

# Carbon nitride-based photocatalysts for hydrogen evolution: A review

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## Abstract

One of the fundamental needs is energy for life and various studies were carried out in search of green, sustainable, and renewable energy sources. Photocatalytic hydrogen evolution is a promising route for the conversion of solar energy to hydrogen energy and in this process, energy production and wastewater treatment could be achieved as concomitant. To achieve higher hydrogen evolution rates, various photocatalysts have been developed and graphitic carbon nitride-based (CN) photocatalysts are promising ones. In this chapter, the photocatalytic properties of pristine CN and element-doped CN and the effect of these properties on the evolution rates were briefly discussed and some related studies from the literature were summarized.

## 1. INTRODUCTION

Energy is one of the leading worldwide concerns; fossil fuels might not compensate for the world's energy needs since they are an exhaustible and limited resource. In addition, fossil fuels could cause some hazardous impacts on human and environmental health. Due to these reasons, the search for alternative energy resources that are renewable, sustainable, and clean has recently gained great attention. Additionally, the production method of energy from renewable sources should be a green route. In this context, hydrogen production via photocatalytic oxidation stands out because hydrogen could be produced by the direct conversion of solar energy into chemical energy at ambient conditions (Zhurenok et al., 2023; Prasad et al., 2020). In this process, there is a requirement for a photocatalyst and light source. In this context, the usage of solar-driven or visible-light-activated photocatalysts attracts particular interest instead of photocatalysts sensitive

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to UV light because UV light comprises only 4% of the sunlight spectrum while visible light composes  $\sim 43\%$  (Zhang et al., 2010). Besides, solar-driven photocatalysts have low band gap energy and thus, they can absorb light at high levels.

Recently, graphitic carbon nitride ( $g\text{-C}_3\text{N}_4$ ) -abbreviated as CN in this chapter- which is a polymeric semiconductor has attracted extensive attention. Figure 1 shows the number of published articles found using “ $g\text{-C}_3\text{N}_4$  photocatalytic hydrogen production” as a keyword in Elsevier for the last nine years and it is clearly seen that interest in photocatalytic hydrogen production using CN has rose year by year.

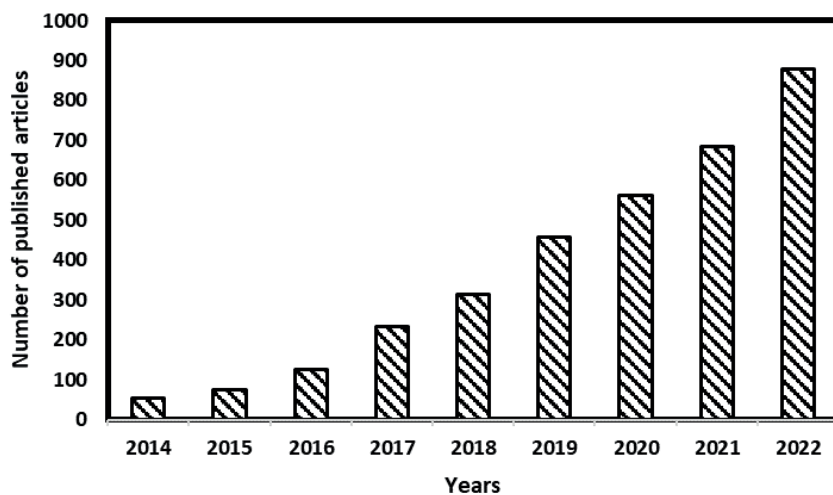


Figure 1. (a) Number published articles based on years in Elsevier using “ $g\text{-C}_3\text{N}_4$  photocatalytic hydrogen production” as a keyword since 2014

CN has a low band gap energy (2.7 eV) and  $E_{\text{CB}}$  and  $E_{\text{VB}}$  of CN were -1.13 eV and 1.57, respectively. Overall water splitting could be achieved when the conduction band-edge potential of the used photocatalyst is more negative than the reduction potential of water to evolve hydrogen and the top of its valence band must be more positive than the oxidation potential of water to produce oxygen. Therefore, CN enables to produce the hydrogen from water (Shaomang Wang et al., 2014; Zhurenok et al., 2023; Zhang et al., 2010). However, hydrogen may evolve in lower amounts when solely water is used so the production efficiency could enhance with the addition of a sacrificial agent (i.e., methanol, TeOA, etc.) (Zarkadoulas et al., 2015; Naga Mahesh et al., 2016). Furthermore, the usage of wastewater streams instead of water or electron donor-containing water provides some benefits.

The remediation of wastewater streams and hydrogen evolution could be achieved as concomitant. In addition, the presence of organic compounds in wastewater streams could reduce the electron donor cost.

This chapter focuses on the modification of CN to improve hydrogen evolution efficiency. In this context, the studies related to pristine CN and element-doped CN for hydrogen evolution from water and/or wastewater streams via photocatalytic oxidation were summarized and the effect of element doping over the evolved hydrogen amounts was discussed.

## **2. STUDIES ON PRISTINE AND MODIFIED CN FOR HYDROGEN EVOLUTION**

CN has thermal stability as well as photochemical stability and it is generally synthesized using nitrogen-rich precursors (i.e., cyanamide, urea, dicyandiamide, melamine, etc.) by thermal condensation. Pristine CN has generally low specific surface area and small active sites on the surface of CN for (photo)reactions. Although it is sensible to visible light, it could insufficiently absorb the visible light and the fast recombination of photoinduced electron–hole pairs causes low efficiency, and hence, some methods -copolymerization, elemental doping, and texture modification- were employed to enhance its photocatalytic activity (Cao et al., 2014; Xing et al., 2021; Wen et al., 2017). In this section, some related studies on photocatalytic hydrogen evolution in the presence of pristine CN and element-doped CNs were summarized.

### **2.1. Pristine CN for hydrogen evolution**

Pristine CN has slow surface reaction kinetics and low charge mobility as well as a high electron–hole recombination rate and low surface area. In addition, it has small active sites and the active sites of CN have great importance on the hydrogen evolution efficiency. The active sites of CN could be affected by improvement technologies, CN synthesis methods, and the location of active sites on the surface of CN. Some studies related to hydrogen evolution using pristine CN in different forms -nanorods (nr), nanosheets (ns), and microspheres (ms)- were summarized in Table 1.

*Table 1. A brief summary of improved active*

CN type	Improvement route	Synthesis method	Active sites of CN	Evolved H <sub>2</sub>	References
Microsphere	Increase specific surface area	Hard template	Edges	5785 $\mu\text{mol h}^{-1} \text{g}^{-1}$	(Liu et al., 2018)
Nanorods	Increase specific surface area	Supramolecular assembly	Edges, nitrogen defects	118.5 $\mu\text{mol h}^{-1}$	(Jiang et al., 2019)
Nanosheets	Increase specific surface area	Thermal treatment	Edges, defective sites	1233.5 $\mu\text{mol h}^{-1} \text{g}^{-1}$	(Su et al., 2018)

The most important factor that affects the photocatalytic activity of CN is morphology because of the stacked bulk structure of conventional CN that had only a few active sites. The active sites of a photocatalyst are generally located at the edge, kinks, unsaturated steps, terraces, and/or corner atoms and thus, the morphology of CN has a great influence on the photocatalytic efficiency of CN. The conventional CN has hydrogen bonds in its frameworks and interlayer force becomes highly stable to be destroyed. To overcome these drawbacks, the surface morphology of CN could be changed by increasing the specific surface area of CN, and hence, the contact area between the reactive substrate and CN augments and additional edges might appear. Therefore, active sites on the surface of CN increase.

## 2.2. Element-doped CN for hydrogen evolution

In order to enhance the charge separation efficiency of CN and to obtain higher recombination of charges, CN could be doped with non-metals and metals. In addition, light absorption could be efficiently achieved and thus, the hydrogen evolution rates could enhance. In this section, the studies related to heteroatoms such as boron (B) and carbon (C) doped CN for hydrogen evolution via photocatalytic oxidation were summarized.

### 2.2.1. Boron doping

The most commonly used B source to incorporate in CN structure is boric acid since its decomposition temperature and volatilization of water occur in the same temperature interval and it helps to obtain a porous structure. B could easily introduce into the CN structure because of the electron-deficient

nature of B; however, agglomeration, and a decrease in pore volume and size could be observed due to higher amounts of B (Patnaik et al., 2021).

Wang et al. synthesized B-doped CN quantum dots (BCNQDs) using the molten salts strategy and in this study, melamine and boron oxide were used as precursors. BCNQDs were incorporated on the surface of CN, forming a heterojunction. Almost  $7 \mu\text{molh}^{-1}$  of  $\text{H}_2$  evolved from TEOA-containing water using the pristine CN whereas approximately  $70 \mu\text{molh}^{-1}$  of  $\text{H}_2$  was produced in the presence of BCNQDs doped CN. Therefore, obtained hydrogen amount increased ten times and it could be concluded that the photocatalytic (Wang et al., 2019). Additionally, B-doped CN was synthesized using boric as a B source. The BET surface area of B-doped CN was  $143.2 \text{ m}^2\text{g}^{-1}$  and  $3800 \mu\text{molg}^{-1}\text{h}^{-1}$  of hydrogen was obtained in the presence of this photocatalyst (Kamal Hussien et al., 2022).

Chen et al. proposed a low-cost and energy-saving synthesis route for B-doped CN using melamine, urea, and boric acid as precursors. the interlayer gallery distance increased because N and C atoms were replaced with B atoms and due to the incorporation of B, the band gap energy of B-doped CN slightly shifted compared to pristine CN. Using this photocatalyst,  $1439 \mu\text{molg}^{-1}\text{h}^{-1}$  of hydrogen was evolved and this value is about 2.3 times higher than the evolved hydrogen amount in the presence of pristine CN. Furthermore, B-doped CN showed high stability as well as recyclability property (Chen et al., 2018).

Thaweesak et al. synthesized a new type of yellow spongy B doped (precursor: ammoniotrihydroborate) CN (precursor: dicyanamide) nanosheet by one-pot thermal polycondensation for hydrogen production via photocatalytic oxidation. This photocatalyst showed a 12 times higher hydrogen evolution rate compared to pristine CN and  $1880 \mu\text{molg}^{-1}\text{h}^{-1}$  of hydrogen was evolved using this B-doped CN photocatalyst. In addition, it has good high stability as well as recyclability property (Thaweesak et al., 2017).

Boron has electron-deficient property and thanks to this property, the electron-hole pairs on the surface of B-doped CN can be effectively separated. Hence, a main requirement of photo redox reaction was achieved. By doping CN with B, an electron-rich atom was replaced with an electron-deficient atom and it accelerated the electron channelization in CN, and also, vacant-p-orbitals formed for electron delocalization. In addition, the usage of boric acid as a doping agent provides to obtain a porous structure.

### 2.2.2. Carbon doping

C-doped CN was synthesized using chitosan (as a C source) and melamine by copolymerization and results showed that N atoms were replaced by C atoms. Therefore, the delocalization of  $\pi$  bonds cause an increase in the visible light absorption efficiency and C-doped CN showed lower recombination rates. Due to these reasons, the photocatalytic activity of CN was enhanced by doping C and Liu et al. reported that  $1224 \mu\text{molg}^{-1}\text{h}^{-1}$  of hydrogen was evolved using C-doped CN. In addition, Au nanoparticles were uniformly distributed on the surface of C-doped CN and by the incorporation of Au nanoparticles hydrogen evolution rate increased three times compared to C-doped CN (Liu et al., 2021).

### 2.2.3. Sulphur doping

S-doped CN was widely used for hydrogen evolution. In this context, various S sources such as thiourea, benzyl disulfide, and thioacetamide were used; however, most commonly thiourea was preferred since it is a low-cost and easily accessible chemical. During the S doping, mostly the location of N atoms in the structure of CN replaces with S atoms and hence, the surface area increases, and a considerable decrease in band gap energy could be achieved because of the interaction of 3p states of S with 2p states of N. Incorporation of S into the CN structure, VB and CB potential could change and a band gap energy could be lowered. Therefore, the hydrogen evolution efficiency could enhance. Hong et al. used S-doped mesoporous CN to produce hydrogen by photocatalytic oxidation and they reported that the addition of S caused an increase in the surface area of the photocatalyst. The pristine CN had  $12.8 \text{ m}^2\text{g}^{-1}$  of surface area while the S-doped CN has  $128.4 \text{ m}^2\text{g}^{-1}$ . S-doped mesoporous CN acts as a good photocatalyst to perform the evolution of  $\text{H}_2$  via water reduction with a very high quantum efficiency (5.8%) at 440 nm.  $\text{H}_2$  evolution rate in the presence of S-doped mesoporous CN was found to be 30 times higher as compared to pristine CN (Hong et al., 2012). In another study, melamine was used as a precursor to synthesize CN and it was doped with S. Using this photocatalyst,  $24.6 \mu\text{molh}^{-1}$  of hydrogen was evolved by water splitting (Lin et al., 2020). Consequently, using S-doped CN, solar energy could be converted into hydrogen energy. In addition, the introduction of S into the CN structure causes mesoporosity and due to this property, the photocatalytic activity of S-doped CN was higher than pristine CN.

### 2.2.4. Phosphorus doping

The optical and electronic properties of CN could be altered by the incorporation of P-atom into the graphitized network of CN. When P is

introduced into the CN network, it can form a chemical bond with the neighboring N and C-atoms and result in planar coordination, and thus, the photocatalytic efficiency of CN enhances. Conductivity and capability of transfer could enhance by strengthening the active sites with lone pairs of electrons. P-doped CN was generally prepared via in-situ thermal copolymerization and in this process, an N-rich precursor for the formation of CN network and a P-containing compound (i.e., melamine phosphate, phosphorous acid, ammonium hexafluorophosphate, phosphoric acid, etc.) as the precursor for interstitial or substitutional P doing depending on the P source were used to synthesize P doped CN (Zhu et al., 2015; Fang et al., 2018; Yang et al., 2018).

For instance; phosphonic acid as a P source could be used to obtain homogeneous and substantial P-doping CN. If organo-phosphonic acid and melamine were used as precursors, cross-linking could be achieved due to the existence of acid-base interaction between phosphonic groups and amino groups of the triazine unit. In addition, throughout the polycondensation, P could directly alter the electronic and textural features resulting in obtaining a promising photocatalyst for hydrogen production. Throughout the thermal treatment, the precursors (organo-phosphonic acid and melamine) undergo polymerization and decomposition as contaminants and obtain homogeneous and substantial P-doping CN forms due to the higher thermal stability of the P source. Obtained P-doped CN had a porous structure and it provides an increase in charge separation and the number of active sites. On the other hand, it causes a considerable decrease in particle size as well as interlayer separation. P atom is relatively bigger compared to C and N atoms and due to its atom size, graphitic CN could be converted into CN nanosheets. Consequently, P-doped CN nanoflowers could form due to the self-assembly effect. Mesoporosity could form due to the solubility difference of organo-phosphonic acid in water and ethylene glycol.

Zhu et al. synthesized flower-like P-doped mesoporous CN and they reported that band gap energy reduced by the introduction of P into CN. It could be deduced that P atoms and CN set up a good electronic integration to absorb visible light efficiently and to favor charge transport. Additionally, the electron-hole recombination rate decreased while electron channelization in P-doped CN increased. These altered features create a route to produce hydrogen in higher amounts. Whereas evolved hydrogen amount was  $11.2 \mu\text{molh}^{-1}$  using pristine CN, it was obtained as  $104.1 \mu\text{molh}^{-1}$  in the presence of P-doped CN due to the reduction in the diffusion distance. Additionally, P-doped CN showed high stability and durability for a long time (more than



16 h) so it became a good candidate for practical applications (Zhu et al., 2015).

Yang et al. prepared a flower-like (FL) porous P (precursor: phosphoric acid)-doped CN (precursor: cyanuric acid-melamine) via thermal condensation which is a low-cost, template-free route. Owing to the enhanced light absorption efficiency, the hydrogen evolution rate was  $256.4 \mu\text{molh}^{-1}$  using FL porous P doped CN and it is approximately 24 times higher than the hydrogen evolution rate in the presence of pristine CN (Yang et al., 2018).

Phosphoric acid provides two benefits that are being a dopant and stabilization of P-doped CN. Fragmented P-doped CN nanoflakes (PCNNFs) were synthesized via thermal copolymerization followed by a post-treatment process comprises of ultrasonication and phacofragmented in water, freeze-dried thoroughly and again heating at high temperatures ( $600 \text{ }^\circ\text{C}$ ) using urea and phytic acid biomass that are precursors for CN and P, respectively. Based on the characterization study (XRD, SEM, TEM), it could be concluded that doping with phytic acid biomass improved the photocatalytic features of CN, and the BET area of PCNNFs was found as  $223.2 \text{ m}^2\text{g}^{-1}$ . Hydrogen evolution rate increased 62 times by using P doping CN instead of bare CN under light had longer wavelength ( $\lambda > 470 \text{ nm}$ ) irradiation and in addition, notable hydrogen evolution rate ( $15921 \mu\text{molg}^{-1}\text{h}^{-1}$ ) and quantum efficiency (6.74%) were obtained at  $420 \text{ nm}$  (Fang et al., 2018).

Guo et al. synthesized P-doped hexagonal CN (PCN) tubes by hydrothermal treatment of CN and phosphoric acid and the partial hydrolysis of precursors (melamine–cyanuric acid supramolecular) was subsequently achieved. The layered stacked structure of CN was responsible for the band gap decrease while doping P was responsible for the increase in surface area and conductivity. Owing to the increase in surface area, the number of active sites on the surface of P-doped CN has risen. In addition, the electron-hole recombination rate decreased thanks to P-doping. The structure of CN causes an increase in the chemical functionality and it became a good carrier material. The apparent quantum efficiency for PCN tubes was 5.68% at  $\lambda > 420 \text{ nm}$  using 1 wt. % Pt, which was approximately 7 times more than that of pristine CN. In addition, the evolved hydrogen rate was  $67 \mu\text{molg}^{-1}\text{h}^{-1}$  in the presence of PCN tubes (Guo et al., 2016).

P (source: sodium pyrophosphate) doped tubular CN (precursor: melamine) with surface C-defects was synthesized by Guo et al. In this study, supramolecular P-doped CN rods were synthesized via hydrothermal



treatment. Additionally, P-doped CN rods which had similar structures were synthesized using different P sources which are sodium phosphate, sodium hypophosphite, and ammonium phosphate to investigate the effect of the P source over hydrogen evolution. The peculiar structure of CN (C-defects and regulated C/N ratio) might enhance the photocatalytic hydrogen evolution rates. The highest hydrogen evolution ( $57 \mu\text{molg}^{-1}\text{h}^{-1}$ ) was observed in the presence of P-doped CN which was prepared using sodium pyrophosphate as a P source under visible light irradiation. It was referred to surface C-defects, hierarchical pore structure, high ratio of P-doping, reduced charge transfer distance, and increased surface area (Guo et al., 2017).

As a conclusion, the P source affects the P-doped CN network, and as P content increases, as photocatalytic activity increases owing to the surface area increase which is responsible for the adsorption/desorption of reactants. Substitutional and interstitial P doping could commonly provide using [Bmim]PF<sub>6</sub> and (NH<sub>4</sub>)<sub>2</sub>HPO<sub>4</sub> as the P sources. If phosphoric acid was used as a P source, it not only stabilizes P-doped CN but also is a dopant. Additionally, if C atoms in CN are replaced with P atoms, there are extra electrons in the pi-conjugated triazine ring to incorporate some positively charged P dopants to form the defective band. It provides a fast charge separation and narrow band gap.

### 2.2.5. Oxygen doping

Doping with oxygen (O) does not affect the structure; however, it affects the morphological and textural properties. For instance; the addition of O into the CN structure causes a decrease in the band gap while it leads to an increase in surface area and light absorption ability. Thus, the photocatalytic hydrogen evolution could enhance (Qu et al., 2018). Doping with O might occur in two ways and one of them is the replacement of C or N atoms with O atoms and the other one is the insertion at the interstitial position. Doping with O cause an increase in hydrophilicity of the CN-based photocatalyst and thanks to this property, hydrophilic contaminants could be easily adsorbed on the surface of O-doped CN.

She et al. synthesized O-doped CN ultrathin nanosheets using bulk CN and oxidizing agents (sulfuric acid and nitric acid) by calcination (at room temperature) and subsequently chemical oxidation was performed and 2D porous O-doped CN was obtained. Owing to the addition of oxidizing agents into the CN network, various O-containing functional groups such as C-O, COOH, and C=O are incorporated, and hence, defect sites formed on CN. The interplanar distance was lowered and interaction between layers was

fortified by the addition of O in the CN structure since the electronegativity of O-atoms is higher than N-atom. O-doped CN had  $109.3 \text{ m}^2\text{g}^{-1}$  of surface area and an increase in the surface area ensures the formation of the adequate number of active sites on the surface of CN. In addition, band gap energy reduced with O doping, and almost  $189.3 \mu\text{molh}^{-1}$  of hydrogen was evolved by the degradation of methyl orange via photocatalytic oxidation (She et al., 2016).

Oxygen (source: 1,3,5-trichlorotriazine) self-doped CN (precursor: dicyanamide) nanospheres were synthesized via the solvothermal process. The O source was used not only to adjust the crystal structure but also to dope the O atom into the CN network. In addition, the doping amount was controlled by adjustment of the dopant precursor amount. The hydrogen was evolved from the Rhodamine B-containing solution and  $3174 \mu\text{molg}^{-1}\text{h}^{-1}$  of  $\text{H}_2$  evolved using the synthesized O self-doped CN photocatalyst. The band gap energy varied between 2.09 and 2.73 eV depending on the amount of O source (Wei et al., 2018).

### 3. CONCLUSION

The efficient photocatalysts for hydrogen evolution via photocatalytic oxidation have still developed and in this chapter, CN-based photocatalysts for hydrogen evolution were briefly introduced. In the light of literature, it could be concluded that elemental (B, C, S, P, O) doping CN has lower band gap energy and low electron-hole recombination rate compared to pristine CN. In addition, the surface area of CN increased by doping with elements and the number of active sites on the surface of elemental-doped CN was more than the pristine one. These altered properties provide to obtain higher hydrogen evolution rates since the photocatalytic properties of CN enhanced with elemental doping. Consequently, the CN-based photocatalysts are promising for hydrogen evolution via photocatalytic oxidation.

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