




Sustainable green approach of simple hydrothermal treatment of g-C₃N₄ to boost the CO₂ conversion into methanol through photoelectrocatalysis

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ABSTRACT

The carbon dioxide (CO₂) transformation into beneficial fuels or chemicals by photoelectrocatalytic (PeC) reaction has been proved sustainable. As a standalone or component in composites, the graphitic carbon nitride (g-C₃N₄) catalysts were significantly utilized for PeC approaches. In this research work, as a first instance, a sustainable green approach of simple hydrothermal treatment at various temperatures, such as 90, 120, 150, and 180 °C, was adopted to synthesize g-C₃N₄ (xgCN) catalysts to enrich their PeC CO₂ reduction into methanol activity. The simple hydrothermal treatment achieved increased stacking layer distance of g-C₃N₄ and fine-tuning of the band gap. The 120gCN catalyst (treated at 120 °C) exhibited a superior current at -1.0 V of 3.62 mA cm⁻² and a 4.39 μmol L⁻¹ h⁻¹ cm⁻² methanol generation in PeC CO₂ reduction reaction, which was around twofold superior compared to that of PgCN (untreated g-C₃N₄) catalyst. The excellent efficiency of the 120gCN catalyst was credited to its better production and lesser electron (e⁻)-hole (h⁺) pairs recombination rate.

Introduction

Developing efficient catalysts for photocatalytic (PC) and/or photoelectrocatalytic (PeC) energy conversion or utilization for valuable works has gained extensive

research interest because of their assistance in addressing the world's energy and environmental issues [1–3]. Different types of photocatalysts, including those based on metal oxides, metal–organic frameworks, graphitic carbon nitride (g-C₃N₄), composites, etc.,

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have been demonstrated for photocatalytic environmental decontamination and other energy applications [4–7]. For the sustainable carbon dioxide (CO₂) conversion into useful products, the PeC process, which involves both PC and electrocatalytic principles, has been proved to be a constructive method [8]. Nevertheless, several new types of photocatalysts have been explored in recent decades, and the efficiency enhancement of promising existing photocatalytic materials remains essential [9–11]. g-C₃N₄, the semiconductor of non-metal type, has been widely studied for several PC and PeC applications, which include organic compound transformations, degradation of pollutants, hydrogen generation, and so on [12, 13]. The g-C₃N₄, having graphitic-like morphology, has played a remarkable role in composite catalysts for achieving significant performances for PC or PeC efficiencies [14, 15], including for the CO₂ conversion into value-added products [16–18].

The properties, for example, good stability, appropriate band edge positions and band gap, and easy preparation methods, have gained additional superiority of the g-C₃N₄ [19]. Besides its significant performance as a standalone catalyst and as a component in composite materials, the fast electron (e⁻)-hole (h⁺) pairs recombination and the heavy agglomeration layers nature of the bulk g-C₃N₄ are detrimental to efficient PeC activities [20]. Many methods, such as coupling with another semiconductor [16], doping with elements [21, 22], and so on, were attempted to increase the g-C₃N₄ performances. However, the composite catalysts' performance could still be better [17, 23]. The potential improvement of pristine g-C₃N₄ should be explored as it is a key building block for many potential catalytic materials. Several types of treatment of pristine g-C₃N₄, such as sonication at higher temperatures with organic solvents [24], sonication at room temperature with water [25], and treatment with a solution of NaOH [26], have been reported to improve their efficiencies. However, the above procedures involve harsh conditions and some chemicals, which can be considered non-environmentally friendly. Therefore, the significant problems of pristine g-C₃N₄, such as rapid charge recombination, increased agglomeration layers, and a non-environmentally friendly synthesis treatment route, should be addressed. Also, PeC enhancement of pristine g-C₃N₄ for converting CO₂ into valuable chemicals or fuels has not been explored so far. However, a variety of methods, such as doping with heteroatoms, deposition

of cocatalysts, and formation of composites, have been widely employed to improve the PeC CO₂ reduction performances of g-C₃N₄ catalysts; these methods involve complex synthetic routes and utilize harsh chemical conditions. Therefore, it remains challenging to enhance the charge separation and transfer behavior of the neat g-C₃N₄ to achieve better PeC CO₂ reduction activities. Particularly, the role of simple and dopant-free modification of g-C₃N₄ to improve selective PeC CO₂ reduction into methanol has not been investigated systematically.

Herein, for the first time, we demonstrate a sustainable green approach using simple hydrothermal treatment at various temperatures to prepare g-C₃N₄ (xgCN) catalysts and their enhanced PeC CO₂ reduction into methanol. In addition to its environmental compatibility, this strategy enables the controlled modification of neat g-C₃N₄ without using or adding any additional chemical components, thus providing a clear correlation between structure and property performance. This work hence revealed fundamental insight into how mild structural modification of neat g-C₃N₄ improved charge separation and transfer behavior, and PeC CO₂ reduction performances. The hydrothermal treatment conditions of g-C₃N₄ play a crucial role in the PC and PeC activity of the catalysts [24–28]. Sano et al. demonstrated the significantly improved morphological and PC NO oxidation properties of g-C₃N₄ by hydrothermal treatment in the range from 90 to 150 °C [26]. Zhai et al. reported that higher hydrothermal treatment conditions, ranging from 250 to 300 °C, improved the morphological and PC properties of g-C₃N₄; however, partial decomposition of g-C₃N₄ occurred at higher temperatures [27]. Ming et al. also demonstrated that the mild hydrothermal treatment temperature of the oxidized g-C₃N₄ improved the photocurrent generation activity by around ten times [28]. Therefore, to systematically study the effects of hydrothermal conditions on the structural, electrical, and PeC CO₂ reduction properties of xgCN, we selected a range of treatment temperatures (mild, mid, and relatively high) such as 90, 120, 150, and 180 °C. The UV-DRS and XRD results showed the fine-tuning of the band gap and increased stacking layer distance of g-C₃N₄ on mild hydrothermal treatment, respectively. The PeC CO₂ reduction activity of 120gCN catalyst (hydrothermally treated at 120 °C) showed twofold superior photocurrent density and methanol generation compared to the PgCN (as-prepared). The exceptional activity of the 120gCN

bulk g-C₃N₄ was demonstrated by the B-doped g-C₃N₄ catalyst developed using an ultrasonic-assisted process. The improved performance was attributed to the isolation of charges and increased charge transportation potential of the catalyst [53]. Similarly, C-doped g-C₃N₄ catalyst prepared by a double-stage polymerization procedure showed 4.6-fold higher PC CO₂ reduction to CO compared to the pure g-C₃N₄ owing to its higher generation and effective trapping of e⁻ and h⁺ [54]. The reports in Table 1 [47–54], which employ acid modification, heteroatom doping, multistep polymerization, and coupling with other components, are not considered sustainable because they use toxic chemicals and harsh experimental conditions to synthesize the catalysts. Additionally, the PeC process was rarely adopted for converting CO₂. Thus, the adopted sustainable green approach resulted in higher photogeneration and reduced recombination of pairs of e⁻-h⁺ and showed around twofold greater reduction activity of PeC CO₂ to methanol than that of untreated g-C₃N₄. From a sustainability outlook, the hydrothermal treatment strategy employed in this work eliminates the utilization of any toxic chemicals as dopants or solvents, or high value metals which are generally used to improve the activity of g-C₃N₄ in the literature. Therefore, this approach can be considered as simple and environmentally friendly. Besides, it should also be noted that the methanol yield efficiency observed in the current research work is comparable with the reported g-C₃N₄-based catalysts synthesized using toxic chemicals and harsh experimental conditions. Therefore, the current simple and green approach represents a trade-off as the achievement of enhanced activity with reduced complex synthetic route and environmental impact. Additionally, the g-C₃N₄ synthesized using a water and thermal treatment procedure in a range of 90 to 180 °C can be considered a scalable and eco-friendly PeC CO₂ conversion pathway. This work may help bridge the gap between high productivity and sustainability by applying this simple and green approach with other promising catalysts or interface engineering to develop an alternative potential catalyst.

Conclusion

A sustainable green approach of the simple hydrothermal process without using any chemicals was adopted to treat the as-prepared g-C₃N₄ under different

temperatures such as 90, 120, 150, and 180 °C. The low angle shifts in the XRD peak revealed the increased stacking layer distance of the hydrothermally treated g-C₃N₄ catalysts (xgCN), which was verified using the microscopic images of agglomerated sheet structure of PgCN and sheet structures of xgCN samples. The fine-tuning of the band gap and suppressed rate of recombination of e⁻-h⁺ of xgCN catalysts were confirmed using the UV-Vis DRS (optical absorption) and PL results, correspondingly. Therefore, refined PeC CO₂ conversion into methanol formation activity was demonstrated over some of the prepared xgCN photocathodes. The 120gCN possessed a 3.62 mA cm⁻² of superior photocurrent density among all the prepared catalysts, which was around two times enhanced than the PgCN (untreated g-C₃N₄) photocathode. Similarly, a superior of 4.39 μmol L⁻¹ h⁻¹ cm⁻² of methanol generation from PeC CO₂ reduction was attained using the 120gCN compared to the other photocathodes, twofold higher than the PgCN. The superior performance of 120gCN catalyst was recognized to their improved photogeneration and suppressed recombination rate of e⁻-h⁺ pairs. Hence, this work demonstrated the enhancement of methanol generation through the PeC reduction of CO₂ and fine-tuning of optical and morphological parameters of the g-C₃N₄ (as-prepared) by a sustainable green approach of an effortless mild hydrothermal treatment.

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Author contributions

Komatireddy DamodarReddy was involved in methodology, data curation, investigation, writing—original draft, and writing—review and editing. Prabhu Saravanan took part in methodology, conceptualization, investigation, writing—review and editing, writing—original draft, funding acquisition, and formal analysis. Pavan. P. Gotipamul participated in data curation and writing—review and editing. Cristian H. Campos was responsible for methodology, formal analysis, conceptualization, and writing—review and

editing. Aravindhan Selvaraj contributed to investigation and writing—review and editing. Siva Chidambaram was responsible for investigation, conceptualization, writing—original draft, and writing—review and editing.

Data availability

The data can be made available on reasonable request.

Declarations

Conflict of interest The authors declare no conflict of interest.

Ethical approval Not applicable.

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