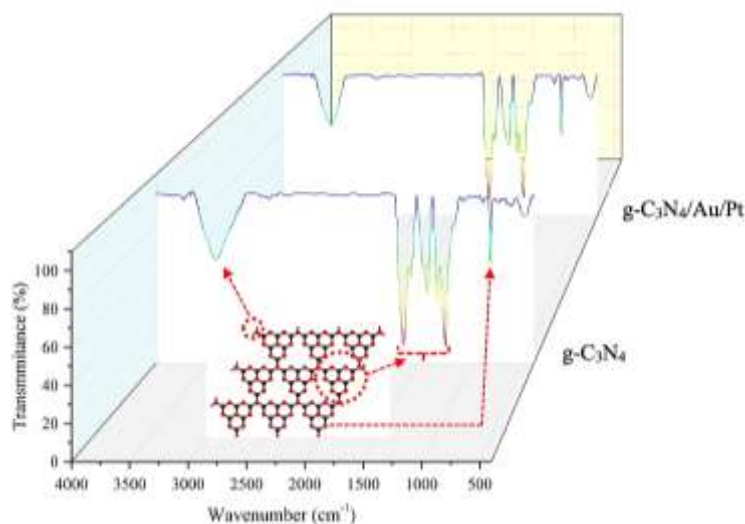


Fig. 5. FTIR spectra of (a) $g\text{-C}_3\text{N}_4$ and $g\text{-C}_3\text{N}_4/\text{Au/Pt}$ samples

The specific surface area and pore size distribution of the samples are presented in Fig. 6. The BET specific surface area of the samples was determined from the adsorption–desorption isotherms. The results show that by adding metal nanoparticles to the surface of carbon nitride, the specific surface area of the samples increased by more than six times, rising from 15 to 96 m² g⁻¹. It appears that the synthesis method plays an important role in the surface characteristics of this composite and may significantly influence its surface properties. The increase in the overall specific surface area of the composite is likely due to the high specific surface area of the nanoparticles after deposition. It has been reported that a large specific surface area can effectively enhance the adsorption, desorption, and diffusion of reactants as a result of the increased number of active catalytic sites.

Figure 6b shows the pore size distribution of the samples, calculated using the BJH method. All samples exhibit a broad pore size distribution ranging from 10 to 50 nm, which falls within the mesoporous range. The presence of hysteresis loops in Fig. 6a further confirms the existence of a mesoporous structure [19,20]. The smaller pores are likely associated with the surface of g-C₃N₄ nanosheets and other nanoparticles, while the larger pores correspond to the spaces between randomly stacked carbon nitride layers. These pores may provide effective pathways for reactant transport and adsorption, thereby enhancing gas reduction performance. Furthermore, as observed after noble metal loading, the average pore size decreases, indicating the deposition of nanoparticles within the pores and interlayer spaces.

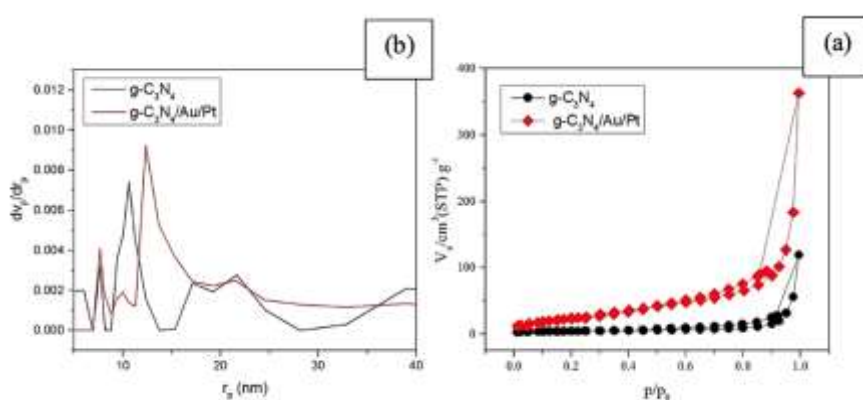


Fig. 6. (a) BET and (b) BJH results of the g-C₃N₄/Au/Pt sample

To investigate the structural features of carbon nitride coated with gold and platinum, TEM images were obtained from this sample, and the result is shown in Fig. 7a. In this image, the layered structure of carbon nitride is clearly observed. Dark spherical particles with sizes of approximately 30 nm are visible on the surface of the samples, which correspond to the deposited metal nanoparticles and indicate successful deposition. In fact, since the atomic numbers of gold and platinum are higher than those of the constituent elements of carbon nitride, the electron beam is scattered more strongly, causing these nanoparticles to appear darker in the TEM images. This figure also shows that the particles are relatively uniformly distributed on the surface and that no significant aggregation of metal nanoparticles has occurred, which facilitates improved electron transfer between the nanoparticles and carbon nitride.

The formation mechanism of these nanoparticles during the coating process can be described as follows: when carbon nitride is placed in an environment containing platinum and gold ions and irradiated with photons, electron–hole pairs are generated on its surface. The photogenerated electrons can then transfer to the metal ions and

reduce them, leading to the deposition of metallic species on the surface. It has been reported that the size of these nanoparticles can extend the light absorption range into the visible region [11,21].

To further examine the effect of these nanoparticles on the optical properties of carbon nitride, UV–Vis spectroscopy was employed, and the results are shown in Fig. 7b. As observed, both the coated and uncoated samples exhibit an absorption edge in the visible-light region. After metal nanoparticle deposition, no change in the absorption edge is observed, confirming that the coating process does not affect the structure of carbon nitride and, consequently, does not alter its electronic structure. However, a new absorption region appears in the visible range of approximately 500–650 nm. This absorption is attributed to the plasmonic resonance effect. This phenomenon enhances the visible-light absorption range of the prepared photocatalyst, thereby improving its efficiency under solar irradiation. As is well known, many commonly used photocatalysts, such as titanium dioxide and zinc oxide, are active only in the ultraviolet region, which is a major limitation of these materials.

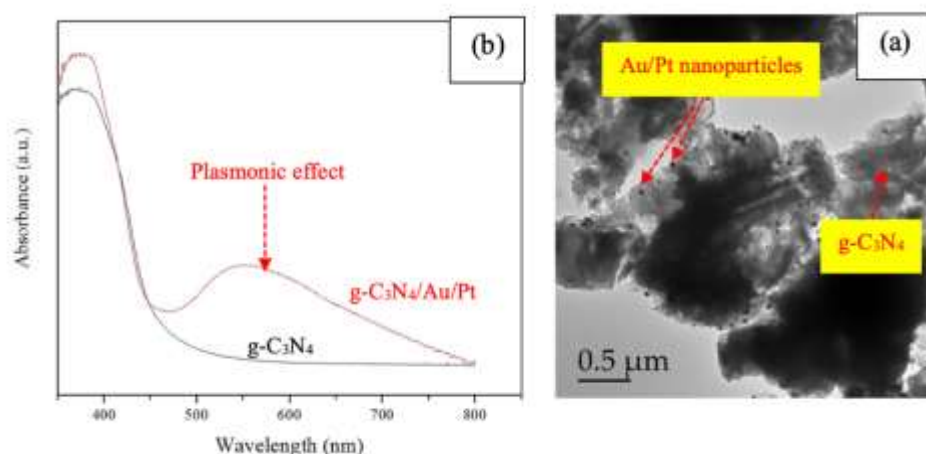


Fig. 7. (a) TEM and (b) UV-Vis spectra of the g-C₃N₄/Au/Pt sample

The photocatalytic CO₂ reduction performance of the samples was evaluated under simulated solar light irradiation (Fig. 8a). The produced product was CO. Control experiments confirmed that no hydrocarbon products were detected in the absence of light or catalyst. As observed, the amount of CO produced over pristine carbon nitride was approximately 9.7 μmol, whereas the composite sample generated about 33 μmol of reduced CO₂.

In general, photocatalytic reactions can be divided into three main processes. First, the photocatalysts absorb effective photons with energies equal to or higher than their band gaps, leading to the generation of electron-hole pairs. Second, the photogenerated charge carriers are separated and transferred to the surface of the photocatalysts. Third, the generated electrons and holes participate in reactions involving adsorbed species on the surface of the samples. The photocatalytic reaction mechanism of the synthesized samples in this study is illustrated in Fig. 8b. When carbon nitride is irradiated with light, electrons in the valence band gain sufficient energy to be excited to

the conduction band, leaving holes in the valence band. As observed, the conduction band potential of carbon nitride is negative; therefore, it is capable of reducing carbon dioxide. In addition, gold nanoparticles generate electrons and inject them into graphitic carbon nitride, thereby reducing electron-hole recombination. Platinum also acts as a cocatalyst by providing suitable reaction sites. The adsorption of CO₂ on the photocatalyst surface is a prerequisite for the photocatalytic reduction of CO₂. It has been calculated that CO₂ molecules are adsorbed on the g-C₃N₄ surface with a maximum adsorption energy of -0.4181 eV, indicating typical physical adsorption. Moreover, it has been reported that g-C₃N₄ exhibits higher adsorption energies for CO₂ and H₂O compared to H₂, CO, and CH₄ [22]. As a result, the reduction products can be readily released from the active sites. The higher specific surface area of the composite sample compared to pristine carbon nitride is another reason for the enhanced photocatalytic activity of this sample.

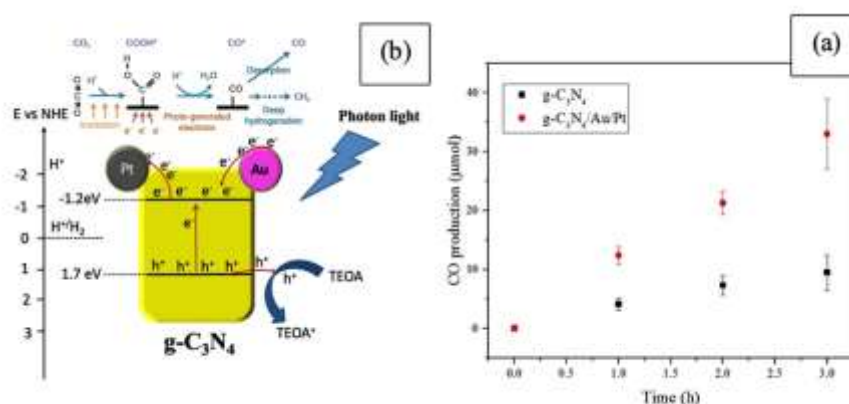


Fig. 8. (a) Photocatalytic CO production via CO₂ reduction using the g-C₃N₄/Au/Pt sample, and (b) photocatalytic mechanism of the g-C₃N₄/Au/Pt sample

4. Conclusion

In this study, carbon nitride, which is a visible-light-responsive semiconductor, was synthesized via a thermal decomposition method and subsequently composited with gold and platinum nanoparticles. The photodeposition method was employed to deposit the metal nanoparticles onto the carbon nitride surface. The gold- and platinum-loaded carbon nitride was characterized using various techniques, including XRD, XPS, TEM, BET, and UV-Vis spectroscopy. The results indicated that gold and platinum nanoparticles were successfully formed on the surface of carbon nitride, and the reduction of metal ions through the photodeposition process was effectively achieved. TEM images showed that metal nanoparticles with sizes of approximately 30 nm were formed on the carbon nitride surface. The specific surface area of the samples increased after the deposition of gold and platinum nanoparticles. In addition, the incorporation of metal nanoparticles extended the light absorption wavelength range, which is likely attributed to the plasmonic resonance effect. The photocatalytic properties of the samples were evaluated through the photocatalytic reduction of carbon dioxide. The results demonstrated that the photocatalytic performance was improved by the addition of metal nanoparticles. The amount of CO produced over pristine carbon nitride was approximately 9.7 μmol , whereas the composite sample generated about 33 μmol of reduced CO_2 .

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