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Recyclable g-C_3N_4 catalyzed decarboxylative alkenylation of *N*-aryl glycines with vinyl sulfones under visible-light irradiation†

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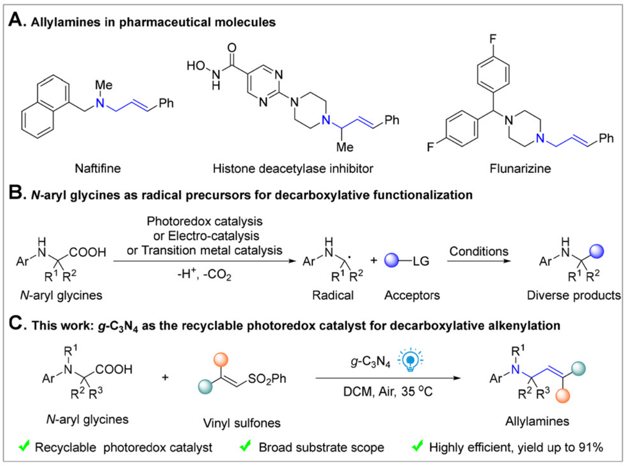
Allylamines are a versatile class of compounds with significant applications in pharmaceuticals and as building blocks in organic synthesis. Herein we present a straightforward protocol for visible-light driven g-C_3N_4-catalyzed decarboxylative alkenylation of *N*-aryl glycines with vinyl sulfones to access allylamines in moderate to excellent yields (up to 91%), and it demonstrated broad substrate compatibility, including primary, secondary, and tertiary *N*-aryl glycines and diverse vinyl sulfones. Notably, the g-C_3N_4 catalyst is recyclable up to five times without obvious loss of catalytic performance. Preliminary mechanistic studies indicated that visible light is essential to achieve the desired transformation efficiently.

Introduction

Allylamines are a class of compounds with diverse pharmacological effects, abundant in the pharmaceutical field as antifungals, antihistamines, antidepressants and more (Scheme 1A).[1] Over the past few decades, numerous methods for synthesizing allylamine derivatives have been developed.[2] However, most of these strategies rely on the use of expensive or toxic transition metal catalysts, high temperatures, and harsh reaction conditions, resulting in limited substrate compatibility. Recently, visible-light promoted photoredox catalysis has been widely used in organic synthesis, leveraging visible light as an energy source to drive chemical reactions in a clean, efficient, and sustainable manner.[3] Homogeneous photoredox catalysts, such as transition metal complexes and organic dyes, have been widely utilized in organic synthesis.[4] However, resource scarcity, high price and metal contamination in products limited their potential practical application. To address these challenges, the development of practical heterogeneous catalysts has become a promising alternative. g-C_3N_4, also known as graphitic carbon nitride, is a semiconductor material composed of carbon and nitrogen elements and exhibits excellent photocatalytic and photothermal properties, making it applicable in various fields.[5] Moreover, owing to its wide availability and remarkable stability, g-C_3N_4 has also been employed as a photoredox catalyst in organic synthesis.[6]

N-Aryl glycines, a class of natural amino acid derivatives, are widely utilized as pharmaceutical intermediates and a key raw material for generating α-aminoalkyl radicals *via* de-



Scheme 1 (A) Allylamines in pharmaceutical molecules. (B) *N*-Aryl glycines as radical precursors for decarboxylative functionalization. (C) This work: g-C_3N_4 as the recyclable photoredox catalyst for decarboxylative alkenylation.

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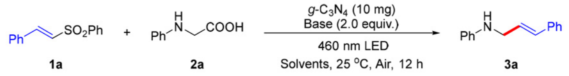
carboxylation, enabling the efficient synthesis of high value-added products (Scheme 1B).[7–17] Recently, silver-catalyzed decarboxylation of carboxylic acids has also emerged as a promising strategy for the synthesis of diverse functional molecules.[18] Since Rueping *et al.* pioneered the first visible light-induced g-C_3N_4-catalyzed decarboxylative alkylation of *N*-aryl glycines in 2018, the recent advances in this field have opened novel avenues for organic transformations and the synthesis of complex molecules.[19,20] Nevertheless, to the best of our knowledge, visible-light-induced g-C_3N_4-catalyzed decarboxylative alkenylation of *N*-aryl glycines has not yet been reported. In this study, we have reported g-C_3N_4 as a recyclable photoredox catalyst which induced the generation of α-aminoalkyl radicals from *N*-aryl glycines to undergo radical addition with vinyl sulfones for the efficient synthesis of allylamines under visible-light irradiation (Scheme 1C).

Results and discussion

Initially, we employed phenyl vinyl sulfones (**1a**) and *N*-phenyl glycine (**2a**) as substrates to optimize the reaction conditions (Table 1). When 10 mg g-C_3N_4 was used as a photoredox catalyst, $NaHCO_3$ (2.0 equiv.) as the base in DCM (2 mL), under 15 W 460 nm LED irradiation in air, the desired alkylation product **3a** was detected in a trace amount (entry 1). Then, various bases were screened and it was found that CsF was the most effective, enhancing the yield to 53% (entries 2–8). Further screening of solvents indicated that DCM was the optimal medium (entries 9–15). When the amount of g-C_3N_4 was adjusted to 15 mg, the efficiency of the reaction was not improved obviously (entry 16). However, **3a** was obtained in 60% yield with the use of 2 equivalents of **2a** (entry 17). Screening of the reaction concentrations demonstrated that 3 mL of DCM resulted in 72% yield (entries 18–20). Finally, the product **3a** was obtained in 85% yield by increasing the reaction temperature to 35 °C (entry 21). Controlled experiments confirmed that visible light, g-C_3N_4 and base were all essential for achieving the desired transformation (entries 22–24).

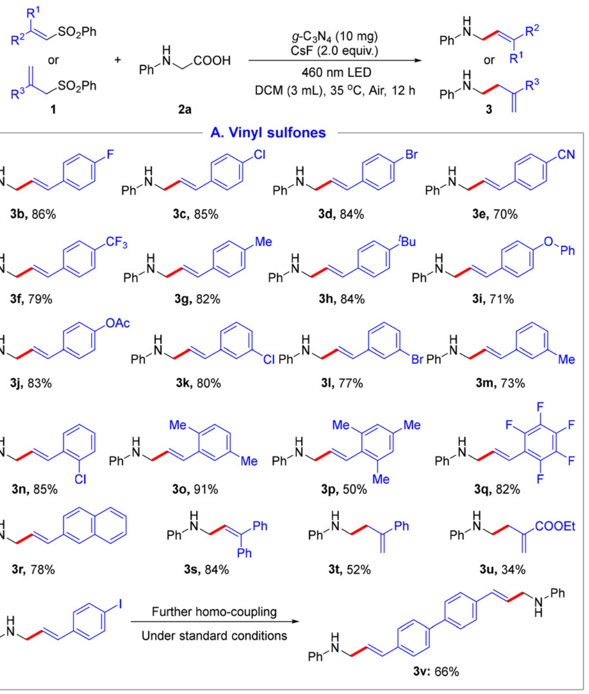
Table 1 Optimization of the reaction conditions[a]



Entry	**2a** (equiv.)	Base	Solvent (mL)	*T* (°C)	Yield[b] (%)
1	1.5	$NaHCO_3$	DCM (2)	25	Trace
2	1.5	CH_3COOK	DCM (2)	25	Trace
3	1.5	AgF	DCM (2)	25	0
4	1.5	NH_4F	DCM (2)	25	34
5	1.5	InF_3	DCM (2)	25	Trace
6	1.5	KF	DCM (2)	25	36
7	1.5	NaF	DCM (2)	25	Trace
8	1.5	CsF	DCM (2)	25	53
9	1.5	CsF	DCE (2)	25	41
10	1.5	CsF	MeOH (2)	25	47
11	1.5	CsF	Toluene (2)	25	50
12	1.5	CsF	EA (2)	25	44
13	1.5	CsF	1,4-Dioxane (2)	25	43
14	1.5	CsF	MeCN (2)	25	47
15	1.5	CsF	THF (2)	25	48
16[c]	1.5	CsF	DCM (2)	25	37
17	2.0	CsF	DCM (2)	25	60
18	2.0	CsF	DCM (1)	25	51
19	2.0	CsF	DCM (3)	25	72
20	2.0	CsF	DCM (4)	25	57
21	**2.0**	**CsF**	**DCM (3)**	**35**	**85**
22[d]	2.0	CsF	DCM (3)	35	Trace
23[e]	2.0	CsF	DCM (3)	35	0
24	2.0	—	DCM (3)	35	28

[a] Reaction conditions: **1a** (0.2 mmol, 1.0 equiv.), **2a**, g-C_3N_4 (10 mg), base (2.0 equiv.), solvents, under 15 W 460 nm LED irradiation in air for 12 h. [b] Isolated yield. [c] g-C_3N_4 (15 mg). [d] In the absence of g-C_3N_4. [e] In the dark.

Under the optimal conditions, we next explored the substrate scope of vinyl sulfones as shown in Scheme 2. Generally, aryl vinyl sulfones bearing diverse electron-donating and electron-withdrawing groups, such as –F, –Cl, –Br, –CN, –CF_3, –Me, –^{t}Bu, –OPh, and –OAc on benzene rings at diverse positions, underwent the reaction smoothly, yielding the corresponding products in 70–86% yields (**3b–3n**). Multi-substituted aromatic vinyl sulfones were also compatible, delivering the products **3o–3q** in moderate to excellent yields. Moreover, naphthyl vinyl sulfone was also tolerated to afford **3r** in 78% yield. When 1,1-diphenyl vinyl sulfone was utilized as the substrate, trisubstituted alkene **3s** was obtained in 84% yield. Furthermore, ester-substituted vinyl sulfone was also employed to achieve the target product **3u** in 34% yield. However, the unexpected dimer product **3v** was formed when 4-iodophenyl



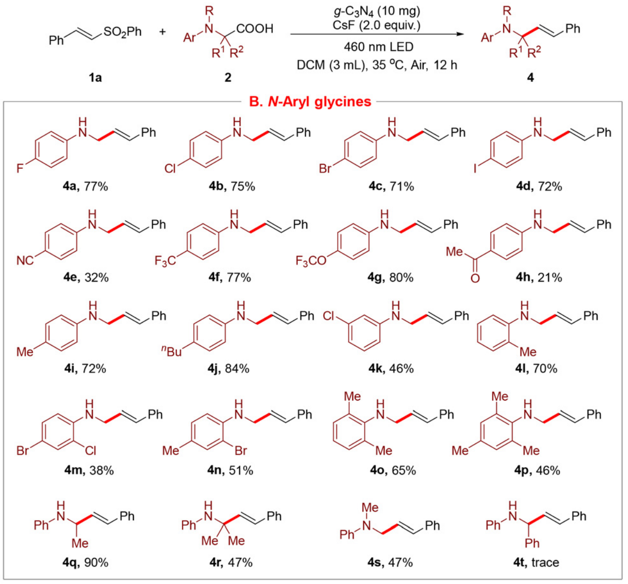
Scheme 2 Substrate scope of vinyl sulfones. Reaction conditions: **1** (0.2 mmol, 1.0 equiv.), **2a** (2.0 equiv.), g-C_3N_4 (10 mg), CsF (2.0 equiv.), DCM (3 mL) at 35 °C under 15 W 460 nm LED irradiation in air for 12 h.

vinyl sulfone was used as the starting material under standard conditions.

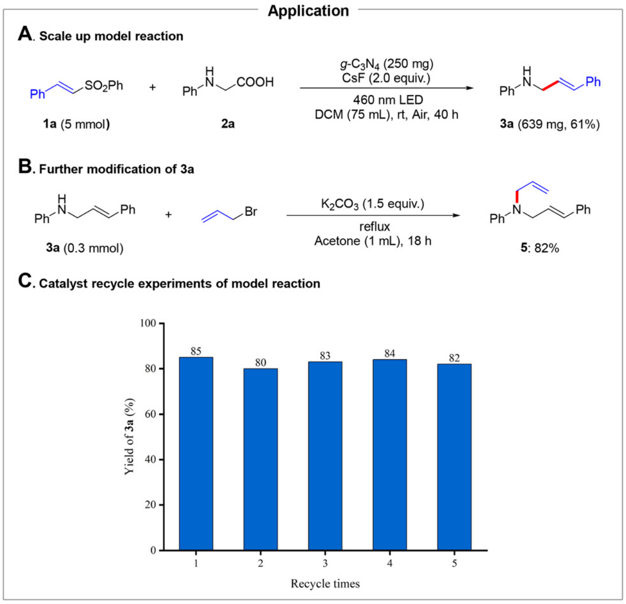
Subsequently, a range of diverse *N*-aryl glycines were explored as shown in Scheme 3. Notably, we varied the steric hindrance or electronic properties of the aryl ring to evaluate the efficiencies, producing the desired products in 21–84% yields (**4a–4l**). Substituents such as –F (**4a**), –Cl (**4b**, **4k**), –Br (**4c**), –I (**4d**), –CN (**4e**) and –COMe (**4h**) at diverse positions of the benzene ring were compatible, allowing the synthesis. *N*-Aryl glycines with di- or tri-substituents were also accommodated under the optimal conditions to generate **4m–4p** in 38–65% yields. In addition, α-methyl or dimethyl substituted *N*-aryl glycines were effective acceptors, affording the corresponding products in 90% and 47% yields, respectively (**4q**, **4r**). Moreover, *N*-methyl-*N*-phenyl glycine was also suitable for the transformation, providing the product **4s** in 47% yield. Unfortunately, when *N*-phenyl-α-phenyl glycine was employed as the substrate, only a trace amount of the product was detected (**4t**).

To showcase the practical application, we scaled up the model reaction to 5 mmol, and the product **3a** was isolated in 61% yield (Scheme 4A). Furthermore, **3a** was efficiently transformed into the diene product **5** in 82% yield, underscoring its potential for practical synthetic applications (Scheme 4B). To evaluate the stability and recyclability of g-C_3N_4, a series of recycling experiments were conducted to assess its performance in multiple reaction cycles. As shown in Scheme 4C, g-C_3N_4 was successfully recycled and reused in five consecutive reaction cycles without any obvious loss in catalytic performance, highlighting the potential for a sustainable process.

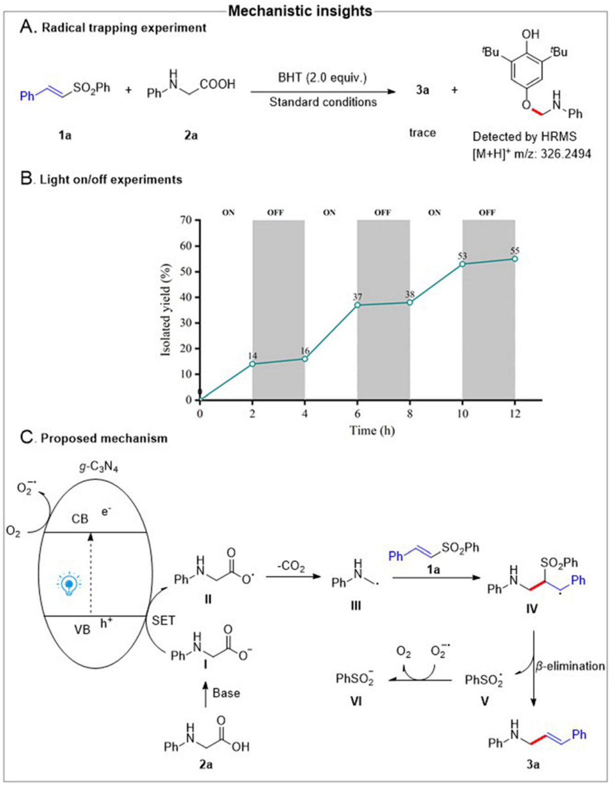
Next, we performed radical trapping and light on/off experiments to elucidate the possible mechanism. As shown in Scheme 5, the addition of a radical scavenger, such as 2,6-di-*tert*-butyl-4-methylphenol (BHT), to the standard reaction conditions completely inhibited the formation of product **3a.** Moreover, the aminomethyl-BHT adduct was detected by



Scheme 3 Substrate scope of *N*-aryl glycines. Reaction conditions: **1a** (0.2 mmol, 1.0 equiv.), **2** (2.0 equiv.), g-C_3N_4 (10 mg), CsF (2.0 equiv.), DCM (3 mL) at 35 °C under 15 W 460 nm LED irradiation in air for 12 h.



Scheme 4 (A) Scale up model reaction. (B) Further modification of **3a**. (C) Catalyst recycle experiments of the model reaction.



Scheme 5 (A) Radical trapping experiment. (B) Light on/off experiments. (C) Proposed mechanism.

high-resolution mass spectrometry, indicating the probable involvement of a radical species (Scheme 5A). In addition, the results of light on/off experiments suggested that a radical chain pathway was excluded, and product formation was sustained under light irradiation but completely ceased in the absence of light (Scheme 5B).

Based on the experimental results and precedent literature,[18–21] the proposed reaction mechanism is illustrated in Scheme 5C. Initially, under visible-light irradiation, g-C_3N_4 absorbed photons to generate holes in the valence band (VB) and electrons in the conduction band (CB). Then, oxygen in air was reduced by electrons in the CB to produce the superoxide radical anion. Simultaneously, the deprotonation of **2a** proceeded in the presence of base to generate carboxylate anion **I**, which was oxidized by holes in the VB to form carboxyl radical **II**. Following the release of CO_2 from **II**, α-aminoalkyl radical **III** was generated, which underwent radical addition with **1a** to form the intermediate **IV**. The intermediate **IV** then underwent β-elimination, leading to the desired alkenylation product **3a** and phenylsulfone radical **V**. Finally, the phenylsulfone radical was reduced to the corresponding phenylsulfone anion **VI** by the superoxide radical anion.

Conclusions

In summary, we have developed an efficient protocol for the decarboxylative alkenylation of *N*-aryl glycines with vinyl sulfones using recyclable g-C_3N_4 as a heterogeneous photoredox catalyst. This strategy accommodated a wide variety of *N*-aryl glycines, demonstrating excellent functional group tolerance and facilitating the rapid synthesis of allylamines. Additionally, this method was scalable to the gram-scale and also provided direct access to valuable diene compounds through straightforward product derivatization, underscoring its practicality and potential application in organic synthesis.

Data availability

The data supporting this article have been included as part of the ESI.†

Conflicts of interest

The authors declare no conflict of interest.

Acknowledgements

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