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Review Article

Research Progress in Preparation and Modification Strategies of Graphitic Carbon Nitride for Efficient Photocatalysis: A Mini Review

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Abstract

With the acceleration of industrialization, energy shortage and environmental pollution are becoming more and more serious. It is urgent to develop green and sustainable new energy. Photocatalytic technology, especially the use of solar energy to convert chemical energy, has attracted much attention due to its environmental friendliness and sustainability. As an excellent photocatalyst, graphitic carbon nitride (g-C_3N_4) has emerged rapidly in the field of photocatalysis due to its low cost, simple preparation and non-toxicity. However, g-C_3N_4 has some limitations in practical applications, such as small specific surface area, weak photogenerated carrier transport capacity and narrow visible light response range. To overcome these challenges, researchers have modified g-C_3N_4 through various strategies to improve its photocatalytic performance. In this paper, the synthesis methods and modification strategies of g-C_3N_4 are summarized from the structural characteristics of g-C_3N_4, and its research focus and application prospect in the field of photocatalysis are prospected.

Introduction

While enjoying the convenience and progress brought about by industrialization, human society is also facing unprecedented energy shortages and environmental pollution [1]. This urgently requires us to develop green, clean and sustainable new energy sources to replace the increasingly depleted traditional fossil energy. Using photocatalytic technology to convert inexhaustible solar energy into chemical energy is a green, sustainable and promising solution [2,3]. As the core of this technology, the design and development of catalyst materials have always been the focus of research in this field. In 2009, graphitic carbon nitride (g-C_3N_4) was first reported to be able to catalyze hydrolysis to produce hydrogen under visible light irradiation [4]. After that, g-C_3N_4, as a catalyst with excellent performance, has rapidly become a research hotspot in the field of photocatalysis. g-C_3N_4 not only exhibits excellent photocatalytic efficiency, but also shows its great potential in practical applications due to its low cost, easy availability of raw materials, simple and fast preparation process, and cleanliness and non-toxicity [5-7].

Nevertheless, the monomeric g-C_3N_4 still presents certain challenges in practical applications. The specific surface area is small, which limits the contact area with the reactants [8]. The transport ability of photo-generated carriers is not strong, which affects the efficiency of photocatalytic reaction [9]. In addition, its response range to visible light is narrow, usually less than 460 nm, which further limits its energy conversion efficiency in the photocatalytic process [10]. These shortcomings largely restrict the application of g-C_3N_4 in a wider range of fields. To overcome these limitations, researchers have continuously explored effective methods to modify g-C_3N_4 to improve its photocatalytic performance [2,5-7,11]. In this paper, we will systematically summarize the main synthetic preparation methods and modification strategies of g-C_3N_4 in terms of its structural properties and fundamental properties. On this basis, the research focus and future development direction of g-C_3N_4 in the field of photocatalysis will be further explored, with a view to providing new perspectives and ideas for solving energy and environmental problems.

Structural Properties of g-C3N4

In 1834, Berzelius and Liebig synthesized for the first time a polymer type of carbon nitride and named it "Melon" [12]. At that time, this polymer was considered to be a classical carbon-nitrogen isomer, known for its stability. However, due to its insoluble and chemically inert nature, carbon nitride did not receive enough attention from scientists for a long time. Until 1989, A.Y. Lin and M.L. Cohen predicted the existence of β-C_3N_4 based on the crystal structure of β-Si_3N_4 by theoretical calculations and creatively replacing silicon with carbon [13]. They pointed out that this material has the potential to become harder than diamond, thus introducing the concept of "g-C_3N_4" for the first time. The monolayer structure of g-C_3N_4 is composed of a triazine ring (C_3N_4) or a heptazine ring (C_6N_7), in which carbon atoms and nitrogen atoms are connected by sp^2 hybridization to form a stable ring structure [3]. These rings are connected by amino groups to form a continuous π-electron conjugated system, as illustrated in Figure 1. The distinctive properties of g-C_3N_4 are attributable to its distinctive energy band structure and chemical composition. These attributes confer exceptional thermodynamic stability and enable the material to exhibit robust photocatalytic activity even under elevated temperatures [14,15]. Furthermore, its exceptional chemical stability allows g-C_3N_4 to perform effective photocatalysis in specific environments, such as acidic or alkaline conditions, which makes it a promising candidate for applications in the field of photocatalysis [16].

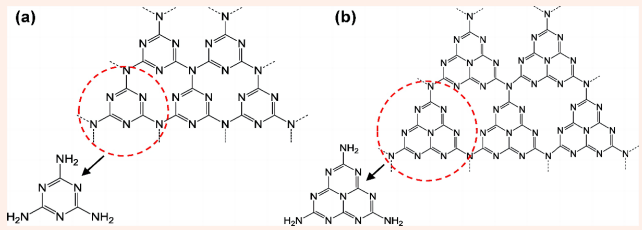

Figure 1: Schematic diagram of g-C_3N_4 structure. (a) triazine ring structure, (b) heptazine ring structure [3].

The Preparation Method of g-C_3N_4

High temperature calcination method

The high-temperature calcination method is a well-established synthetic technique that offers significant advantages in the preparation of g-C_3N_4 materials [17]. These advantages include controllable conditions, ease of operation and low cost. In this method, the most used raw materials are nitrogen-containing compounds, including melamine, thiourea and urea [18-20]. During high-temperature calcination, these monomers are excited to generate free radicals under the effect of high temperature, which in turn triggers the polymerization reaction to form g-C_3N_4 [21]. However, it is worth noting that different feedstock choices and reaction conditions can lead to significant differences in the physicochemical properties of the final products. For example, Jorge et al. [22] found in their study that the C/N ratio and specific surface area of g-C_3N_4 prepared using melamine and dicyandiamide as precursors change with increasing reaction temperature. These changes directly affect the catalytic properties of the material. Sun et al. [23] prepared fluorine-doped g-C_3N_4 (FCN) by subjecting melamine to high-temperature calcination in a fluorinated atmosphere. The experimental results demonstrated that the pore structure of FCN exhibited a gradual increase with the elevation of fluorination temperature. FCN-150, obtained under the fluorination condition at 150 °C, exhibited a hydrogen production efficiency that was 11.6 times higher than that of undoped g-C_3N_4. However, when the fluorination temperature was increased to 180 °C, the porous structure of FCN-180 exhibited a significant deterioration, accompanied by a notable reduction in thickness. This suggests that fluorination at elevated temperatures may potentially lead to structural damage to g-C_3N_4.

Self-assembly method

The self-assembly technique, as an innovative nanomaterial preparation method, shows unique advantages in synthesizing g-C_3N_4 with two-dimensional nanosheet structures [24]. The g-C_3N_4 prepared by this method usually has a planar few-layer or monolayer nanosheet morphology [25]. These nanosheets not only possess a large specific surface area but also a large pore size [26]. These properties are crucial for improving the photocatalytic performance of the materials. Li et al. [27] employed a closed self-assembly method to synthesize highly crystalline g-C_3N_4 nanosheets. The nanosheet structure not only optimizes the separation efficiency of photogenerated carriers, but also significantly reduces the charge transfer resistance, thus substantially enhancing the photocatalytic activity. In particular, the hydrogen precipitation efficiency of these highly crystalline nanosheets was increased by a factor of six in comparison to that of bulk g-C_3N_4. Zhang et al. [28] prepared multilayered g-C_3N_4 by electrostatic self-assembly, which effectively increased the number of photocatalytic active sites and further improved the efficiency of carrier separation. In the presence of visible light, the multilayered g-C_3N_4 displays markedly enhanced hydrogen precipitation activity and apparent quantum efficiency.

Solvothermal method

The solvothermal method, as a derivative of the hydrothermal synthesis technique, has become a common means of preparing g-C_3N_4 materials [29]. This method is favored for its simplicity, ease of control and ability to effectively prevent the volatilization of toxic substances [30]. It promotes the transformation and recombination of precursor substances by applying high temperature and pressure conditions in a solvent to obtain materials with specific structures and properties. Wang et al. [30] prepared oxygen-doped modified g-C_3N_4 (CNO) using an innovative pre-hydrothermal treatment method, in which dicyandiamide was first hydrothermally treated with water as solvent at 180 °C. Subsequently, porous O-doped graphitic phase carbon nitride (P-CNO) with highly ordered structure was obtained by thermal polymerization. P-CNO exhibited a significantly enhanced photocatalytic hydrogen precipitation rate compared to undoped g-C_3N_4. In contrast, Hu et al. [31] went in the opposite direction first mixing thiourea and ammonium hydrogen phosphate as precursors and then thermal polymerization to prepare preliminary g-C_3N_4 materials. Subsequently, these preliminary products were again subjected to hydrothermal reaction to successfully prepare phosphorus and sulfur co-doped g-C_3N_4 nanorods (P-SN). This co-doping strategy not only significantly increased the specific surface area of the material, but also reduced the band gap energy, thus improving the separation efficiency of photogenerated electrons and holes.

Modification Strategy of g-C_3N_4

Despite the advantages of excellent thermochemical stability and low raw material cost, g-C_3N_4 has some significant limitations for photocatalytic applications. For example, large forbidden band width (2.7 eV), narrow response range to visible light, which is generally less than 460 nm; high photogenerated electron-hole pair complexation rate, and small specific surface area, and so on [8-10,32]. To address these challenges, researchers have explored a series of various modification strategies, including doping modification, vacancy defect modulation, and semiconductor composite, with the aim of enhancing the photocatalytic performance of g-C_3N_4. These methods have enhanced the photocatalytic performance of g-C_3N_4 to different degrees and opened new possibilities for its application in photocatalysis.

Doping modification

The strategic incorporation of energy traps between the valence and conduction bands of g-C_3N_4 enables a notable expansion of its visible light absorption capacity and enhances the separation efficiency of photogenerated electron-hole pairs [33]. This is crucial for enhancing the photocatalytic performance. The doping modification strategies can be broadly classified into three categories: non-metallic doping, metal doping, and molecular doping. Non-metal doping modified g-C_3N_4 by introducing Oxygen (O), Carbon (C), Phosphorus (P), Sulfur (S), Boron (B), Lithium (Li), Fluorine (F) and other anions. The doping of these elements not only modulates the electronic structure of the material, but also effectively broadens its absorption range of visible light [34,35]. Metal doping is achieved by introducing transition metal ions with multiple valence states, including Cobalt (Co), Zinc (Zn), Copper (Cu), Iron (Fe), and Nickel (Ni), into the g-C_3N_4 lattice. This introduces additional energy levels to the material, enhancing the ability to capture photons and improving the photocatalytic efficiency [36]. In contrast, molecular doping aims to enhance performance by modifying the molecular chain structure of the g-C_3N_4 polymer itself [37].

For example, Faisal et al. [38] investigated the doping effect of gold nanoparticles (Au NPs) of different weight percentages (0.2-5.0%) on g-C_3N_4 (Au/g-C_3N_4). As shown in Figure 2, the uniform distribution of Au on g-C_3N_4 produced small-sized NP junctions, which directed the photogenerated electrons to the surface efficiently, thus enhancing the effective charge separation process and improving the photocatalytic efficiency. Chu et al. [39] significantly promoted the charge separation on the heptazine ring and attracted photoexcited electrons through the modification of g-C_3N_4 with P, S, and O co-doping, thereby enhancing the photocatalytic degradation activity of doped g-C_3N_4 for methyl blue. Yang et al. [40] synthesized hydrophilic bifunctional layered structures by assembling B-doped g-C_3N_4 nanosheets. This layered B-doped g-C_3N_4 was able to take full advantage of its significantly enhanced visible light absorption and photogenerated carrier separation efficiency in aqueous media to exhibit excellent photocatalytic performance, which greatly promotes the potential of g-C_3N_4 photocatalysts in practical applications.

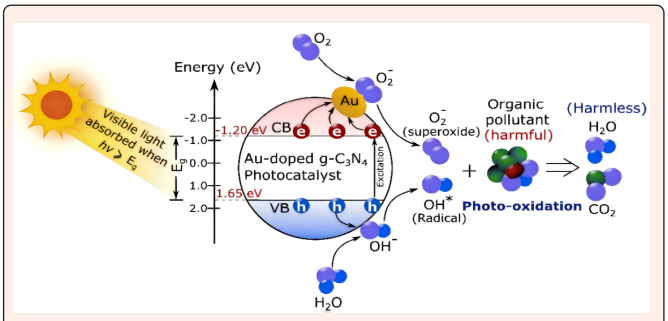

Figure 2: The mechanism diagram of Au doped g-C_3N_4 photocatalyst [38].

Vacancy defect regulation

It has been demonstrated that the electronic properties of g-C_3N_4 can be significantly modified by introducing carbon and/or nitrogen vacancy defects into its crystal structure, thereby enhancing its photocatalytic performance [41]. In particular, the introduction of nitrogen vacancies has been demonstrated to exert a beneficial influence on the photocatalytic activity of the material [4]. On the one hand, nitrogen vacancy defects facilitate the capture of photoexcited electrons, thereby inhibiting the complexation of photogenerated electrons and holes. This enhances the charge separation efficiency in the photocatalytic process. On the other hand, the surplus electrons present within the nitrogen vacancies can react with oxygen molecules, resulting in the generation of superoxide radicals that exhibit high reactivity. Furthermore, it can be combined with metal substances to form capture sites for photogenerated electrons, thereby promoting the photocatalytic reaction [42].

Li et al. [9] prepared ultrathin g-C_3N_4 nanosheets rich in N vacancies (Nv-rich-CN) by employing two distinct molecular self-assembly and molecular embedding strategies. As illustrated in Figure 3, the incorporation of N-vacancy-rich nanosheets not only altered the electronic structure of the material, but also resulted in electronic distortions at the local level and an expansion of the region encompassing the disordered energy gradient. This, in turn, facilitated the adsorption of CO_2 molecules by the CN units in proximity to the nitrogen vacancies, thereby enhancing the photocatalyst's capacity to reduce CO_2. Zheng et al. [43] introduced Boron Nitrogen vacancy (BN) aggregates in g-C_3N_4. The introduction of this structure weakened the strong exciton effect in g-C_3N_4 and promoted the spontaneous dissociation of bound excitons into free charge carriers under ambient conditions. The g-C_3N_4 composite with BN aggregates displays remarkable photocatalytic efficacy in the visible-light-driven two-electron oxygen reduction reaction.

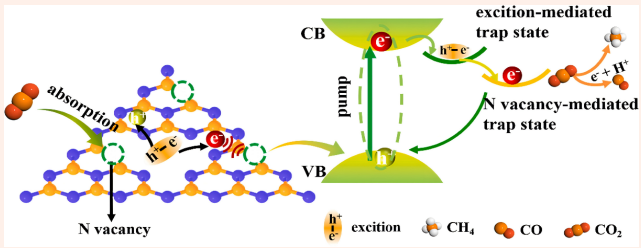

Figure 3: Schematic of the photocatalytic reduction of CO_2 for Nv-rich-CN [9].

In contrast to N vacancies, the introduction of carbon vacancies narrows the band gap by increasing the Valence Band (VB) energy of g-C_3N_4 and exciting more electrons. This structural change breaks the symmetry of the C-N bond and forms unsaturated N atoms [44]. These unsaturated N atoms act as paramagnetic centers and can attract electrons in the conduction band, producing an electron delocalization effect. This promotes the effective separation of electrons and holes, which further enhances the photocatalytic performance [45]. Liu et al. [46] prepared g-C_3N_4 with a certain number of carbon defects by using the affinity of single atom Pd and N atoms. As shown in Figure 4, the presence of Pd atoms significantly improves the separation and transport of the photogenerated charge carriers, which enables this g-C_3N_4 to exhibit a high and stable photocatalytic activity in the conversion of NO.

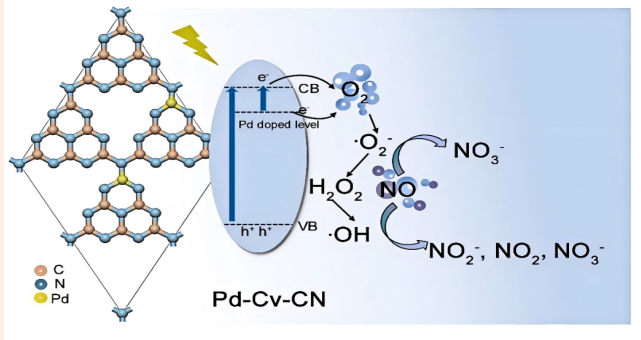

Figure 4: Light-induced charge separation and proposed mechanism for photocatalytic oxidation of NO over Pd-Cv-CN [46].

Semiconductor composite

Compositing the semiconductor matching the energy band of g-C_3N_4 with it can effectively inhibit the photogenerated electron-hole complexation and thus significantly improve the photocatalytic performance of g-C_3N_4 (Figure 5), [47]. The composite systems of semiconductors and g-C_3N_4 cover multi-component oxides ($BiWO_4$, $ZnFe_2O_4$, $DyVO_4$, etc.), metal sulphides ((MoS_2, CdS, $ZnIn_2S_4$, etc.), metal oxides (ZnO, Fe_2O_3, CeO_2, etc.), and silver-based semiconductors (AgCl, AgBr, Ag_2S, etc.) [48]. Li et al. [49] successfully prepared a composite photocatalyst of g-C_3N_4 and Bi_2WO_6 by a hydrothermal method and applied it to the photocatalytic degradation of methyl orange. This composite system effectively separated the photogenerated electron-hole pairs at the interface of g-C_3N_4 and Bi_2WO_6 through the synergistic effect of semiconductors, which increased the specific surface area and stability. The experimental results showed that the photocatalytic activity of this composite photocatalyst was nearly 2.7 and 8.5 times higher than that of g-C_3N_4 and Bi_2WO_6 alone, respectively. Wang et al. [50] constructed 0D/2D Pt-C_3N_4/CdS heterojunctions by uniformly dispersing CdS quantum dots on g-C_3N_4 nanosheets. This Z-type heterojunction enhanced the charge transfer and separation by regulating the carrier transfer and exhibited excellent photocatalytic hydrogen production performance. Jiang et al. [51] constructed a Z-type heterojunction ZnO/g-C_3N_4 photocatalyst. The catalyst significantly improved the separation efficiency of photogenerated electrons and holes, which enabled more photogenerated electrons to combine with O_2 to generate -O_2^-, and then effectively oxidized benzyl alcohol to benzaldehyde.

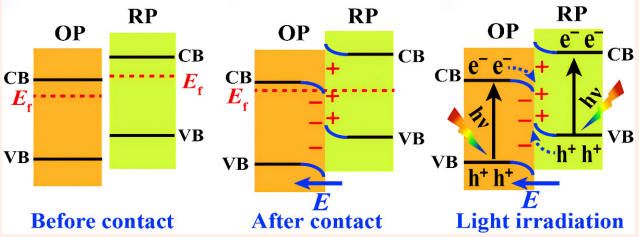

Figure 5: Charge transfer processes in heterojunctions with staggered band structures [47].

Shang et al. [52] prepared an S-type heterojunction photocatalyst AgCl/g-C_3N_4 for photocatalytic hydrogen production. In comparison to AgCl, the conduction band and valence band positions of g-C_3N_4 are higher, while the work function is smaller. This results in the formation of an internal electric field from g-C_3N_4 to AgCl, which in turn causes band bending. This synergistic effect results in the recombination and elimination of photogenerated electrons in the conduction band of AgCl and holes in the valence band of g-C_3N_4. The photogenerated electrons in the conduction band of g-C_3N_4 and the holes in the valence band of AgCl are retained, thereby participating in the reduction and oxidation reactions, respectively. This significantly improves the photocatalytic hydrogen evolution rate.

Summarized and Prospected

In conclusion, the unique energy band structure and chemical structure of g-C_3N_4 give it excellent stability and photocatalytic properties. At the same time, the many advantages of g-C_3N_4, such as easy availability of raw materials, non-toxicity, low manufacturing cost and green environmental protection, make it possible to prepare it in various ways. However, its inherent defects such as small specific surface area, poor photogenerated carrier transport ability, and narrow visible light response range greatly limit its application in practice. Researchers have improved the photocatalytic performance of g-C_3N_4 through various methods. Despite many successes, some challenges still need to be addressed in future work. For example, the controllability of morphological and structural modulation is poor, the mechanism of photocatalytic enhancement by elemental doping is unclear, the operation of semiconductor composite is complicated and costly, and there are limitations in the single modification approach.

Therefore, future research efforts should address the core content of g-C_3N_4 research in the following aspects: (1) Investigate in-depth the physicochemical mechanisms in the modification process through a combination of experiments and theoretical calculations to provide guidance for the material design. (2) To develop new preparation methods with strong controllability of morphology and structure, and ensure its yield, and promote the application of industrialization. (3) To further carry out the research on multivariate composite modification and deepen its mechanism to

improve the performance of g-C_3N_4 and enrich its application in photocatalysis. With a deeper understanding of g-C_3N_4 and the advancement of the modification technology, we believe that g-C_3N_4 will play an even more important role in the future photocatalysis field and provide an effective solution to solve energy and environmental problems.

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