

REVIEW ARTICLE

Morphology engineering of graphitic carbon nitride for enhanced photocatalytic performance: A mini-review

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Abstract

Graphitic carbon nitride ($g\text{-C}_3\text{N}_4$) has emerged as a promising visible-light-driven photocatalyst for environmental remediation and energy conversion due to its suitable bandgap, high stability, and low cost. However, bulk $g\text{-C}_3\text{N}_4$ prepared by conventional thermal polymerization suffers from low specific surface area, limited active sites, and severe charge recombination. To address these limitations, various morphology engineering strategies have been developed. This review systematically summarizes the preparation methods of $g\text{-C}_3\text{N}_4$ with different morphologies, including ultrathin nanosheets via thermal oxidation etching, liquid-phase ultrasonic exfoliation, and chemical exfoliation; mesoporous structures using hard templating; tubular structures through molecular self-assembly; as well as spherical, rod-like, and quantum dot morphologies. These morphological modifications effectively increase the specific surface area, shorten charge migration pathways, and provide more reactive sites, thereby significantly enhancing photocatalytic hydrogen evolution and pollutant degradation. This review aims to offer guidance for designing high-performance $g\text{-C}_3\text{N}_4$ photocatalysts.

Keywords: Photocatalysis; Graphitic carbon nitride; Morphology engineering; Ultrathin nanosheets; Mesoporous structures

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1. Introduction

Photocatalytic technology, as a green and sustainable approach for environmental remediation and energy conversion, has garnered widespread attention in recent years.¹ Its core principle is based on the energy band structure of semiconductor materials: when the incident light energy is greater than or equal to the semiconductor bandgap (E_g), electrons in the valence band (VB) are excited and transition to the conduction band (CB), leaving behind holes in the VB, thereby forming electron-hole pairs.² These photogenerated charge carriers migrate to the material surface, participate in redox reactions, generate reactive oxygen species (ROS), or drive processes, e.g., H_2 evolution and CO_2 reduction.^{3,4} The positions of the E_{CB} and E_{VB} of a semiconductor determine its reduction and oxidation capabilities, while parameters, e.g., Fermi level (E_f) and work function (W_s), influence the built-in electric field strength of heterojunctions and the charge carrier separation efficiency.⁵

Among the numerous semiconductor photocatalysts, graphitic carbon nitride (g-C₃N₄) stands out due to its unique advantages.⁶ As an organic polymeric semiconductor composed of carbon and nitrogen, g-C₃N₄ features abundant earth reserves, low synthesis cost, and excellent thermal and chemical stability.⁷ Its energy band structure enables direct utilization of visible light, and its photogenerated electrons and holes possess high redox potentials, presenting broad application prospects in environmental remediation and energy fields.^{8,9} However, bulk g-C₃N₄ prepared by traditional thermal polymerization suffers from low specific surface area, insufficient active sites, and severe photogenerated carrier recombination, which limit its photocatalytic efficiency.¹⁰⁻¹²

To overcome these drawbacks, researchers have developed various morphology control strategies.^{13,14} This article systematically reviews the preparation methods of different morphologies of g-C₃N₄ and their effects on photocatalytic performance, including ultrathin nanosheets (via thermal oxidation etching, liquid-phase ultrasonic exfoliation, and chemical exfoliation), mesoporous structures (hard templating), tubular structures (molecular self-assembly), as well as other morphologies, *e.g.*, spherical, rod-like, and quantum dots. By tuning the morphology, one can effectively increase the specific surface area, shorten charge carrier migration pathways, and provide more reactive active sites, thereby significantly enhancing the efficiency of photocatalytic hydrogen production and pollutant degradation. This review aims to provide a reference for the design and development of high-performance g-C₃N₄ photocatalysts.

2. Basic principle of photocatalytic technology

In general, the photocatalysis technology is based on semiconductor, although the precious metals with the surface plasmon resonance (SPR) effect, *e.g.*, gold (Au) and organic molecules have been reported to be available for photocatalytic H₂ production and CO₂ reduction.¹⁵⁻¹⁷ The energy bands of semiconductors (Figure 1) formed by the splitting of energy levels with similar energies are discontinuous, where a E_g consisting of conduction band (CB) and valence band (VB) exists.¹⁸ When a semiconductor absorbs photons with energy equal to or greater than its E_g, electrons are excited from the VB to the CB, leaving holes in the VB. The number of photoexcited electrons equals that of holes, forming electron-hole pairs.¹⁹ The separation of electron-hole pairs formed electrons and holes, which migrated to the surface of semiconductor for participating in the subsequent redox reaction. It should be noted that not all photogenerated carriers reach the

surface, recombination can occur in the bulk or at surface defects, which is a key limitation for semiconductor.²⁰

The reduction potential of CB and the oxidation potential of VB determine whether electrons can reduce O₂ and holes can oxidize H₂O to ROS.^{21,22} For ·O₂⁻ formation: the CB edge potential must be more negative than O₂/O₂⁻ (-0.33 eV vs. NHE). For ·OH formation: the VB edge potential must be more positive than OH⁻/·OH (2.40 eV vs. NHE) or H₂O/·OH (2.72 eV vs. NHE), the holes on the VB can oxidize OH⁻ or H₂O to ·OH and H₂O₂. In addition to E_{CB}, E_{VB} and E_g, the energy band structures of semiconductor also include E_f and W_s. The energy level corresponding to E_f has a 1/2 probability of being occupied by electrons, and the W_s is the difference between vacuum energy level (E_{vac}) and E_f.²³ There are three types of semiconductors, *i.e.*, intrinsic, n-type and p-type. The E_f of intrinsic, n-type and p-type semiconductor is located in the middle of E_g, near the E_{CB} and near the E_{VB}, respectively.²⁴ With regard to the composites composed of semiconductor and metal, metallic compound or other semiconductor, their E_f and W_s affect the strength of the built-in electric field, and thus affect the separation efficiency of electron-hole pairs.

2.1. The semiconductor g-C₃N₄

Recently, the g-C₃N₄ as a crystalline all-organic middle band gap semiconductor has attracted extensive attention in the field of environmental remediation and energy fields.²⁵ The g-C₃N₄ is mainly composed of two elements, *i.e.*, C and N, which are abundant on the Earth. The organic chemistry nature means that the cost required to prepare g-C₃N₄ is relatively low, and its properties can be simply adjusted without changing the overall composition too much.²⁶

2.1.1. Energy band structures of g-C₃N₄

The basic structural units of g-C₃N₄ are triazine and heptazine (tri-s-triazine) rings (Figure 2a and 2b).²⁷ Density Functional Theory (DFT) calculations showed that the heptazine-based structure is ~30 kJ/mol more stable than the triazine-based one.²⁸ Experimental evidence confirmed that pyrolysis of cyanamide, dicyandiamide, urea, or melamine yields a melon polymer with heptazine units.^{29,30} These units stack along the z-axis via van der Waals forces and possible hydrogen bonding (due to defects and residual H atoms), forming a graphite-like interlayer structure with an interlayer distance (d_s) of ~0.327 nm. Higher thermal condensation temperatures reduce defects and lead to tighter stacking, while nanostructures or porous g-C₃N₄ may exhibit slightly decreased d_s.^{31,32}

2.1.2. Structural characteristics of g-C₃N₄

The g-C₃N₄ possesses high thermal and chemical stability.

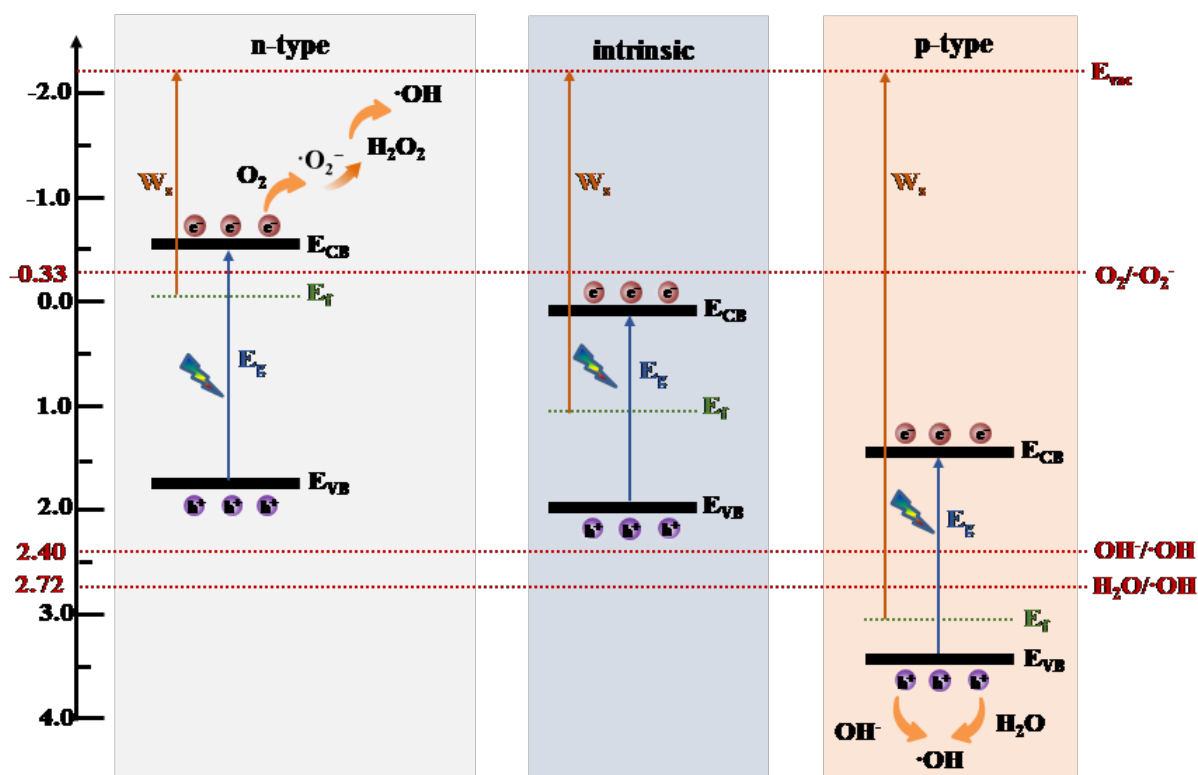


Figure 1. The energy band structure of n-type, intrinsic and p-type semiconductors. We assumed that the $E_{CB} \geq O_2/\cdot O_2^-$ (-0.33 eV vs. NHE) of a n-type semiconductor, and the $E_{VB} \geq OH^-/\cdot OH$ (2.40 eV vs. NHE) and $H_2O/\cdot OH$ (2.72 eV vs. NHE) of a p-type semiconductor.

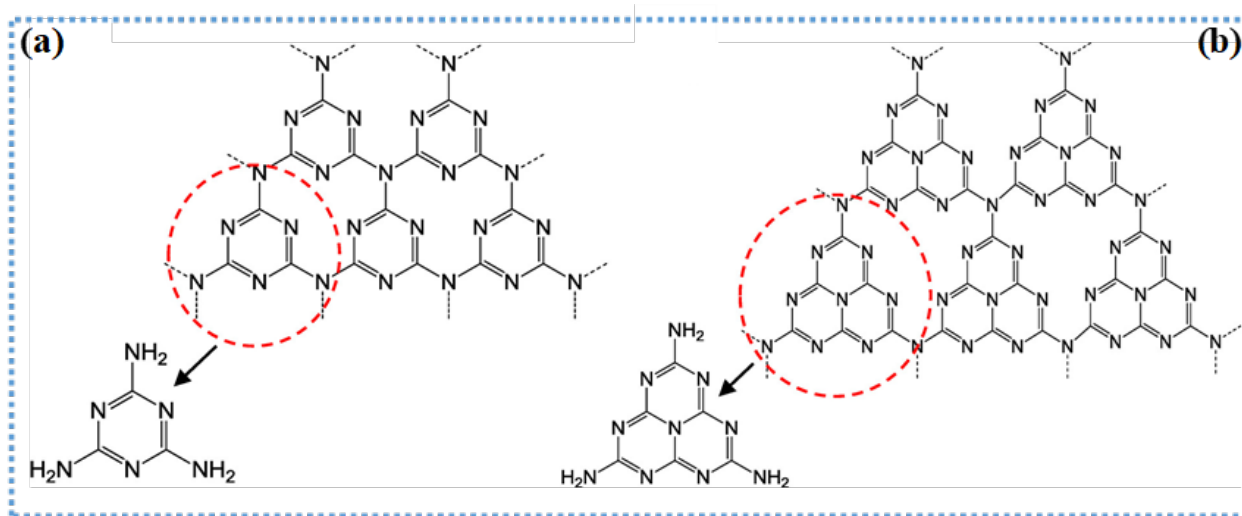


Figure 2. The basic structural units: (a) triazine and (b) tri-s-triazine (heptazine) structures of g-C₃N₄. Both panels are reproduced with permission from Ong *et al.*²⁷ Copyright © 2016 American Chemical Society.

Thermogravimetric analysis showed no significant volatilization up to 600 °C in air, with decomposition starting at 630 °C.^{33,34} It is insoluble in most solvents (water, acetone, ethanol, N,N-dimethylformamide [DMF], dilute NaOH, *etc.*); Fourier transform infrared spectroscopy (FT-IR) spectra remain unchanged after 30 days of soaking.^{35,36} As a mid-bandgap semiconductor, g-C₃N₄ exhibits $E_g = 2.7$ eV, $E_{CB} = -1.1$ eV and $E_{VB} = +1.6$ eV, enabling direct visible-light utilization and high redox potentials.³⁷ It was not until 2006 that Goettmann *et al.*³⁸ first adopted g-C₃N₄ as a non-metallic catalyst to activate benzene, avoiding the use of metal chlorides and halogenated compounds. In 2009, Wang *et al.*³⁹ manifested that g-C₃N₄ could decompose water into H₂ under visible light irradiation in the presence of sacrificial donor. However, bulk g-C₃N₄ prepared by direct thermal polymerization has small specific surface area (SB_{ET}) (<10 m² g⁻¹), limited active sites, and severe electron-hole recombination due to long interlayer migration paths and strong Coulomb interactions.⁴⁰⁻⁴²

3. Preparation methods of g-C₃N₄ with different morphologies

In recent years, researchers have employed a variety of methods to prepare the g-C₃N₄ with a specific morphology, *e.g.*, the ultrathin g-C₃N₄ nanosheets⁴³, the monatomic layer mesoporous g-C₃N₄ nanonets⁴⁴, the g-C₃N₄ quantum dots⁴⁵, the ball g-C₃N₄⁴⁶, and hollow tubular g-C₃N₄⁴⁷ for improving the photocatalytic performance of monomer bulk g-C₃N₄.

3.1.1. Thermal oxidation etching method

The thermal oxidation etching is a simple top-down strategy, which has the advantages of low cost, easy scale and environmental friendliness. The preparation process is as follows⁴⁸: The calcination of as-synthesized bulk g-C₃N₄ is conducted in a muffle furnace (500~550 °C). If the stability of the van der Waals force or hydrogen bonding in the interlayers of bulk g-C₃N₄ is not enough to resist the high temperature oxidation process in muffle furnace, the outer layers and the bare inner layers of the bulk g-C₃N₄ would gradually peel off. With the process of oxidation, the thickness of the bulk g-C₃N₄ decreased continuously and the ultrathin g-C₃N₄ nanosheets are generated. The ultrathin g-C₃N₄ nanosheets with a thickness of ~2 nm was successfully prepared by the thermal oxidation etching method. The large SB_{ET} (306 m² g⁻¹) of the ultrathin g-C₃N₄ nanosheets could provide abundant reactive sites. In addition, the ultrathin thickness could shorten the path length of electrons and holes from the interior to the surface, reducing the probability of their recombination. Furthermore, the larger E_g ($E_g = 2.97$ eV) of the ultrathin

g-C₃N₄ nanosheets, indicating the redox potential of electrons and holes are stronger as compared to bulk g-C₃N₄. Therefore, the ultrathin g-C₃N₄ nanosheets showed higher photocatalytic performance in producing ·OH and H₂ than that of the bulk g-C₃N₄. It cannot be ignored that the reaction conditions, such as heating rate, calcination temperature, and holding time, should be reasonably controlled during the thermal oxidation etching process to avoid the insufficient reaction produce g-C₃N₄ with a large thickness or the reaction is too intense, the bulk g-C₃N₄ completely oxidized without product formation.

3.1.2. Liquid phase ultrasonic stripping method

The liquid phase ultrasonic stripping is usually conducted by dispersing the bulk g-C₃N₄ in various solvents, such as isopropyl alcohol (IPA), N-methylpyrrolidone (NMP), water, ethanol, and acetone. Subsequently, the dispersion solution was ultrasonic-treated at room temperature.⁴⁹ By controlling the ultrasonic time and selecting an instrument with the appropriate power, the bulk g-C₃N₄ was gradually stripped to the ultrathin g-C₃N₄ nanosheets.⁵⁰ By using this method, the layered material is stripped into nanosheets through the interaction of solvent molecules, which is affected by the surface energy of the solvent. If the surface energy of the bulk g-C₃N₄ matches that of the solvent, the bulk g-C₃N₄ can be stripped into nanosheets and the enthalpy of the mixture (ΔH_{mix}) is the minimum.⁵¹ Zhang *et al.*⁵² reported that the relationship between ΔH_{mix} and the surface energy is shown in the following formula:

$$\frac{\Delta H_{mix}}{V_{mix}} = \frac{2}{T_{bulk}} (\delta_G - \delta_{sol})^2 \varnothing \quad (1)$$

where δ_G and δ_{sol} are respectively the square root of g-C₃N₄ and solvent' surface energy, T_{bulk} represents the average thickness of bulk g-C₃N₄, and $\varnothing$ is the volume proportion of g-C₃N₄. They calculated the surface energy of g-C₃N₄ is ~115 mJ m⁻² by using the DFT calculations, which matched that of water (at ~102 mJ m⁻²) very well. The results demonstrated that water has the excellent dispersion ability for the stripping of bulk g-C₃N₄. Meanwhile, they prepared the ultrathin g-C₃N₄ nanosheets with a thickness of ~2.5 nm for the first time by the liquid phase ultrasonic stripping method using water as the dispersion solution. Coleman *et al.*⁵³ showed that the optimal surface energy of solvent were 30 mJ m⁻² larger than that of the van der Waals bonding surface (70 mJ m⁻²). The surface energies of IPA and NMP are ~40 mJ m⁻², which indicated that both of them can be used as the dispersion solution for the stripping of bulk g-C₃N₄. It was reported that the ultrathin g-C₃N₄ nanosheets with a thickness of ~2 nm and the high S_{BET} was successfully prepared by using IPA as the dispersion

solution.⁵⁴ The ultrathin thickness and high S_{BET} not only provided more reactive sites, but facilitated the separation and migration of electrons and holes. The as-prepared g-C₃N₄ nanosheets exhibited average H₂ production rate of 93 $\mu\text{mol h}^{-1}$ under visible light irradiation.

Besides, Han *et al.*⁵⁵ synthesized the ultrathin g-C₃N₄ nanosheets with monatomic layer by adopting freeze-dried self-assembled dicyandiamide as a precursor, water and IPA as solvents. Due to the unique structural advantages in energy level arrangement, electrons and holes transfer, light absorption and reaction sites, the monatomic layer mesoporous g-C₃N₄ nanonets prepared by the liquid phase ultrasonic stripping method have the excellent photocatalytic H₂ evolution performance. The photocatalytic H₂ production rate is as high as 8510 $\mu\text{mol h}^{-1} \text{g}^{-1}$ and the apparent quantum efficiency is 5.1% under visible light irradiation ($\lambda > 420 \text{ nm}$), which is the highest among the reported g-C₃N₄ nanosheets. It manifested that the liquid phase ultrasonic stripping method is simple, mild, environmentally friendly (solvent recyclable) and is capable of preparing the high-quality 2D materials on a large scale.

The ultrathin g-C₃N₄ nanosheets prepared by the thermal oxidation etching and liquid phase ultrasonic stripping method revealed the higher photocatalytic H₂ evolution and organic pollutants degradation performance as compared to the direct thermal polymerization process prepared bulk g-C₃N₄.⁵⁶⁻⁵⁸ The theoretical thickness of the monatomic layer g-C₃N₄ is 0.327 nm, meaning that these ultrathin g-C₃N₄ nanosheets with a thickness of 2~3 nm contain 6~9 monatomic layers.⁵⁹ It can then be expected that the ultrathin g-C₃N₄ nanosheets with fewer atomic layers might have the higher photocatalytic performance. The two-step method, involving liquid-phase ultrasonic stripping in water as a solvent followed by thermal oxidation etching, produced ultrathin g-C₃N₄ nanosheets with a thickness of approximately 1 nm (ca. three atomic layers), which showed efficient photocatalytic H₂ production performance.⁶⁰

3.1.3. Chemical stripping method

The chemical stripping is another frequently-used method for the preparation of the ultrathin g-C₃N₄ nanosheets.⁶¹ Xu *et al.*⁶² prepared the monatomic layer ultrathin g-C₃N₄ nanosheets by the chemical stripping method for the first time. Specifically, 1 g bulk g-C₃N₄ was mixed with 10 mL of concentrated H₂SO₄ (98 wt%) in a 50 mL flask at room temperature for 8 h. Then, the mixture was slowly poured into a beaker filled with 100 mL of water for ultrasonic stripping. The resulting suspension was centrifuged at a rate of 3000 rpm/min for 10 min to

separate the large particles of g-C₃N₄. The monatomic layer ultrathin g-C₃N₄ nanosheets was obtained by further centrifugation and water washing the luminous yellow suspension to remove the residual H₂SO₄ and drying in an oven at 80 °C for 12 h. The ultrathin g-C₃N₄ nanosheets with a thickness of 0.4 nm have great advantages over the bulk g-C₃N₄ in the separation of electrons and holes, the intensity of photocurrent, the photocatalytic H₂ generation and organic pollutants degradation performance.

3.2. Preparation of mesoporous g-C₃N₄

The hard template method is able to prepare the ordered mesoporous g-C₃N₄ with the high S_{BET} , small particle size, adjustable pore size and controllable morphology by replicating the uniform pore template structure.⁶³

3.2.1. Hard template 2D mesoporous silica nanoparticles

Two-dimensional (2D) mesoporous silica nanoparticles, such as SBA-15 and IBN-4, are hard templates that are commonly adopted for the preparation of mesoporous g-C₃N₄.⁶⁴ Vinu *et al.*⁶⁵ successfully prepared the 2D hexagonal ordered mesoporous g-C₃N₄ with the adjustable S_{BET} pore size and pore volume for the first time through a simple polymerization reaction of the precursor carbon tetrachloride and ethylenediamine using SBA-15 with the different pore sizes as the hard template. However, the mesoporous g-C₃N₄ prepared with SBA-15 has the low nitrogen content and large particle size due to the poor thermal stability. Subsequently, Jin *et al.*⁶⁶ adopted the ultrafine mesoporous silica nanoparticles (IBN-4) as a hard template to prepare mesoporous g-C₃N₄ with a particle size less than 150 nm and the high nitrogen content. It is worth noting that the presence of air in the silica template pore greatly hinders the diffusion of organic precursor of g-C₃N₄ synthesis, completely obstructing penetration of the precursor into the silica template pore. Moreover, water or other solvents contained in the raw material might compete with the absorption of precursor on the template. Therefore, the synthesized mesoporous g-C₃N₄ has a certain degree of structural defects or volume collapse after the removal of template.

In view of that, Zhang *et al.*⁶⁷ proposed an optimized synthesis method aimed at effectively removing the previously trapped air from the template to accelerate the diffusion of organic precursors and avoid competitive solvent-induced absorption. Specifically, the SBA-15 was first acidified with 1 M dilute HCL to enhance the surface reactivity on cyanamide containing amido through acid-base interaction. Then, the air in the SBA-15 channel was removed by a technology combining ultrasonic

and vacuum that utilizes relatively low saturated vapor pressure of liquid cyanamide. Besides, the cyanamide was simultaneously pumped into the SBA-15 channel via the bubble collapse effect. Accordingly, the precursor cyanamide could permeate into the SBA-15 channel completely. The g-C₃N₄ prepared with SBA-15 as the hard template using this optimization method has a 2D mesoporous hexagonal skeleton, a larger S_{BET} and a higher photocatalytic H₂ production performance.

3.2.2. Hard template 3D cage mesoporous silica nanoparticles

It should not be ignored that due to the inherent porous structure limitations of the hard template 2D mesoporous silica nanoparticles, the mesoporous g-C₃N₄ prepared have a pore size of <6 nm and a pore volume of <1.2 cm³ g⁻¹. In order to further optimize the structure of mesoporous g-C₃N₄, the 3D cage mesoporous silica nanoparticles, *e.g.*, SBA-X (X = 1, 2, 6, 12, 16), FDU-X (X = 1, 2, 5, 12), and KIT-X (X = 5, 6), were employed as the hard templates.⁶⁸ For example, Lakhi *et al.*⁶⁹ prepared the 3D cage mesoporous g-C₃N₄ for the first time by directly polymerizing the precursor carbon tetrachloride and ethylenediamine in the FDU-12 template pore with different pore sizes. In addition, the 3D cage mesoporous g-C₃N₄ prepared with SBA-16 and KIT-6 as the hard template has also been reported.⁷⁰ Not only the mesoporous but spherical and rod-shaped g-C₃N₄ can be prepared by the hard template method.⁷¹ Although great progress has been made in the preparation of g-C₃N₄ with the novel layered micro-nanostructures by the hard template method, the removal of template requires etching with the toxic chemicals ammonium difluoride (NH₄HF₂) or hydrogen fluoride (HF), which not only affects the environment and experimental operators but also prevents the further functionalization of g-C₃N₄.⁷² Therefore, it is ideal and challenging to prepare g-C₃N₄ with the specific morphology by the template free method.

3.3. Preparation of tubular g-C₃N₄

Ring-shaped organic channel structure (ROCS) linked by 1:1 multiple hydrogen bonding between melamine and cyanuric acid (trithiocyanic acid) created by Rao *et al.*⁷³ by means of hydrothermal synthesis in 1999 (Figure 3a and 3b) has been widely adopted as the starting material for the preparation of tubular g-C₃N₄. For example, the ordered hollow g-C₃N₄ tube was prepared using the ROCS linked between melamine and cyanuric acid as the starting material.⁷⁴ In addition, fish scale tubular g-C₃N₄ was prepared using the ROCS linked between melamine and trithiocyanic acid as the starting material.⁷⁵ The molecular self-assembly method to prepare tubular g-C₃N₄ using the ROCS linked between melamine and cyanuric acid

(trithiocyanic acid) as the starting material can be divided into three steps, as follows⁴⁷:

- (1) the formation of ROCS;
- (2) the ROCS form the rod-like supermolecular precursors through the π - π stacking interactions;
- (3) the rod-like supermolecular precursors was placed in a tubular furnace with protective gas for the calcination (500–550 °C).

The pyrolysis starts from the center of the rod-like supermolecular precursors and diffused outward to generate tubular g-C₃N₄.⁷⁶ The molecular self-assembly method can also be used to prepare the spherical and rod-like g-C₃N₄ through the regulation of solvent and the intermolecular force.⁷⁷ The tubular g-C₃N₄ prepared by the molecular self-assembly method has the advantages of orderly structure, large S_{BET} , abundant reaction sites, high separation efficiency of electrons and holes, strong light absorption ability, and superb photocatalytic performance compared with bulk g-C₃N₄.

3.4. Preparation of other morphologies g-C₃N₄

Other morphologies of g-C₃N₄ include spherical, rod-like, and quantum dot. For example, the mesoporous hollow spherical g-C₃N₄ was prepared by the molecular self-assembly method using the ROCS linked between melamine and cyanuric acid as the starting material. The porous spherical g-C₃N₄ was prepared using the spherical mesoporous silica foam and melamine as the hard template and precursors, respectively.⁷⁸ It was reported that the g-C₃N₄ nanorods were prepared by the thermal polycondensation method using cyanamide as precursors with eutectic mixture of LiCl-KCl.⁷⁹

Furthermore, the morphologic transformation of g-C₃N₄ from nanoplates to nanorods was realized by a simple reflux method.⁸⁰ The formation of g-C₃N₄ nanorods was attributed to the possible peel off and regrowth of nanoplates during the reflux process as well as the edging mechanism of layered structure. As presented in Figure 4a, the g-C₃N₄ quantum dots (CNQDs) was prepared by the multiple stripping steps with bulk g-C₃N₄ as the starting material.⁸¹ Specifically, the bulk g-C₃N₄ was firstly stripped into nanosheets by the thermal oxidation etching method. Then, the g-C₃N₄ nanosheets were further stripped into nanoribbons by the chemical stripping method using the mixed solution of concentrated H₂SO₄ and HNO₃ as solvent. Finally, the g-C₃N₄ quantum dots with diameter distribution of smaller than 5 nm were successfully prepared by the shearing of nanoribbons using the two-step method of liquid phase stripping and hydrothermal (Figure 4b and 4c). The prepared g-C₃N₄ quantum dots with a diameter less than 10 nm have the upconversion

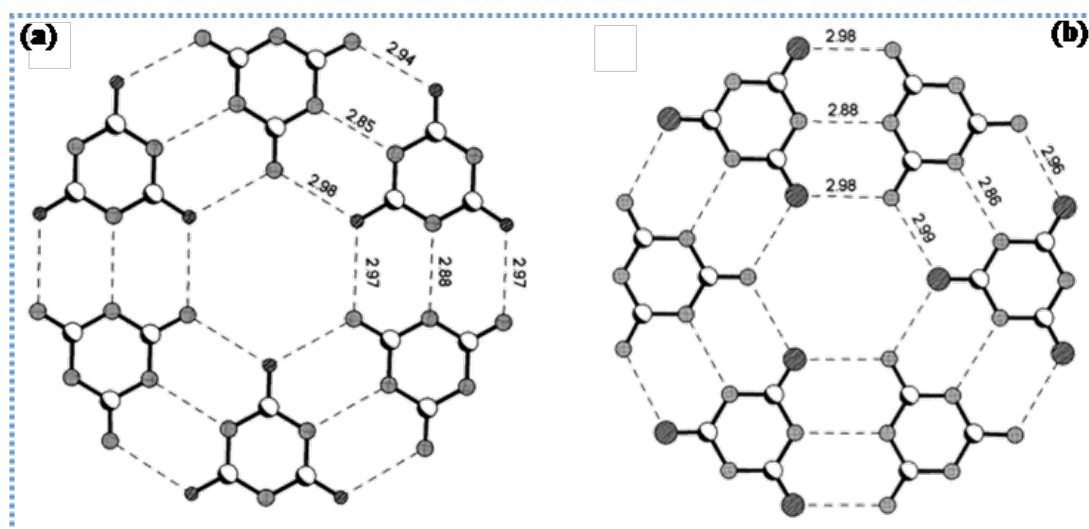


Figure 3. The ring organic channel structure of melamine-cyanuric acid (a) and melamine-trithiocyanuric acid (b). Both panels are reproduced with permission from Ranganathan and Rao.⁷³ Copyright © 1999 Wiley.

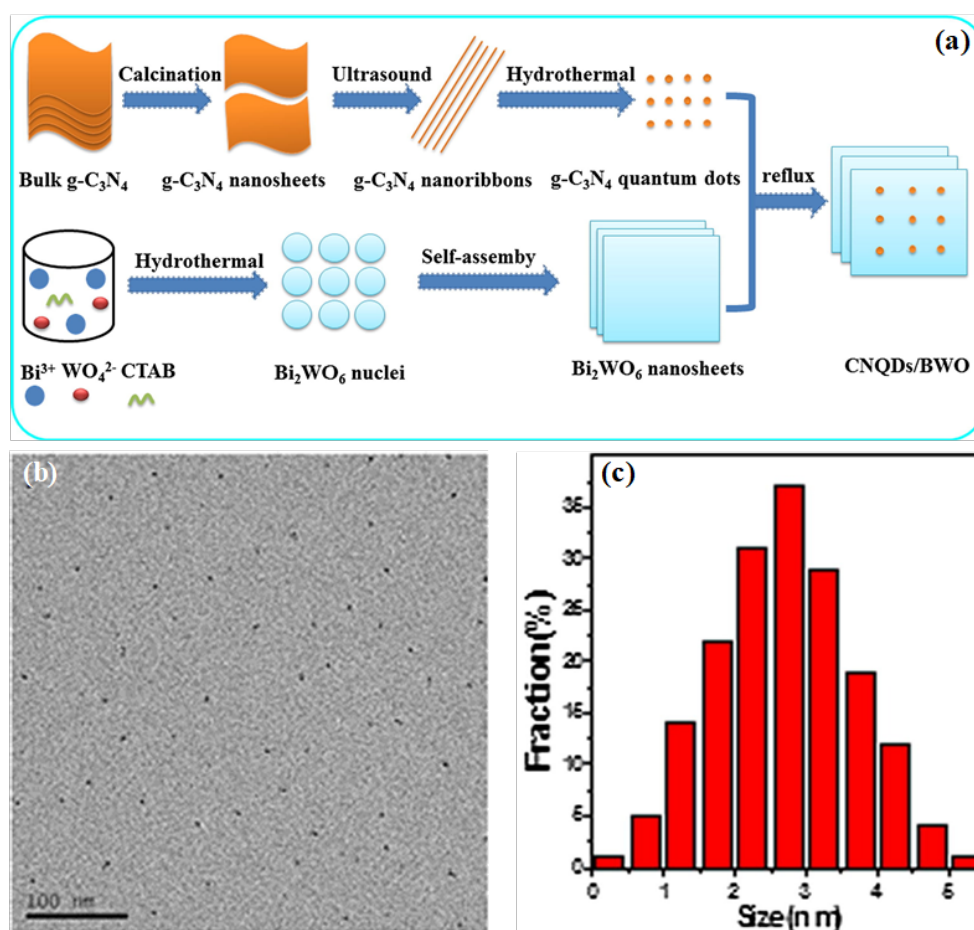


Figure 4. The preparation process, morphology, and size distribution of CNQDs. (a) The schematic diagram illustrating the synthesis of CNQDs/BWO composites. (b) The g-C₃N₄ quantum dots. (c) The diameter distribution of the CNQDs. Reproduced with permission from Zhang *et al.*⁸¹ Copyright © 2019 Elsevier.

effect and are able to convert the near-infrared (NIR) light into visible (Vis) light for the degradation of organic dyes.

The preparation methods of different g-C₃N₄ and their influences on photocatalytic performance are summarized in Table 1. It can be seen that ultrathin nanosheets achieve high photocatalytic H₂ production and ·OH generation activity by widening the E_g and shortening the carrier migration path. Their preparation method is simple but the yield is relatively low, and the ultrasonic exfoliation method has moderate scalability. Mesoporous structures significantly enhance the efficiency of active site and charge separation due to ordered channels and high S_{BET}. However, the removal of hard templates requires the use of toxic reagents, which is costly and limited in scale. The tubular structure enhances light absorption and electron-hole separation through one-dimensional charge transport and multiple light scattering. It does not require a template

but needs precise control of self-assembly conditions, and has moderate scalability. Although spherical/rod-shaped structures can increase the S_{BET} and shorten the diffusion path, the hard template method has the problem of toxic etching, and the reflux method has a limited yield and low scalability. Quantum dots have a wide E_g, upconversion luminescence and abundant edge sites, which can achieve a wide spectral response. However, their preparation steps are numerous and the yield is low. Currently, they are only suitable for laboratory-scale applications. Overall, different morphology engineering strategies each offer distinct advantages in enhancing photocatalytic performance, but the greenness, controllability and large-scale potential of their preparation processes still need further improvement.

4. Conclusion and outlooks

The photocatalytic performance is closely related to the

Table 1. Comparison of different morphology engineering strategies of g-C₃N₄

Morphology	Band structure/Charge behavior	Photocatalytic performance	Advantages/Limitations/Scalability
Ultrathin nanosheets	Broadened bandgap; shortened charge carriers migration path	High H ₂ evolution; enhanced ·OH production	<i>Advantages:</i> Simple; high activity <i>Limitations:</i> Low yield <i>Scalability:</i> Moderate ultrasonic exfoliation scalable
Mesoporous structures	Enhanced active sites; improved charge carriers separation	High H ₂ evolution; effective pollutant degradation	<i>Advantages:</i> Ordered porosity; tunable pore size; high S _{BET} <i>Limitations:</i> Toxic template removal; limited scalability <i>Scalability:</i> Low-hazardous etching; template synthesis costly
Tubular structures	1D charge transport along tube walls; strong light absorption due to multiple scattering; efficient electron-hole separation	Superior H ₂ evolution; enhanced visible light activity	<i>Advantages:</i> Template-free; ordered structure; high activity <i>Limitations:</i> Requires precise control of self-assembly conditions; batch-to-batch variation <i>Scalability:</i> Moderate-supramolecular synthesis relatively simple, but calcination step energy-intensive
Spherical/ rod-like	Increased S _{BET} ; shortened charge carriers diffusion pathways	Efficient degradation of organic dyes; high H ₂ evolution	<i>Advantages:</i> Diverse morphologies <i>Limitations:</i> Hard templating requires toxic etching; reflux method yields limited quantity <i>Scalability:</i> Low to moderate dependence on method
Quantum dots	Broadened bandgap; upconversion converts near-infrared (NIR) to visible light; abundant edge sites	Efficient photocatalytic degradation of organic pollutants under wide spectrum light	<i>Advantages:</i> Unique optical properties; excellent for photosensitization <i>Limitations:</i> Multi-step process; low yield; difficult to scale <i>Scalability:</i> Low, currently limited to lab scale only

morphology of semiconductors. This review clarifies the common strategies for preparing g-C₃N₄ with different morphologies and explains the improvement in photocatalytic performance from the perspectives of light absorption and charge transport. Morphology engineering is further elaborated in the following:

(1) Morphology engineering is an effective strategy to

enhance the photocatalytic performance of g-C₃N₄. Converting bulk g-C₃N₄ into different morphologies, such as ultrathin nanosheets, mesoporous structures, and tubular structures, can significantly increase the specific surface area, shorten the migration path of photogenerated charge carriers, and provide more active sites, thereby effectively suppressing electron-

hole recombination and improving photocatalytic efficiency.

- (2) Various morphology adjustment methods have their own advantages: thermal oxidation etching and liquid-phase ultrasonic exfoliation are suitable for preparing ultrathin nanosheets; hard templating is effective for constructing ordered mesoporous structures; and molecular self-assembly excels in producing special morphologies, such as tubular and spherical structures. Among these, liquid-phase ultrasonic exfoliation is more promising for green synthesis due to its mild conditions and recyclable solvents.
- (3) Morphology modification also contributes to optimized energy band structure and reaction activity. Ultrathin nanosheets and quantum dots not only increase the specific surface area but also broaden the bandgap and enhance the redox capability. For instance, single-atom-layer mesoporous g-C₃N₄ nanonets exhibit a photocatalytic hydrogen evolution rate as high as 8510 $\mu\text{mol h}^{-1}\text{g}^{-1}$, far superior to conventional bulk materials.

Although significant progress has been made in optimizing the photocatalytic performance of g-C₃N₄, further work is required to enhance its practical applicability.

- (1) Green and controllable template-free synthesis: Current hard templating methods require toxic reagents (HF or NH₄HF₂) for template removal. Future efforts should focus on template self-sacrifice, vapor deposition, or supramolecular self-assembly to achieve precise morphology control and environmental friendliness.
- (2) Coupling morphology engineering with heterojunction/defect engineering: Morphology modification alone provides limited charge separation improvement. Integrating Z-scheme or S-scheme heterojunctions, elemental doping, and defect engineering into hierarchical morphologies (e.g., nanosheets grown on tubes, mesoporous spheres loaded with quantum dots) can synergistically boost performance.
- (3) Scalable production: While liquid-phase ultrasonic exfoliation and molecular self-assembly showed moderate scalability, the yield of ultrathin nanosheets and quantum dots remained low. Developing continuous-flow or energy-efficient processes is urgently needed.
- (4) *In situ* characterization and structure-activity relationships: Real-time tracking of charge carrier dynamics and surface active sites using *in situ* X-ray photoelectron spectroscopy (XPS), transient

absorption spectroscopy, and *in situ* FT-IR, combined with DFT calculations, is essential to reveal the mechanistic roles of specific morphologies and guide rational design.

- (5) Expanding application scenarios: Beyond H₂ evolution and pollutant degradation, morphology-optimized g-C₃N₄ should be explored for CO₂ reduction, N₂ fixation, photothermal catalysis, and self-cleaning surfaces.
- (6) Data-driven analysis and systematic assessment: Beyond traditional qualitative reviews, emerging bibliometric and meta-analytic approaches have been successfully applied to analyze research trends and quantitatively assess the environmental effects of nanomaterials.^{82,83} Future studies on g-C₃N₄ morphology engineering could adopt such data-driven strategies to identify knowledge gaps, evaluate conflicting results, and guide the rational design of high-performance photocatalysts.

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Conflict of interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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