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# **Priority Communication**

# Solar-driven conversion of arylboronic acids to phenols using metal-free heterogeneous photocatalysts



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#### 1. Introduction

Solar energy as an abundant, sustainable, and environmentfriendly energy may be beneficial to solve the problem of global energy shortage. The vast majority of energy on earth is derived from solar energy, but it cannot be employed directly, and must be captured and converted into a useful form of energy. Meanwhile, solar energy is a thin energy source (the solar constant,  $I_{sc} = 1367 \text{ W/m}^2$  [1], and the obtained energy is proportional to the light receiving area. Fortunately, solar energy has the advantage of being widely distributed on the earth. In order to make efficient use of solar energy, two problems should be resolved: (1) find out how to convert sunlight into a usable form; (2) explore the operation of the light-harvesting complex. The photosynthesis of nature plants provides a good reference for researchers, which sunlight is initially absorbed by chloroplast containing chromophores, followed by energy transfer to a dedicated reaction center, and finally the captured energy is converted into chemical energy. The green leaves with elaborate hierarchical porous structures and functional components have a large receiving area and can enhance the light harvesting property. Photovoltaic cells with large light receiving area are one of the main forms for the solar energy utilization [2]. They can condense thin solar energy into electrical energy, which is an easy-to-use form of energy (Scheme 1) [3].

### ABSTRACT

Solar-driven conversion of arylboronic acids to phenols was achieved by employing graphitic carbon nitride (g- $C_3N_4$ ) as heterogeneous photocatalyst, where  $O_2^-$  was the main active species. By loading g- $C_3N_4$  onto the easy weaving low melting point sheath-core composite polyester fibers (LMPET), g- $C_3N_4$ -based artificial photosynthetic catalytic fabric (g- $C_3N_4/LMPET$ ) with a large light receiving area was prepared. It displayed the efficient conversion of arylboronic acid and excellent recycling performance. This system offers more possibilities to construct an artificial photosynthetic system with excellent solar-to-chemicals conversion efficiency.

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Therefore, it is highly desired to design a system that simulates the energy conversion and structural characteristics of the green leaves, which can effectively convert sunlight into other energy that can be utilized.

Photocatalysis has aroused intensive interests from the perspective of solar energy utilization, as it provides a green and potential route for the wide applications in the degradation of pollutants [4], water splitting [5], and CO<sub>2</sub> reduction [6]. Besides, the visible-light-induced organic transformation is also an important research field due to its high efficiency and environmentally friendly reaction conditions. Phenols are essential ingredient in the synthesis of natural products and organic chemistry. The oxidative hydrolysis of arylboronic acids is an effective way for the preparation of phenols, which often employs transition-metal catalysts such as copper [7], palladium [8], gold [9], ruthenium [10], and silver [11]. It is feasible for the arylboronic acids transformation to phenols by photocatalysis [12] and several methods have been reported in recent times using ruthenium complex [13], iridium complexes [14], methylene blue [15], and 2chloroanthraquinone [16] as photocatalysts. However, most catalysts used in the conversion of arylboronic acids to phenols are small molecules or powdery, and it is prerequisite to find out a method to assemble the photocatalysts onto the carrier to increase the light receiving area like photovoltaic cells and the convenience of recycling for the conversion of arylboronic acid to phenol under solar irradiation.



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 $\label{eq:Scheme 1. Scheme 1. Sche$ 

Graphitic carbon nitride  $(g-C_3N_4)$  as the organic semiconductor material is a metal-free and visible-light-responsive heterogeneous photocatalyst with low cost, chemical stability and appealing electronic band structure [17]. It can be easily synthesized by pyrolysis of cheap precursors like melamine, urea and dicyandiamide. g-C<sub>3</sub>N<sub>4</sub> has also been exploited for the synthesis of value added commodities and complex organic molecules [18]. However, powdery g-C<sub>3</sub>N<sub>4</sub> is inclined to aggregate and deposit in solutions, restricting its application. It is necessary to find an appropriate carrier to immobilize g-C<sub>3</sub>N<sub>4</sub> to avoid the time consuming trouble of filtration and recovery. Fiber materials possess the characteristics of the large surface area and easy weaving [19]. Taking advantage of these superiorities, g-C<sub>3</sub>N<sub>4</sub> was loaded onto the low melting point sheath-core composite polyester fibers (LMPET) to prepare g-C<sub>3</sub>N<sub>4</sub>-based artificial photosynthetic catalytic fabric (g-C<sub>3</sub>N<sub>4</sub>/ LMPET) with large light receiving area, which is mimicking the role of leaves in natural plants for the two-dimensional planar reaction system.

Herein, g-C<sub>3</sub>N<sub>4</sub> and g-C<sub>3</sub>N<sub>4</sub>/LMPET fabric dominated by superoxide radicals ( $\cdot$ O<sub>2</sub>) was firstly reported for the conversion of arylboronic acids to phenols with isopropanol (2-PrOH) as solvent in the presence of N,N-diisopropylethylamine (iPr<sub>2</sub>NEt) under the visible light or solar irradiation (Scheme 1). g-C<sub>3</sub>N<sub>4</sub>/LMPET with large receiving light area simulates the structural characteristics of green leaves and solves an intrinsic problem of powdered catalyst separation from the reaction mixture. The characterization data of g-C<sub>3</sub>N<sub>4</sub> and g-C<sub>3</sub>N<sub>4</sub>/LMPET, such as SEM images, DSC curves, FTIR spectra, DRS spectra, XRD and EPR spectra, can be found in the Supporting Information, Figs. S1–S8. g-C<sub>3</sub>N<sub>4</sub> and g-C<sub>3</sub>N<sub>4</sub>/LMPET fabric as the metal-free heterogeneous photocatalysts both show high selectivity in the reaction of arylboronic acid to phenolic compounds. The reaction takes place under mild conditions, air, ambient temperature and pressure.

#### 2. Materials and methods

In a typical reaction, 10 mg of g-C<sub>3</sub>N<sub>4</sub> or about 250 mg g-C<sub>3</sub>N<sub>4</sub>/ LMPET (containing 3.18% g-C<sub>3</sub>N<sub>4</sub>), 0.5 mmol of N,Ndiisopropylethylamine (iPr<sub>2</sub>NEt), and 0.1 mmol of boronic acid were added to 5 mL of isopropanol (2-PrOH) in the reactor. When g-C<sub>3</sub>N<sub>4</sub> was used as the photocatalyst, the reaction mixture was illuminated by a CEL-HXF300 Xenon lamp with a UV filter (filtered light at  $\lambda > 400$  nm; CEL-HXF300, Beijing Education Au-light Co., Ltd, China) under magnetic stirring. For post-reaction analysis, the g-C<sub>3</sub>N<sub>4</sub> was separated from the reaction mixture through filtration. The filtered liquid was then quantitatively analyzed through a GC (Agilent 6890N) equipped with a mass spectrometric detector (MS, Agilent 5973) and Agilent Technology HP-5MS capillary column (30 m × 0.25 mm × 0.25 mm) using high-purity He as the carrier gas and dodecane as the internal standard. When g-C<sub>3</sub>N<sub>4</sub>/ LMPET was used as the photocatalyst, the reaction mixture was then illuminated by a SUN-Q-Light photoreactor (Q-Lab Corporation, Xe-1-BC, USA) under shaker oscillation. The filtered liquid was then quantitatively analyzed through ultra-performance liquid chromatography with a PDA detector (UPLC, Waters) and a HSS T3 column (1.8  $\mu$ m, 2.1  $\times$  100 mm) using acetonitrile and water as the mobile phase by external standard method.

## 3. Results and discussion

Initially, the hydroxylation of 4-methoxyphenylboronic acid (0.1 mmol) was chosen as a model reaction for the optimization of reaction conditions. First, the optimal reaction time of 4methoxyphenylboronic acid to 4-methoxyphenol was evaluated, and the yield of phenol increases with the prolonged irradiation time (Table 1, entries 1–4). To our surprise, g-C<sub>3</sub>N<sub>4</sub> showed satisfying catalytic activity after 12 h with visible light irradiation at room temperature, giving the product 2a in 97% yield. Then several reactions were carried out as controls and to explore the effects of solvents and amines. First, we used different solvents including MeOH, MeCN, DMSO, DMF (Table 1, entries 5-8), to see the best conversion. While 2-PrOH has given best results, reaction in other solvents has not afforded good results. Moreover, the reaction did not take place when the photocatalyst was not present (Table 1, entry 9). When Et<sub>3</sub>N was added as the amine, the reaction gave the product in lower yields of 82% (Table 1, entry 10), and we found that iPr<sub>2</sub>NEt (5.0 equiv) was the ideal amine. Interestingly, the reaction gave no yield in the absence of iPr<sub>2</sub>NEt (Table 1, entry 11), which might act as a sacrificial electron donor. Then the reaction was carried out under O<sub>2</sub> and N<sub>2</sub> atmosphere, respectively. It was found that the reaction proceeded smoothly under O<sub>2</sub> conditions but with a yield of 92% (Table 1, entry 12). However, the product was formed under N<sub>2</sub> atmosphere but with very poor yield of 16% (Table 1, entry 13). Additionally, the superoxide radical capture agent 4-benzoquinone (BQ) was added as the scavengers of super oxide radical ( $\cdot O_2^-$ ), the reaction could proceed in the presence of BQ but gave the 4-methoxyphenol in low yields (21%). It suggested that the key step in the oxidative of arylboronic acids was the generation of the superoxide radical  $(O_2)$  and it was further proved by EPR. As can be seen in Fig. S8A, the DMPO- $O_2^-$  signal could be detected when the g-C<sub>3</sub>N<sub>4</sub> were irradiated for 2 min, while no signals appeared without light irradiation. It also indicates that  $O_2^-$  plays a major role for the hydroxylation of boronic acid in this study, similar to previous reports. When using other photocatalysts, TiO<sub>2</sub> gave only the desired phenol in 29% yield (Table 1, entry 15) and the reaction could hardly proceed in the presence of WO<sub>3</sub>. Thus, we determined the optimal reaction in the presence of g-C<sub>3</sub>N<sub>4</sub> (10 mg) and i-Pr<sub>2</sub>NEt (5.0 equiv) in isopropanol (5 mL) under air atmosphere by visible light irradiation at room temperature.

To explore the tolerance of the present method toward more reactive functional groups, a series oxidative hydroxylation of arylboronic acids with the optimal reaction conditions (Table 1, entry 3) were carried out, and the results were summarized in Table 2. With g-C<sub>3</sub>N<sub>4</sub> as photocatalyst, a wide range of arylboronic acids, bearing either electron-donating group such as methoxy, biphenyl and naphthalenyl or electron withdrawing groups such as cyano, fluoride, bromide, and nitro proceeded smoothly to the corresponding aryl alcohols in satisfactory yields. The conversion efficiency was affected by electronic effects of the substrates. For example, 4-fluorophenylboronic acid afforded the product 2c in a 100% yield after 12 h, while 4-methylphenylboronic acid gave the corresponding product 2g in a 35% yield after 12 h. It was clear that arylboronic acids bearing electron-withdrawing groups (Table 2, 2b-2f) showed higher reactivity than those bearing electrondonating groups (2g). What's more, this photocatalytic method

Table 1
Optimization of reaction conditions. <sup>a</sup>



Entry	Amine	Catalyst	Solvent	t (h)	Yield (%) <sup>b</sup>
1	iPr <sub>2</sub> NEt	g-C <sub>3</sub> N <sub>4</sub>	2-PrOH	4	46
2	iPr <sub>2</sub> NEt	g-C <sub>3</sub> N <sub>4</sub>	2-PrOH	8	65
3	iPr <sub>2</sub> NEt	g-C <sub>3</sub> N <sub>4</sub>	2-PrOH	12	97
4	iPr <sub>2</sub> NEt	g-C <sub>3</sub> N <sub>4</sub>	2-PrOH	16	99
5	iPr <sub>2</sub> NEt	g-C <sub>3</sub> N <sub>4</sub>	MeOH	12	76
6	iPr <sub>2</sub> NEt	g-C <sub>3</sub> N <sub>4</sub>	MeCN	12	77
7	iPr <sub>2</sub> NEt	g-C <sub>3</sub> N <sub>4</sub>	DMSO	12	78
8	iPr <sub>2</sub> NEt	g-C <sub>3</sub> N <sub>4</sub>	DMF	12	78
9 <sup>c</sup>	iPr <sub>2</sub> NEt	/	2-PrOH	12	NR
10	Et <sub>3</sub> N	g-C <sub>3</sub> N <sub>4</sub>	2-PrOH	12	82
11 <sup>d</sup>	/	g-C <sub>3</sub> N <sub>4</sub>	2-PrOH	12	NR
12 <sup>e</sup>	iPr <sub>2</sub> NEt	g-C <sub>3</sub> N <sub>4</sub>	2-PrOH	12	92
13 <sup>f</sup>	iPr <sub>2</sub> NEt	g-C <sub>3</sub> N <sub>4</sub>	2-PrOH	12	16
14 <sup>g</sup>	iPr <sub>2</sub> NEt	g-C <sub>3</sub> N <sub>4</sub>	2-PrOH	12	21
15	iPr <sub>2</sub> NEt	TiO <sub>2</sub>	2-PrOH	12	29
16	iPr <sub>2</sub> NEt	WO <sub>3</sub>	2-PrOH	12	NR

<sup>a</sup> Reaction conditions: 1a (0.1 mmol), amine (5.0 equiv), catalyst (10 mg), solvent (5 mL), air, irradiation was performed with visible light (filtered light at λ > 400 nm), r.t. <sup>b</sup> Yields were determined by GC–MS, internal standard method.

<sup>c</sup> No photocatalyst added.

<sup>d</sup> No amine added.

<sup>e</sup> Under O<sub>2</sub> atmosphere.

f Under N<sub>2</sub> atmosphere.

<sup>g</sup> With 4-benzoquinone. r.t. = room temperature; NR = no reaction.

can be used to product the electron-rich phenols, which are difficult to obtain from the traditional nucleophilic substitution of arylhalides [20].

Fig. 1A displayed the device diagram when the  $g-C_3N_4$  powder was reacted as the catalyst. A 300 W Xenon lamp with a UV filter and the light intensity of 500 mW/cm<sup>2</sup> was used as the visible light

source. The optimization of reaction conditions for the hydroxylation of 4-methoxyphenylboronic acid and the scope for various phenols derived from boronic acids are both carried out in this device. In order to imitate the reaction under sunlight, we use SUN-Q-Light photoreactor with the light intensity of 125 mW/ cm<sup>2</sup> as a simulated sunlight when g-C<sub>3</sub>N<sub>4</sub>/LMPET was used as the



**Fig. 1.** (A) The device diagram of  $g_{-C_3N_4}$  powder reaction system (The illustration shows the  $g_{-C_3N_4}$  precipitation diagram without stirring.). (B) The device diagram of  $g_{-C_3N_4/LMPET}$  reaction system. (C) Photograph of  $g_{-C_3N_4}$  powder and  $g_{-C_3N_4/LMPET}$  (about 25.00 × 17.00 cm) with about equivalent mass of  $g_{-C_3N_4}$ . (D) Photograph of the  $g_{-C_3N_4/LMPET}$  reaction system in real outdoor sunlight. (E) Time courses of oxidative hydroxylation of arylboronic acid using  $g_{-C_3N_4/LMPET}$  under solar light irradiation. Yields were determined by UPLC, external standard method. (F) Recycling tests for oxidative hydroxylation of arylboronic acid using  $g_{-C_3N_4/LMPET}$  under solar light irradiation for 16 h. Reaction conditions: 1a (0.1 mmol),  $ip_{2}NEt$  (5.0 equiv), photocatalyst (10 mg),  $g_{-C_3N_4/LMPET}$  (250 mg), air, irradiation was performed with solar light, r.t. Yields were determined by UPLC, external standard method.

photocatalyst (Fig. 1B). Obviously, powder catalysts here are exhibited as bulk catalysis, and the light receiving area during the reaction is limited. Additionally, the powder catalyst can be evenly dispersed only under high-speed agitation, otherwise it will sink



Scheme 2. Schematic diagrams of possible reaction mechanism over g- $C_3N_4$  under solar light irradiation.

Table 2 Scope and limitations.

to the bottom of the container (the illustration in Fig. 1A). g- $C_3N_4/LMPET$  with equivalent mass of g- $C_3N_4$  has a larger area than g- $C_3N_4$  powder (Fig. 1C). As can be seen in Fig. 1D, g- $C_3N_4/LMPET$  can also be amplified in real outdoor sunlight. The oxidative hydroxylation of arylboronic acids with the optimal reaction conditions was performed when g- $C_3N_4/LMPET$  was used as the catalyst, 4-methoxyphenylboronic acid gradually reduced and 4-methoxyphenol yield gradually increased during the reaction process (Fig. 1E). The yield reached 82% after 16 h, and we chose 16 h as the optimal reaction time. The reaction efficiency is also characterized by residual rate:

Residual rate(%) =  $C/C_0 \times 100 = A/A_0 \times 100$ 

where  $C_0$ , C,  $A_0$  and A represent the initial concentration of substrate, the concentration of substrate for the corresponding reaction time, the initial substrate area detected by HPLC, and variable substrate area detected by HPLC, respectively.

Although the light intensity of the two reaction systems differs by four times, their conversion efficiencies are very close. We can get a conclusion that the efficiency of photocatalytic reaction is



Reaction conditions: substrate (0.1 mmol), iPr<sub>2</sub>NEt (5.0 equiv), photocatalyst g-C<sub>3</sub>N<sub>4</sub> (10 mg), 2-PrOH (5.0 mL), air, irradiation was performed with visible light (filtered light at  $\lambda$  > 400 nm), r.t. Reaction time was 12h.

proportional to the light receiving area. The reusability of  $g-C_3N_4/$ LMPET was also investigated (Fig. 1F). Following each reaction, the catalyst was separated, washed with water and ethanol. Afterwards, g-C<sub>3</sub>N<sub>4</sub>/LMPET was dried at 60 °C and then reused for the ensuing reaction. The results indicate that the activity of the catalyst has no obvious decrease even after 8 cycles. In addition, it can be seen in Fig. S8B that the catalytic system is also dominated by superoxide radicals.

In consideration of the aforementioned results and the previous reports [12,13a], a plausible mechanism of the oxidative hydroxylation of arylboronic acids is depicted in Scheme 2. When g-C<sub>3</sub>N<sub>4</sub> was subjected to the visible light irradiation, it was excited to generate electron-hole pairs. The photogenerated electrons were excited from the VB to the CB, leaving the holes in the VB of g-C<sub>3</sub>N<sub>4</sub>. The photoexcited electron can be quenched by oxygen, forming the superoxide radical anion  $O_2^-$ .  $O_2^-$  then reacts with boronic acid to generate the intermediate a. Meanwhile, the oxidation of iPr<sub>2</sub>NEt by the photogenerated hole in the excited state of g-C<sub>3</sub>N<sub>4</sub> gives radical cation iPr<sub>2</sub>NEt<sup>+</sup>. The radical anion intermediate **a** then abstracts a hydrogen atom from iPr2NEt+ radial cation to form intermediate **b**. The formation of phenol product takes place by rearrangement of intermediate **b** into intermediate **c** with subsequent hydrolysis.

In conclusion, we have firstly developed that  $g-C_3N_4$  as the metal-free photocatalyst can be utilized for selective hydroxylation of arylboronic acids to phenols under the visible light irradiation. In this reaction, the high yield of phenols were achieved under metal-free, ambient-pressure, and room-temperature conditions with using air as the terminal oxidant source and isopropanol as the solvent. It exhibits good substrate tolerance and provides a fast and efficient access to a variety of functionalized phenols. It's worth mentioning that g-C<sub>3</sub>N<sub>4</sub>/LMPET as the metal-free artificial photosynthetic catalytic fabric also demonstrated efficient conversion efficiency under solar irradiation with the same conditions after 16 h. This discovery highlights a significant role that a large receiving light area can improve the utilization of sunlight, thus opening up the possibility of utilizing fiber material in photocatalvtic organic transformations. The new system provides a reference for the effective use of clean solar energy and such environmentally method in the selective photosynthesis of other valuable chemicals.

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#### **Appendix A. Supplementary material**

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jcat.2019.07.062.

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