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Photocatalyst-free visible light driven synthesis of (*E*)-vinyl sulfones from cinnamic acids and arylazo sulfones

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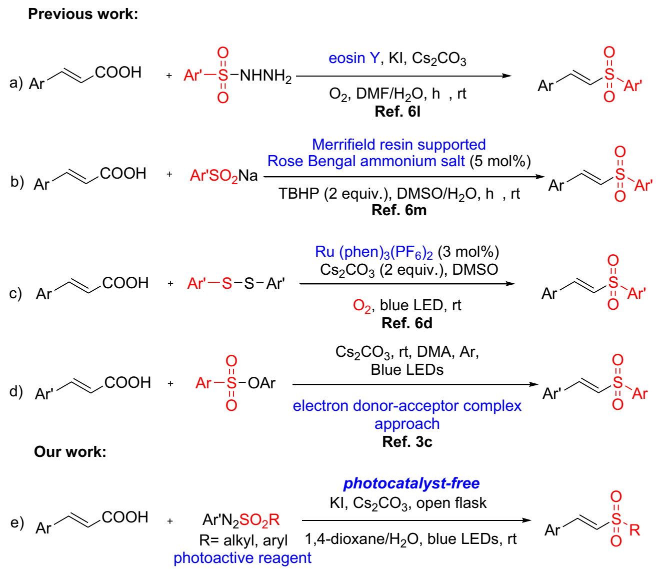
A photocatalyst-free visible light mediated decarboxylative sulfono functionalization protocol has been explored for the synthesis of (*E*)-vinyl sulfones from cinnamic acids and bench-stable arylazo sulfones. The latter have been utilized as sulfonyl radical precursors under blue LED irradiation along with Cs_2CO_3, air and KI to obtain the vinyl aryl sulfones as well as vinyl alkyl sulfones in moderate to excellent yields. Besides the non-photocatalytic conditions, high ecosustainability, low cost, ease of operation and access to vinyl alkyl sulfones are certain indispensable traits of the developed method.

Visible light driven decarboxylative cross-coupling reactions have transpired as an extremely resourceful tool for the construction of C—C and C-heteroatom bonds over the last few years [1]. Using these reactions, formation of new bonds takes place in a highly ecosustainable manner owing to the readily available starting materials and the innocuous and easily removable by-product (CO_2). Moreover, milder reaction conditions, higher efficiency, greater selectivity and exceptional functional group compatibility add to the credentials of this strategy. In general, these visible light mediated transformations are brought about by a photocatalyst (polypyridyl metal complexes or organic dyes) [2]. An attractive alternative to these expensive or degradable catalysts can be the use of a photoactive substrate or reagent which precludes the use of any external photosensitizer. The concept is highly appealing but the number of reports on such photocatalyst-free visible light mediated decarboxylative cross-coupling reactions is limited [1,3] which allows ample room for the development of novel protocols based on this approach.

Vinyl sulfones, as a class of organosulfur compounds, are interesting targets of synthesis due to their potential biological activity [4] and versatile synthetic utility [5]. Among the various methods available for their synthesis, the decarboxylative sulfonylation of cinnamic acids is a simple and direct method for their synthesis [6]. This synthetic approach has drawn significant attention over the last few years and the target molecules have been synthesized under both transition-metal catalysed [6a-d] and transition-metal-free conditions [6e-m]. However, most of these methods suffer from drawbacks, viz. use of expensive metal catalysts, elevated



Scheme 1. Visible light mediated synthesis of vinyl sulfones from cinnamic acids.

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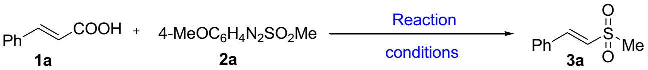
temperatures, difficult-to-remove solvents and limited substrate scope. Some of these obstacles have been overcome by employing visible light as a reagent for this reaction but the number of such reports is limited [3d,6d,l,m]. In 2016, Cai et al. reported the eosin Y catalyzed visible light induced decarboxylative cross-coupling reactions of cinnamic acids with sulfonylhydrazides at room temperature for vinyl sulfone synthesis (Scheme 1a) [6l]. Recently, Li and Wang addressed the degradation issue of the organic dye by using Merrifield resin supported Rose Bengal as a catalyst and sodium sulfinates as sulfonylating agent and 2 equiv. of TBHP as oxidant (Scheme 1b) [6m]. In another report, Wang and co-workers employed disulfides as sulfonylating agents in the presence of $Ru(phen)_3(PF_6)_2$ as a photocatalyst, Cs_2CO_3 as a base in DMSO at room temperature under blue LED (450–455 nm) irradiation and air atmosphere (Scheme 1c) [6d]. All these reports rely on the use of expensive or degradable catalysts, stoichiometric amounts of external oxidants and suffer from limited substrate-scope, i.e. only vinyl aryl sulfones can be synthesized by these methods and not vinyl alkyl sulfones. To the best of our knowledge, there is only one visible light mediated photocatalyst-free method reported so far for the synthesis of vinyl sulfones from cinnamic acids by Xuan et al. enabled by the electron donor-acceptor complex approach (Scheme 1d) [3c,d]. Even in this method, the synthesis of vinyl alkyl sulfones was not reported. All the above stated reasons and our work on visible light mediated reactions [7] and sulfones [7c,8] prompted us to develop a photocatalyst-free visible light driven protocol for the synthesis of vinyl sulfones from cinnamic acids and arylazo sulfones with a wider substrate-scope. Arylazo sulfones are the photoactive reagents which served as the sulfonyl radical precursors in the reaction. They are colored bench-stable compounds which can be easily prepared in two steps from anilines [9] and have recently drawn great interests of chemists towards photocatalyst-free visible light induced arylation

Table 1
Optimization of experimental conditions.[a]



Entry	PC	Base	Solvent v/v	Time (h)	Yield (%)[b]
1	–	Cs_2CO_3	DMF/H_2O 15:1	18	32
2	–	–	DMF/H_2O 15:1	18	0
3	–	Cs_2CO_3	DMF/H_2O 15:1	18	0[c]
4	–	Cs_2CO_3	DMF/H_2O 15:1	18	0[d]
5	Eosin Y	Cs_2CO_3	DMF/H_2O 15:1	18	23
6	Rose Bengal	Cs_2CO_3	DMF/H_2O 15:1	18	27
7	$Ru(bpy)_3Cl_2{\cdot}6H_2O$	Cs_2CO_3	DMF/H_2O 15:1	18	29
8	–	Cs_2CO_3	DMF	18	traces
9	–	Cs_2CO_3	H_2O	18	0
10	–	Cs_2CO_3	DMSO/H_2O 15:1	18	22
11	–	Cs_2CO_3	EtOH/H_2O 15:1	18	30
12	–	Cs_2CO_3	1,4-dioxane/H_2O 15:1	18	46
13	–	Cs_2CO_3	THF/H_2O 15:1	18	31
14	–	Cs_2CO_3	CH_3CN/H_2O 15:1	18	24
15	–	Cs_2CO_3	1,4-dioxane/H_2O 9:1	18	52
16	–	Cs_2CO_3	1,4-dioxane/H_2O 5:1	18	66
17	-	Cs_2CO_3	1,4-dioxane/H_2O 2:1	18	72
18	-	Cs_2CO_3	1,4-dioxane/H_2O 1:1	18	58
19	–	Na_2CO_3	1,4-dioxane/H_2O 2:1	18	trace
20	–	K_2CO_3	1,4-dioxane/H_2O 2:1	18	0
21	–	DBU	1,4-dioxane/H_2O 2:1	18	39
22	–	Cs_2CO_3	1,4-dioxane/H_2O 2:1	18	54[e]
23	**–**	$\mathbf{Cs_2CO_3}$	**1,4-dioxane/$\mathbf{H_2O}$ 2:1**	**18**	**77**[f]
24	–	Cs_2CO_3	1,4-dioxane/H_2O 2:1	18	78[g]
25	–	Cs_2CO_3	1,4-dioxane/H_2O 2:1	18	63[h]
26	–	Cs_2CO_3	1,4-dioxane/H_2O 2:1	24	77[f]
27	–	Cs_2CO_3	1,4-dioxane/H_2O 2:1	14	60[f]
28	–	Cs_2CO_3	1,4-dioxane/H_2O 2:1	18	32[f,i]
29	–	Cs_2CO_3	1,4-dioxane/H_2O 2:1	18	traces[f,j]
30	–	Cs_2CO_3	1,4-dioxane/H_2O 2:1	18	66[f,k]
31	–	Cs_2CO_3	1,4-dioxane/H_2O 2:1	18	52[f,l]

[a] Reaction conditions: **1a** (0.5 mmol), **2a** (2.2 equiv.), KI (1.0 equiv.), PC (0.01 equiv.), base (3.0 equiv.), solvent (1 mL), 3 W blue LEDs and open flask at rt.
[b] Isolated yield.
[c] No visible light irradiation.
[d] No KI.
[e] **2a** (1.5 equiv.).
[f] **2a** (2.5 equiv.).
[g] **2a** (3.0 equiv.) and Cs_2CO_3 (3.5 equiv.).
[h] Cs_2CO_3 (2.5 equiv.)
[i] 3W white LED lamps.
[j] 3W green LED lamps.
[k] Solvent 3 mL.
[l] Solvent 0.5 mL.

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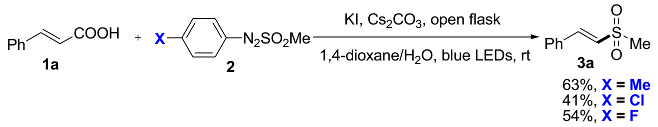
reactions [10]. Very recently, Wei et al. have employed these compounds as sulfonylating agents as well [11]. Consequently, herein we describe the synthesis of (*E*)-vinyl sulfones from cinnamic acids and arylazo sulfones mediated by visible light under photocatalyst- and external oxidant-free conditions (Scheme 1e).

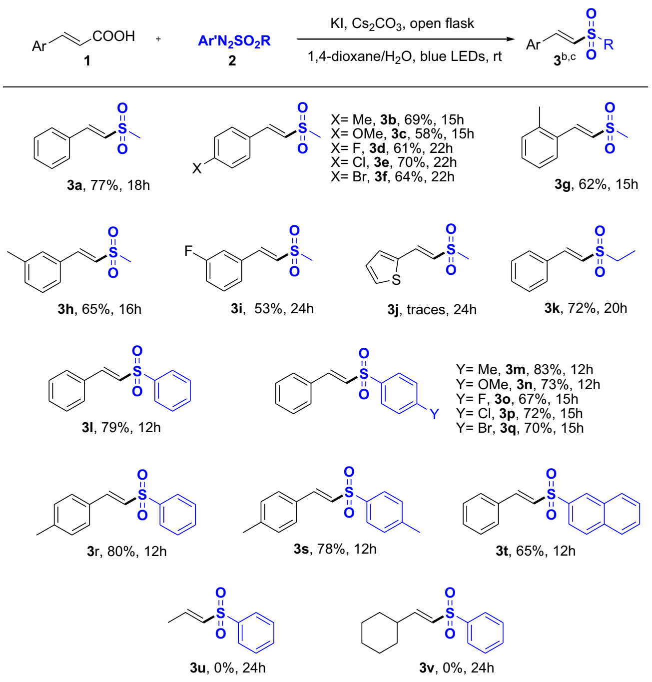
To probe the feasibility of our strategy, a model reaction was performed using 4-methoxyphenylazo mesylate (**2a**) as the sulfonylating agent for the decarboxylative sulfonylation of cinnamic acid **1a** under 3 W blue LED lamps irradiation and photocatalyst-free conditions (Table 1). Most satisfactorily, under these conditions the desired product **3a** was obtained in 32% yield (Table 1, entry 1). In the absence of Cs_2CO_3, KI or light, no product could be obtained similar to the observations of the previous report [6l] (Table 1, entries 2–4). The use of a photocatalyst was found to have no significant impact on the efficiency of the reaction (Table 1, entries 5–7). In fact, the lower yields of the product can be attributed to the partial absorption of light by the photocatalysts leading to less dissociation of **2a** into sulfonyl radical.

Subsequently, a variety of solvents and solvent-systems were screened to decide the best media for our reaction (Table 1, entries 8–14) and the best yield of the product was obtained in 1,4-dioxane/H_2O (15:1) (entry 12). The ratio of solvents was also optimized (entries 15–18) and it was found that the ratio 2:1 of 1,4-dioxane/H_2O worked best for our reaction (entry 17). This observation can be attributed to the fact that water as a solvent may promote the homophotolysis of **2a** but its addition in excess may lead to solubility issues. Replacing the base Cs_2CO_3 with Na_2CO_3, K_2CO_3 or 1,8-diazabicyclo[5.4.0]- undec-7-ene (DBU)) did not show any better results and Cs_2CO_3 remained the choice of base for the reaction (entry 17 vs entries 19–21). The use of 1.5 equiv. of **2a** led to a substantial decrease in the yield of **3a** (entry 22). An increase in the loading of **2a** from 2.2 equiv. to 2.5 equiv. was conducive to the efficiency of the reaction (entry 23) but further increase in the amount of **2a** and Cs_2CO_3 produced no significant increment in the yield of **3a** (entry 24). However, the yield of **3a** decreased with the decrease in the amount of Cs_2CO_3 (entry 25). Increasing the reaction time to 24 h led to no further increase in the yield of the product but lowering the reaction time was detrimental to the efficieny of the reaction (entries 26 and 27). We next examined the visible light source. In the presence of 3 W white LED and green LED lamps, the product **3a** was isolated in 32% yield and traces respectively (entries 28 and 29). On dilution as well as on decreasing the amount of solvent, the yield of product decreased (entries 30 and 31). As regards the use of arylazo sulfones, derivatives other than **2a** such as 4-methylphenylazo mesylate, 4-chlorophenylazo mesylate or 4-fluorophenylazo mesylate were also tested but the yield of the desired product **3a** was observed to be lower than the model reaction in all these cases (Scheme 2).



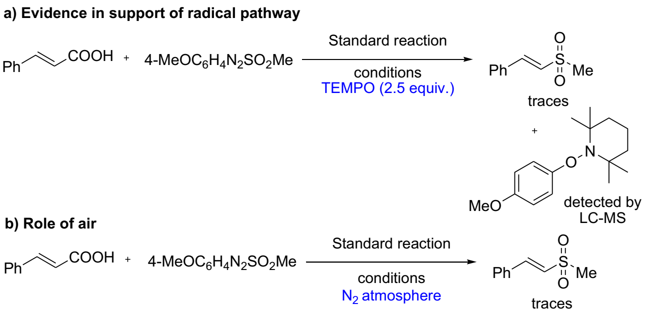
Scheme 2. Reaction of cinnamic acid **1a** with other arylazo sulfones **2**.

Table 2
Substrate scope.[a]



[a] Reaction conditions: **1a** (0.5 mmol), **2a** (2.5 equiv.), KI (1.0 equiv.), Cs_2CO_3 (3.0 equiv.), 1,4-dioxane/H_2O 2:1 (1 mL), 3 W blue LEDs and open flask at rt, 12–24 h.
[b] Isolated yield.
[c] All compounds are known in literature [3c,6d,12] and gave satisfactory spectral (^1H NMR, ^{13}C NMR and HRMS) data.

Having identified the optimal reaction conditions, we next proceeded to examine the substrate scope of the reaction with respect to cinnamic acids using **2a**. A good variety of substituents on the aromatic ring of cinnamic acids were amenable with our protocol (Table 2, 3b-**i**). Electron-donating (Me, OMe) as well as electron-withdrawing groups (halogens) on the aromatic ring of cinnamic acids afforded the desired products in moderate yields. Cinnamic



Scheme 3. Control experiments.

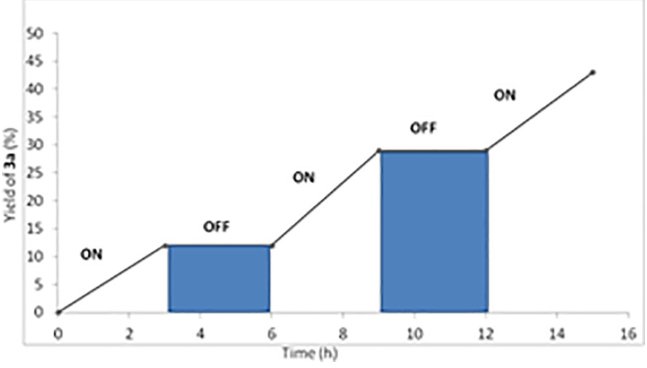


Fig. 1. Result of irradiation ON/OFF experiment.

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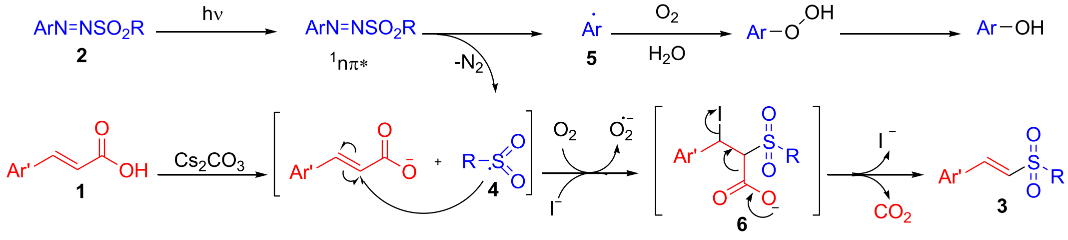

Scheme 4. Plausible mechanism for the formation of vinyl sulfones.

acids with a methyl substituent at the *para*, *ortho* and *meta* position of the aromatic ring led to the formation of the corresponding vinyl sulfones **3b**, **3g** and **3h** in 69%, 62% and 65% yield, respectively. 3-(Thiophen-2-yl)acrylic acid, a heteroaromatic derivative of cinnamic acid, was also tested but it did not lead to any encouraging result (**3j**) even after 24 h.

Further, the scope of this decarboxylative sulfonylation was extended to a series of arylazo sulfones. Not only arylazo methylsulfone, arylazo ethyl sulfone was also found to be a suitable reagent for this transformation (**3k**). Arylazo aryl sulfones containing both electron-donating and electron-withdrawing groups on the aryl rings attached to the sulfonyl group were well-tolerated under our reaction conditions and the corresponding vinyl sulfones (**3m**–**s**) were generated in moderate to excellent yields. The developed protocol was amenable with the naphthyl derivative of azo sulfone as well (**3t**) but the method failed with propenoic acid (**3u**) and 3-cyclohexylacrylic acid (**3v**).

In order to gain insight into the reaction mechanism, a couple of control experiments were performed (Scheme 3). When TEMPO (a free radical scavenger) was added to the reaction under standard conditions, product formation was inhibited and the aryl-TEMPO adduct was also detected, indicating a free radical pathway (Scheme 3a). No product was formed in the absence of air, indicating that air is playing a role in the formation of product which is in accordance with earlier observations (Scheme 3b) [6l]. The result of On/Off irradiation experiments established that continuous visible light irradiation is necessary for the decarboxylative sulfonylation reaction (Fig. 1).

On the basis of above results and previous literature, [6l,11a] a possible mechanism for the decarboxylative sulfonylation has been depicted in Scheme 4. Firstly, photoactivation of arylazo sulfone **2** generated its singlet $^1n\pi^*$ excited state. Further, the homolysis of N—S bond of this species led to the formation of sulfonyl radical **4** and aryl radical **5** along with the escape of N_2. The aryl radical **5** is then converted into the side product phenol (detected by LC-MS analysis) in the presence of air and water. Simultaneously, selective addition of sulfonyl radical to cinnamic acid followed by oxidation, and iodide ion capture led to the formation of intermediate **6**. Ultimately, intermediate **6** was converted into the final product **3** with the elimination of carbon dioxide and iodide ion. Detection of H_2O_2 by KI/starch indicator confirmed the formation of the superoxide radical anion in the reaction [13].

A simple and green protocol with wider substrate scope has been developed for the synthesis of (*E*)-vinyl sulfones from cinnamic acids and arylazo sulfones. The reaction follows a radical pathway and it is mediated by visible light in the absence of a photocatalyst and external oxidant. The decarboxylation process is facilitated by the use of a base Cs_2CO_3, air and KI. Even the vinyl alkyl sulfones could be obtained in good yields via our strategy. This is an exceptional advantage of our method over the existing visible light mediated decarboxylative sulfonylation methods for the synthesis of vinyl sulfones, which afford only vinyl aryl sulfones.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.tetlet.2020.151898.

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