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Chapter

Recent Development of Graphitic Carbon Nitride-Based Photocatalyst for Environmental Pollution Remediation

Mohamad Fakhru Ridhwan Samsudin, Nurfatien Bacho and Suriati Sufian

Abstract

Globalization today has helped fuel the global socioeconomic growth of the world and reshaping the growth of the industries. While the development had been remarkable, the rapid rise of industrialization had provoked the sustainable chain of diversity which is reflected by rising pollution level, particularly on the water pollution. On account of the cutting edge of water security issue, engineering photocatalytic material remains crucial in finding new ways to combat the challenge of water pollution through photocatalytic pollutants degradation while at the same time acts as the frontlines for energy conversion and environmental protection. To date, graphitic carbon nitride, g-C₃N₄ had emerged as a promising material of interest in photocatalytic application due to its appealing characteristics such as excellent optical properties and high physiochemical and thermal stability. This chapter will comprehensively discuss an insight into the most recent progress in synthesis, properties and the photocatalytic application of g-C₃N₄, particularly in environmental pollution remediation. Special emphasis is also placed on the most recent strategies for enhancing the photocatalytic performance of the g-C₃N₄ photocatalyst. Finally, the future directions and perspectives will be presented.

Keywords: graphitic carbon nitride, photocatalyst, degradation, nanomaterial

1. Introduction

The exponential growth of the industries over the past decade had exerted substantial pressure on sustainability, mainly on the pillars of the environment. While the gap towards an improved well-being brings many intended benefits. The environmental sustainability challenge is also growing at a large scale and complexity, resulting in severe environmental impacts including water pollutions. Within this context, the rise of industrialization alongside with the rapid growth of the global population has been intimately linked with a higher generation of wastewater [1–3]. The statistical glance from United Nations World Water Assessment Programme (2003) has reported that around 2 million tons of industrial sewage and agricultural waste have been discharged into the water bodies every day. Based on

the report by the WHO/UNICEF Joint Monitoring Programme, the current statistics reveal that there are approximately 2.1 billion people who are lack access to clean drinking water. Another report by UNESCO (2017) claims that approximately 80% of the wastewater flow is discharged into the ecosystem without any treatment thus contaminating a large portion of the water bodies. In this sense, the excessive release of anthropogenic pollutants originated from industrial use such as the phenolic compound, heavy metals and dyes had resulted in a deterioration in water quality and pose harmful effect on the living organism, which further emphasizes the need to tackle the water pollution issue [4–6].

Engineering photocatalytic material had emerged as a promising technology to bridge the gap between global energy challenge and environmental remediation. Since the pioneering discovery of photocatalytic water splitting by Fujishima and Honda in 1972, photocatalytic material has attracted interdisciplinary attention due to its diverse potential in various discipline such as solar energy conversion, photocatalytic water splitting for hydrogen production and carbon dioxide reduction, organic pollutants degradation and synthesis of organic compounds [7–9]. For the wastewater treatment field, photocatalytic degradation of pollutants is favorable over the conventional method due to its several advantages. This technique does not require non-renewable energy consumption as it exploits the sustainable solar energy [10–12]. Applicable both for gaseous and aqueous treatment, photocatalysis technology reportedly can degrade a wide range of pollutants and toxic compounds without causing any secondary pollutants. Moreover, the photocatalyst can be easily synthesized through various methods from an abundant readily available precursor. The whole process is not only simple to conduct, low in cost and require a relatively short process time, making the method sustainable for wastewater purification in a large scale application.

At presents, there are a various ongoing effort for the development of the sustainable photocatalytic system, with the focused centered on the development of noble metal-free photocatalyst as TiO_2 , $\text{g-C}_3\text{N}_4$, BiVO_4 , ZnO , and carbonaceous materials [12–17]. Among these photocatalysts, graphitic carbon nitride ($\text{g-C}_3\text{N}_4$) has elicited significant interest as the next generation of the photocatalyst in engineering photocatalytic field for environmental pollutants degradation due to its excellent physiochemical properties [18–21]. The $\text{g-C}_3\text{N}_4$ is novel, metal-free photocatalyst with good light absorption properties owing to its medium band-gap energy of 2.7 eV [19, 22, 23]. The polymeric nature of this conjugated materials allows for facile modification of the photocatalyst to improve its optical properties besides permit multiple excitations from absorption of a single photon, both of which are favorable for efficient pollutants degradation. However, the practical application of $\text{g-C}_3\text{N}_4$ is still hindered by some of its individual properties such as low visible light utilization, the high recombination rate of photogenerated electron–hole pairs and slow electron transfer which lead to lower photocatalytic performance [22]. Hence, various strategies have been adopted such as energy band engineering, copolymerization with nitrogen precursor and development of heterostructure system in order to overcome the individual drawbacks of pristine $\text{g-C}_3\text{N}_4$ [24–26].

In recognition of the great potential of $\text{g-C}_3\text{N}_4$ as a promising visible light driven photocatalyst, this chapter is aiming to provide an overview on the most recent related studies on the development of $\text{g-C}_3\text{N}_4$ photocatalyst in the environmental pollution remediation. The history and basic principle of photocatalyst system are well explained in order to promote better understanding on the $\text{g-C}_3\text{N}_4$. Afterward, the fundamental properties of $\text{g-C}_3\text{N}_4$ and the synthesizing techniques are briefly summarized. Next, the current strategies to enhance the

overall performance of g-C₃N₄ photocatalyst are explained. Subsequently, the current perspective and future directions of the g-C₃N₄ photocatalyst are included in this chapter.

2. General overview on g-C₃N₄ photocatalyst

The history of engineering photocatalytic material could be traced back to 1972, where photoelectrochemical splitting of water in the presence of TiO₂ and ultraviolet light by Fujishima and Honda had served as the starting point for photocatalytic reaction [27]. Since then, numerous semiconductor-based photocatalysts have been investigated in an attempt to produce a robust photocatalyst for an efficient novel photocatalytic system. Among them, TiO₂, BiVO₄, Fe₂O₃ and ZnO had been identified as a potential promising photocatalytic material [28–30]. Although Titanium oxide (TiO₂) had dominated the photocatalytic arena, this photocatalyst suffers from its negative characteristics which hinder the practical exploitation of this material for large scale application [8, 9]. Hence, other potential materials have been tested and explored in the search for robust photocatalyst for large scale application. Among them, g-C₃N₄ had emerged as one of the promising material and become the new research hotspot for various scientific application owing to its excellent features.

2.1 Introduction to g-C₃N₄ photocatalyst

Graphitic carbon nitride is one of the oldest artificial polymer reported back in 1834. Structurally analogous to graphene, this conjugated polymer is a novel, metal-free with a medium band gap of 2.7 eV [31]. Generally, there are several allotropes of C₃N₄ such as α -C₃N₄, β -C₃N₄, pseudocubic C₃N₄, cubic C₃N₄, g-h triazine and g-C₃N₄. However, g-C₃N₄ is considered the most stable form of C₃N₄ under ambient conditions. **Figure 1** illustrates the basic tectonic units to establish the allotropes of the g-C₃N₄ photocatalyst. It was reported that the tri-s-triazine-based g-C₃N₄ photocatalyst was the most stable phases of C₃N₄ at ambient conditions. This postulation was further verified by Kroke et al. [32] with their first-principles density functional theory (DFT) calculations. Meanwhile, only the pseudocubic and g-h triazine phases have direct band gaps while the other phases have indirect band gap energies [32].

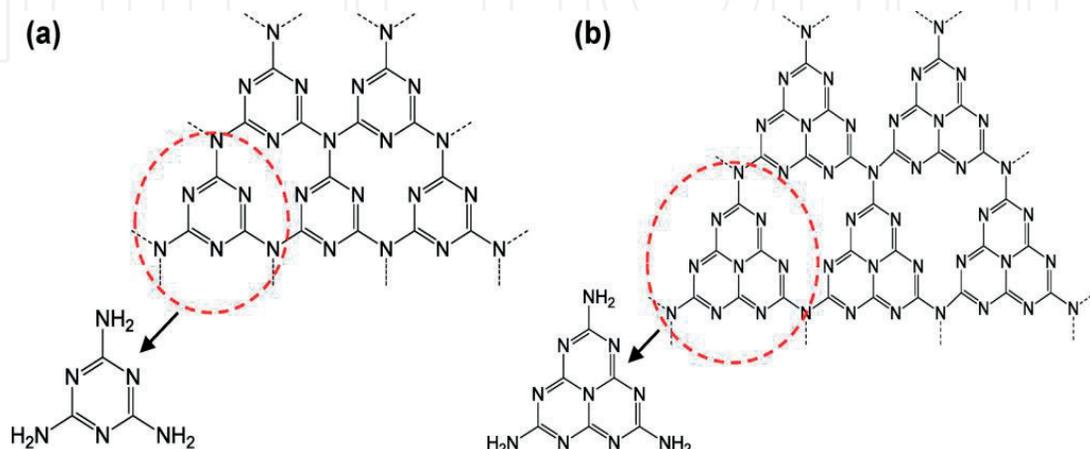


Figure 1. The basic tectonic units for g-C₃N₄ photocatalyst (a) triazine and (b) tri-s-triazine (heptazine) structures. (Adapted with permission from Ref. [22]).

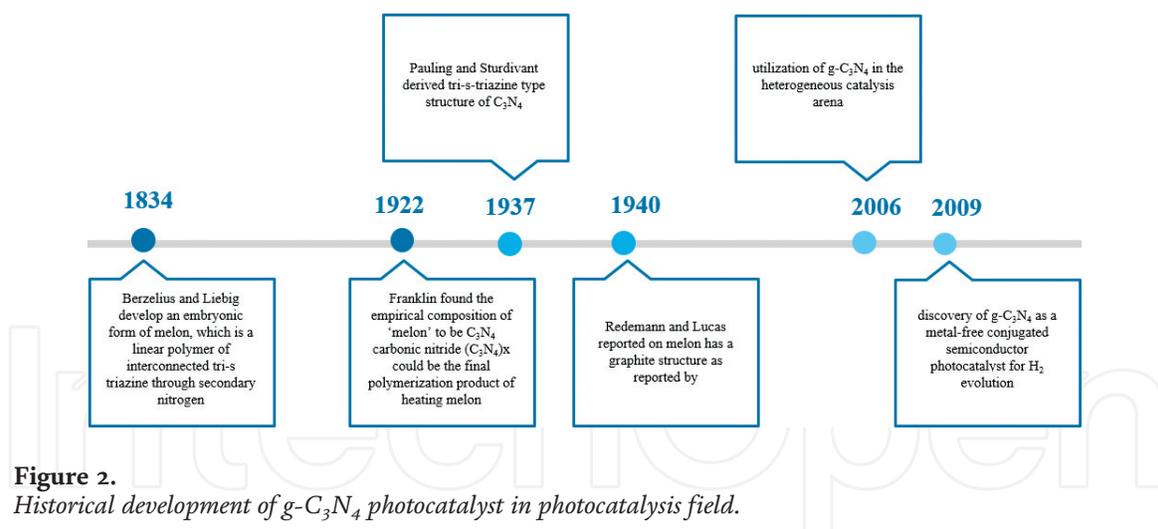


Figure 2. Historical development of $g-C_3N_4$ photocatalyst in photocatalysis field.

Carbon nitride, C_3N_4 is not new at all, and it is considered one of the oldest reported artificial polymers in the scientific literature [26]. **Figure 2** shows the historical development of $g-C_3N_4$ in photocatalysis arena. This $g-C_3N_4$ was first reported by Berzelius and Liebig in the year 1834 and named as melon [33]. Although it was first discovered back in 1834, the material is not exploited until 2009 when Wang et al. first reported the utilization of this material in photocatalysis field [26]. Since then, a lot of researchers started to unravel the promising potential of the $g-C_3N_4$ photocatalyst in a wide range of different photocatalytic applications. Nevertheless, owing to the limited experimental data, there is a prevailing discussion about the actual existence of a graphitic material with idealized composition C_3N_4 and possible structure models for $g-C_3N_4$ photocatalyst. Gratifyingly, due to the similar structure with graphite, triazine (C_3N_4) had been put forward as the elementary block of $g-C_3N_4$. Moreover, tri-s-triazine rings also shown promises to be energetically favored with respect to the triazine-based modification as the tri-s-triazine rings are cross-linked by trigonal nitrogen atoms [34]. Ideally, the condensed $g-C_3N_4$ consists of only carbon and nitrogen atoms with a C/N molar ratio of 0.75. Nonetheless, there is no perfectly condensed $g-C_3N_4$ was reported and they are as-grown polymer materials that are not single crystals. Hence, the $g-C_3N_4$ photocatalyst can be seen as a family of layered graphitic carbon nitride compounds with a C/N ratio close to 0.75.

2.2 Synthesis method for the development of $g-C_3N_4$ photocatalyst

Since the potential of carbon nitride in the photocatalysis arena had been known, various synthesis method and technique had been instigated such as chemical vapor deposition, sonochemical, solvothermal, and thermal annealing of nitrogen-rich precursor [24, 32, 33]. Following the variation in synthesis technique, the various compound can be used as the precursor such as melamine, cyanamide, dicyanamide, urea, and thiourea. However, thermal condensation of nitrogen-rich precursor has emerged as the most attractive methods due to its simplicity and use of cheap, earth-abundant precursors.

In the first reported work by Wang et al. [35], cyanamide was used as the starting precursor of $g-C_3N_4$. It was found that the cyanamide molecules were condensed to dicyanamide and melamine at temperatures of ca. 203 and 234°C. Next, the ammonia was removed via condensation process, resulting in the formation of melamine-based products at the temperature around 335°C. When the temperature was heated up to ca. 390°C, the tri-s-triazine units was formed via rearrangements of melamine. Finally, the polymeric $g-C_3N_4$ photocatalyst was

formed at ca. 520°C through the further condensation of the unit. Nevertheless, the overheating of the sample over 700°C resulted in the disappearance of g-C₃N₄ to “residue-free” through the production of nitrogen and cyano fragments. With respect to the experimental approaches, the reaction mechanism of the combined polyaddition and polycondensation process was further verified by the ab initio calculations using a plane wave basis set with a 550 eV energy cutoff [34]. Based on the calculations, **Figure 3** illustrates the cohesive energy of the molecules increased following the polyaddition pathway, confirming that melamine was produced upon heating the cyanamide [22].

Meanwhile, the most commonly used precursor in thermal condensation method includes those with nitrogen-rich and oxygen-free compounds, which contain the required C-N structure. The compounds with pre-bonded C-N core structure such as melamine, cyanamide, dicyanamide, urea and thiourea are among the commonly used precursors for the synthesis of g-C₃N₄. In addition to that, triazine and heptazine derivatives also had been tested as a precursor. For example, Mo et al. [34] prepared the g-C₃N₄ photocatalyst via manipulating the calcination temperature on the morphological structure of melamine. It was found that the g-C₃N₄ photocatalyst can only be formed when the calcination temperature above 500°C, evidently from the XRD analysis. Furthermore, it was found that the absorption band edge was red shifted along with the change of color from light yellow to dark orange, indicating the enhanced visible light absorption was obtained for samples with increasing calcination temperatures.

On the other hand, Dong et al. [36] prepared the g-C₃N₄ using urea via a facile template-free. Their group studies the effects of pyrolysis time on the microstructure and activity of g-C₃N₄ photocatalyst. They suggested that the surface areas of the photocatalyst can be significantly increased by just prolonging the pyrolysis time to 240 minutes at under 550°C. They claimed that the surface area of g-C₃N₄ photocatalyst prepared via this method is higher than g-C₃N₄ photocatalyst prepared via templating method. Similarly, Chen et al. [37] prepared g-C₃N₄ photocatalyst by pyrolyzing urea in a muffle furnace at 550°C for 2 hours with a heating rate of 5°C/min. Meanwhile, Yang et al. [38] prepared ultrathin g-C₃N₄ photocatalyst nanosheets via thermal exfoliation of bulk urea-derived g-C₃N₄ under an argon atmosphere. In addition, **Figure 4** summarizes the synthesis process of g-C₃N₄ photocatalyst by thermal polymerization of different precursors.

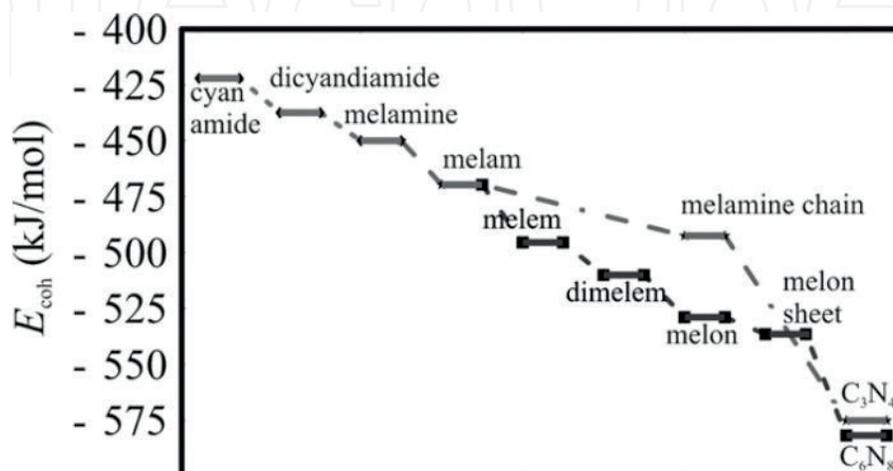


Figure 3. Calculated energy diagram for the development of C₃N₄ using cyanamide as the precursor. Cyanamide was condensed to melamine. Further condensation proceeded by a triazine route (dash-dot line) or tri-s-triazine (dashed line). (Adapted with permission from Ref. [33]).

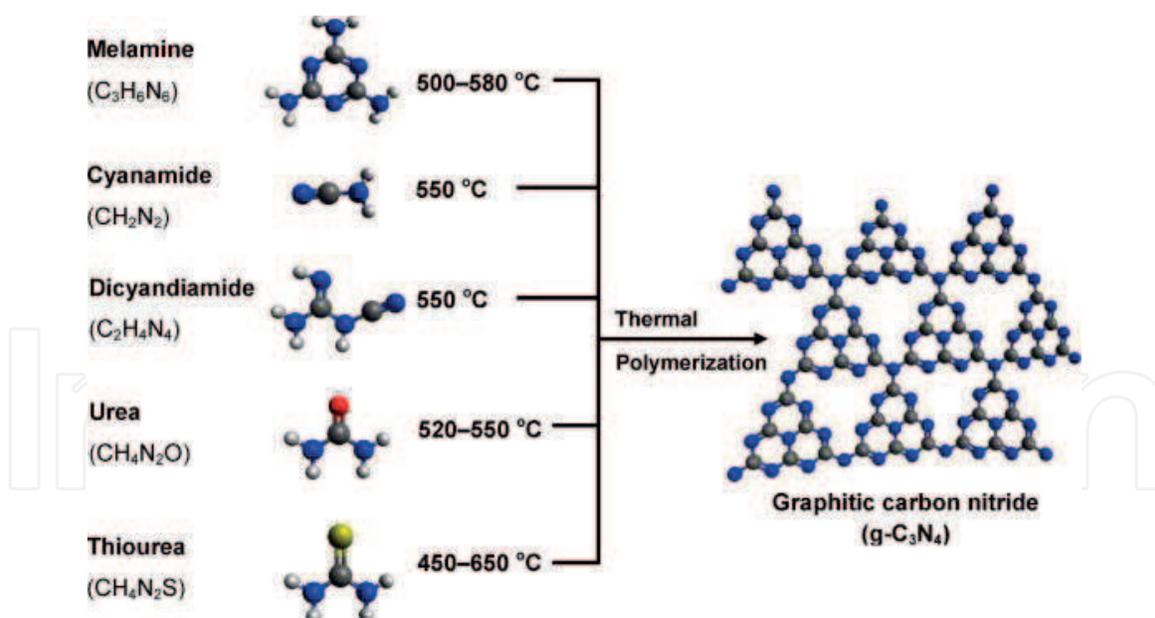


Figure 4.

Schematic illustration of the synthesis process of $g-C_3N_4$ photocatalyst via thermal polymerization of different precursors. The black, blue, white, red and yellow balls denote C, N, H, O and S atoms, respectively. (Adapted with permission from Ref. [22]).

2.3 Fundamental properties of $g-C_3N_4$ photocatalyst

Graphitic carbon nitride possesses an excellent physical, chemical and mechanical properties, giving a reason on why researchers are eager into the science of nanotechnology of $g-C_3N_4$. This compound possesses excellent visible light absorption than most of the metal oxide photocatalyst owing to its mild band gap energy. Given its π -conjugated properties, $g-C_3N_4$ can act as an electron sink, leading to suppression of recombination of photo excited charge carriers. Moreover, the polymeric nature of this material allows for multiple excitations from absorption of a single photon, leading to an efficient generation of the reactive species responsible for pollutant degradation. In addition, $g-C_3N_4$ possesses high resistance to thermal and chemical environment, as well as too acidic and basic media, making it as a stable material.

Furthermore, Wang et al. [39] performed a density functional theory (DFT) calculations in order to gain insight into the electronic structure of $g-C_3N_4$ photocatalyst (as shown in Figure 5). They reported that the valence band and conduction band are mainly composed of the nitrogen p_z orbitals and carbon p_z orbitals, respectively. The light illumination excited the electrons and holes for the oxidation and reduction process to occur independently in the nitrogen atoms and carbon atoms. Congruously, the $g-C_3N_4$ photocatalyst has a specific microstructure, with surface termination as defects and nitrogen atoms for electron localization or anchoring inorganic/organic functional motifs as the active sites [32].

However, the practical application of $g-C_3N_4$ is still hindered by some of its undesirable properties which lead to lower photocatalytic performance. The individual structure of $g-C_3N_4$ has low specific surface area and quantum efficiency, which limit the sorption capacity of the photocatalyst [26]. This photocatalyst also suffers from high recombination rate of the photogenerated electron–hole pairs.

2.4 Photocatalytic principles and mechanism over $g-C_3N_4$ photocatalyst

Photocatalyst can be described as a combination of catalysis and photochemistry in which absorption of photon energy from light via catalyst is the

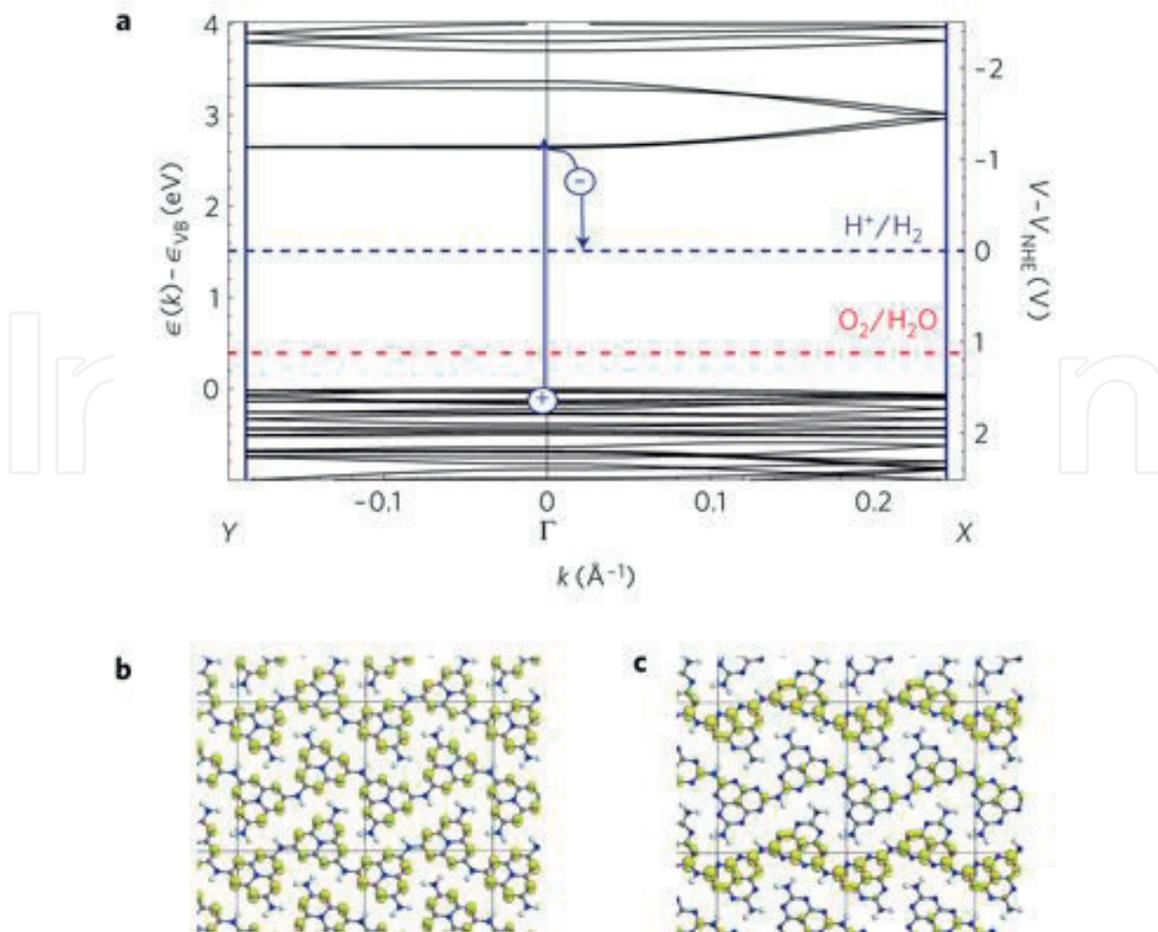
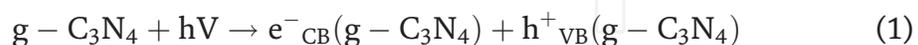


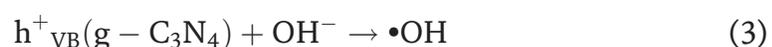
Figure 5.
 Electronic structure of the polymeric $g\text{-C}_3\text{N}_4$ photocatalyst. (Reproduced with permission from Ref. [39]).

key towards photoreaction process. In this system, photon energy is required to activate the photocatalyst in which make this photocatalyst system to be one of the frontier renewable energy technology in which it can utilize the solar energy. The $g\text{-C}_3\text{N}_4$ photocatalyst has a small band gap energy around 2.6–2.7 eV which falls within the visible-light region [25]. It is estimated that the valence band (VB) and the conduction band (CB) of the $g\text{-C}_3\text{N}_4$ photocatalyst are 1.56 and 1.09 eV, respectively [40].

Initially, the $g\text{-C}_3\text{N}_4$ photocatalyst will absorb photon energy with an energy equivalent or greater than its band gap energy, causing an electron (e^-) in the valence band (VB) to be excited and migrate to the conduction band (CB).



Then, the electron will be excited and leave photogenerated holes (h^+) in the valence band. The photogenerated holes and photoexcited electrons will migrate to the surface of the photocatalyst and trapped there. The photogenerated holes then react with adsorbed water to produce strong oxidizing $\cdot\text{OH}$ radicals whereas the photoexcited electrons react with adsorbed oxygen to generate $\cdot\text{O}_2^-$ radicals [41].



The formation of these radicals will further react with recalcitrant pollutant and subsequently degraded the recalcitrant pollutant.



2.5 Strategies to improve g-C₃N₄ photocatalytic performance

In order to overcome the individual drawback of pure g-C₃N₄, many attempts have been made to improve the photocatalytic capability including surface modification of the photocatalyst structure. Generally, the surface modification aims to improve the photocatalyst specific surface area, charge separation and optical. There are currently five modification techniques which have been investigated including the introduction of heteroatoms (i.e. metals and non-metals) within g-C₃N₄ framework, noble metal deposition, hybridizing g-C₃N₄ with carbon nanomaterials and coupling g-C₃N₄ with a photocatalyst. The principle, advantages and disadvantages of each technique are summarized in **Table 1**.

On the other hand, the development of the heterostructure photocatalyst via the introduction of additional compounds into the g-C₃N₄ network is one of the most promising strategies to enhance the overall catalytic performance of g-C₃N₄ photocatalyst [22]. In general, the g-C₃N₄-based heterostructure can be developed by coupling the g-C₃N₄ with other types of photocatalyst as co-catalyst. The formation of the heterostructure with suitable band position would suppress the recombination rate of the photogenerated electron-hole pairs, which lead to higher

Modification method	Principle	Advantages	Disadvantages
Metal doping	Doping of various metallic species such as the alkali metals, rare earth metals and noble metals into g-C ₃ N ₄	Bandgap narrowing, surface area improvement, charge separation and fine-tuning the band structure	Can often cause secondary pollution due to leaching of the metal ions
Non-metal doping	Doping g-C ₃ N ₄ with non-metals	No secondary pollution, improve visible light absorption and charge separation	Non-metal species does not take part in charge transportation hence recombination centers are formed
Noble metal deposition	Deposition of noble metal nanoparticles such as Cu, Pt, Au and Pd on g-C ₃ N ₄	Metal content positively influence the photocatalytic activity until the optimum loading is reached	Beyond the optimum metal loading, the excess metal ions act as recombination centers for the electron/hole pairs
Hybridizing g-C ₃ N ₄ with carbon nanomaterials (CNM)	Carbon nanomaterial such as carbon nanotubes (CNTs), carbon nanospheres (CNS), graphene oxide (GO) and reduced graphene oxide (RGO)	High thermal, electronic conductivity, remarkable adsorption properties for organic and inorganic compounds	Excess CNM (i.e. RGO) facilitate adsorption of large amounts of the dye molecules onto the catalyst surface thereby reducing light penetration to the photocatalyst
Coupling g-C ₃ N ₄ with semiconductor	Coupling two or more semiconductors to form a semiconductor heterojunction	Improved stability, visible light utilization, charge separation and transfer and more efficient formation of the oxidizing species	Difficult to find a proper semiconductor photocatalyst with suitable band edge position

Table 1.

Surface modification of g-C₃N₄ technique to improve its photocatalytic performance.

photocatalytic performance efficiency. For example, Wang et al. [19] reported the enhanced photocatalytic performance of g-C₃N₄/ {010} facets BiVO₄ photocatalyst fabricated via ultrasonic dispersion method. The aforementioned heterostructure photocatalyst was capable of removing 88.3% of RhB pollutant within 30 minutes under visible light irradiation. Meanwhile, Huo et al. [20] successfully formed the BiVO₄/Polydopamine/g-C₃N₄ heterostructure photocatalyst via facile ultrasonic dispersion and self-assembly at the room temperature. They observed a remarkable photocatalytic degradation of glyphosate under visible light irradiation in comparison to the unmodified photocatalyst.

Furthermore, the photocatalytic activity of pure photocatalyst can be enhanced by the addition of carbon materials as an electron bridge mediator. The addition of carbon nanomaterial provides a structure with a larger specific surface area over which the active component can be well-dispersed, thus increasing the active sites. During the photocatalytic degradation of organic pollutants, carbon materials can be used as an adsorbent to improve the adsorption capacity of semiconductors. Besides, carbon materials can be doped as a photosensitizer for band gap narrowing, which is favorable for expanding the visible light absorption region of semiconductors. The incorporation of electron bridge mediator within the network of the heterostructure system will facilitate the migration of the electron transfer within the photocatalyst, leading to the enhanced charge separation efficiency and photocatalytic activity. In this sense, GO/RGO and CNTs are among the carbon nanomaterial that has been explored to acts as an electron bridge mediator.

3. Recent progress on the development of g-C₃N₄ photocatalyst for environmental remediation

Photocatalyst	Synthesized Method	Type of pollutants	Pollutants concentration	Source of light	Degradation efficiency	Ref.
ZnO/g-C ₃ N ₄	Impregnation	Phenol	5 mg/L	Simulated sunlight irradiation	99.5% of phenol removal achieved after 60 min of irradiation	[42]
BiVO ₄ /PDA/g-C ₃ N ₄	Ultrasonic dispersion and self-assembly	Glyphosate	0.1 mM	125 W mercury lamp	100% degradation after 150 minutes	[20]
g-C ₃ N ₄ /BiVO ₄	NR	Methylene blue	10 mg/L	Visible light	g-C ₃ N ₄ /BiVO ₄ photocatalyst was about 8 and 7 times higher than that of pure BiVO ₄ and g-C ₃ N ₄	[43]
g-C ₃ N ₄ /TiO ₂ /CNT	Facile hydrothermal method	MB	6 mg/L	150 W halogen lamp	Ternary hybrid exhibits 5 times higher photocatalytic activity compared to bare g-C ₃ N ₄	[44]

Photocatalyst	Synthesized Method	Type of pollutants	Pollutants concentration	Source of light	Degradation efficiency	Ref.
g-C ₃ N ₄ /AC	NR	Phenol	50 mg/L	350W Xe lamp	100% phenol removal after 160 min	[45]
g-C ₃ N ₄	Polycondensation	Pharmaceutical	20 mg/L 10 mg/L	35 W Xenon lamp	Photocatalyst showed the capacity to degrade the pollutants in the sequence: tetracycline > ciprofloxacin > salicylic acid > ibuprofen	[46]
g-C ₃ N ₄ /CNTs/Au	Ultra-sonication	Aqueous Rhodamine (RhB)	250 mL	Visible light	100 vol% Au dispersion with a 60: 40 mass ratio of g-C ₃ N ₄ to CNTs exhibited optimal photocatalytic activity	[47]

4. Conclusions and future directions

The growing concern over the scarcity of clean water sources due to a fast development of industrialization has forced a rapid breakthrough dedicated to the development of advanced photocatalyst systems. Over the past few years, the studies on g-C₃N₄ based photocatalysts have witnessed auspicious potential promises by this photocatalyst in environmental remediation applications. To date, the profound photocatalytic performance of g-C₃N₄ based photocatalyst is mainly governed by its intrinsic features such as metal-free photocatalyst and good light absorption properties owing to its medium band-gap energy of 2.7 eV. In this chapter, the synthesis, properties and photocatalytic application of g-C₃N₄ are summarized. Then, the most recent strategies for enhancing the photocatalytic performance of the g-C₃N₄ photocatalyst are highlighted.

Although profound performance had been reported in most of the recent studies, the promising potential of g-C₃N₄ based photocatalyst has yet to be exploited fully. The main challenges which are yet to be mitigated are (i) green synthesizing method which can produce high surface area and good photostability photocatalyst, (ii) the control of surface kinetics on g-C₃N₄ photocatalyst which can promote the photocharge separation and migration, (iii) the use of real industrial wastewater in analyzing the performance of g-C₃N₄ based photocatalyst, (iv) improving the reactor design to achieve the optimum photocatalytic performance with the lowest cost and (v) the utilization of real sunlight as a light source during the analysis process.

Acknowledgements

The authors would like to express their appreciation to the Chemical Engineering Department, Universiti Teknologi PETRONAS, Centre of Innovative

Nanostructures & Nanodevices (COINN), Universiti Teknologi PETRONAS and The Murata Science Foundation Grant (015ME0-033) for the financial and laboratory support. The authors would like to thank the Centre Analytical Lab, Universiti Teknologi PETRONAS for the sample characterization facilities.

Conflict of interest

The authors declare no conflict of interest.

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