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Visible-Light-Mediated Decarboxylative Thiocyanation of Cinnamic acids: An Efficient Photocatalytic Approach to the Synthesis of (*E*)-Vinyl Thiocyanates

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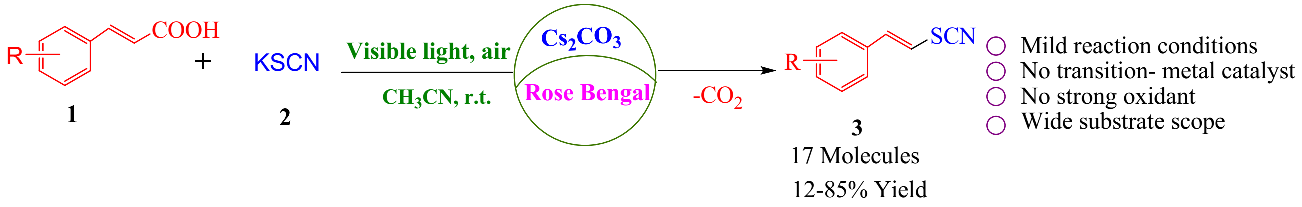
Abstract

A simple and novel methodology for the synthesis of vinyl thiocyanates from decarboxylative cross-coupling reaction of cinnamic acids with KSCN under the synergistic interactions of visible light irradiation, Cs_2CO_3, Rose Bengal as the photocatalyst and air as the terminal oxidant at room temperature is reported. The reaction takes place by a radical pathway as evidenced from our experiments and literature. The report is the first example on the visible-light mediated thiocyanation of cinnamic acids, which employs environmentally benign and inexpensive starting materials and is characterized by easily removable by-product CO_2.

Graphic Abstract



Keywords Visible light · Photoredox catalysis · C-S coupling · Radicals · Environmentally benign synthesis

1 Introduction

Recently, visible-light-mediated decarboxylative cross-coupling reactions have emerged as an attractive strategy to construct C–C, C–P, C–S, C–O and C–N bonds [1–6]. The credit of such widespread recognition of the strategy is linked with the potential advantages associated with it, e.g. environmental sustainability, cost-effectiveness, higher efficiency, greater selectivity and easily removable by-product [7–10]. Despite the great advances in the visible-light-mediated decarboxylative reactions, the formation of C–S bond via this strategy remains an underdeveloped process and deserves greater attention from the synthetic community [11, 12].

In synthetic organic chemistry, thiocyanation is a convenient method to form C-S bond [13–15]. In general, $Pb(SCN)_2$, KSCN, NaSCN, AgSCN and NH_4SCN salts are used as a SCN source to introduce the sulfur functionality to a substrate [16–21]. Thiocyanates, the product of this reaction, are valuable and versatile intermediates for the synthesis of sulfur containing heterocycles [22–25]. They also exhibit significant drug properties and biological activities, such as antifungal, antimicrobial and antiparasitic [26].

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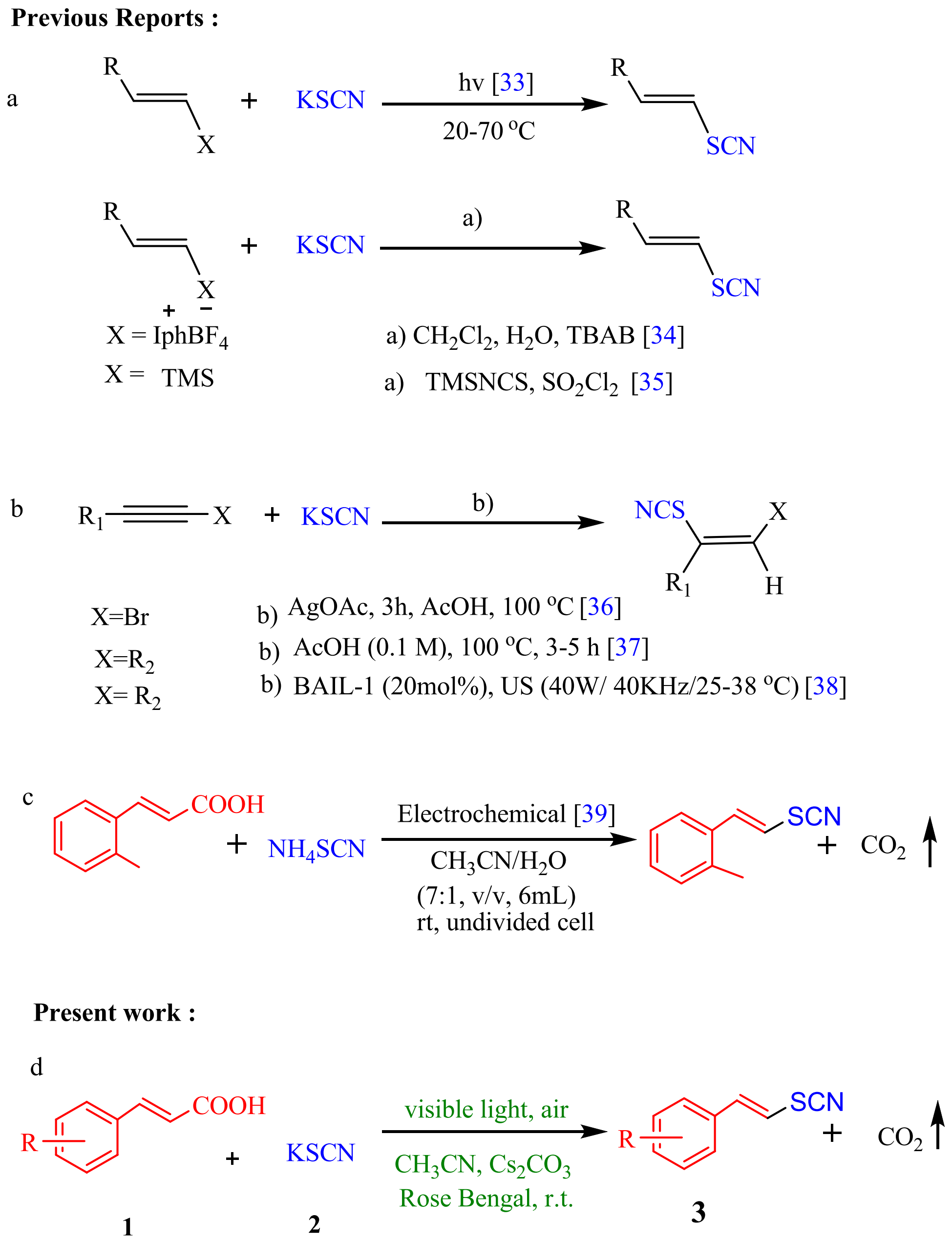
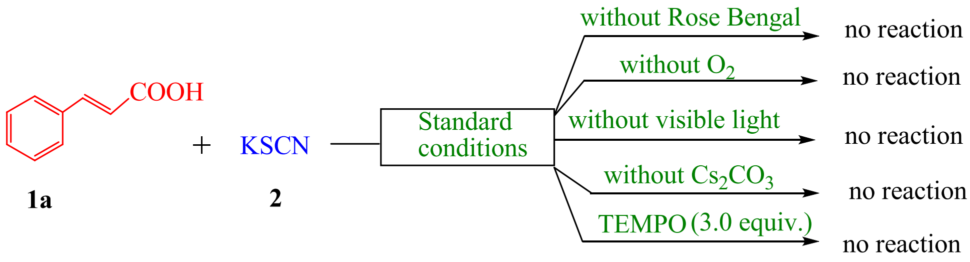
In this context, significant efforts have been undertaken to develop efficient methods for the formation of aryl or vinylic thiocyanates. However, the reports on the construction of C_{vinyl}-SCN bonds are limited [27–32]. In 1990, Kitamura et al. reported the use of bromoalkenes as substrates to get thiocyanates under photochemical conditions [33].Thereafter, some attractive methods to synthesize C_{vinyl}-SCN compounds were consequently reported by Chen [34] and Kawabata [35] with the use of activated olefins (Scheme 1, eq a). Simultaneously, vinyl thiocyanates were prepared from alkynes and their derivatives by Jiang [36], Dwivedi [37] and Wu [38] (Scheme 1, eq b). It is valuable to remark that most of the previous methods suffer from at least one of the following general drawbacks such as low-to-moderate yields, long reaction time, harsh conditions and use of toxic as well as expensive metals or complex reagents. Thus, the development of greener and straightforward approach for the synthesis of vinyl thiocyanates is highly desirable. Recently, Yang et al. reported the synthesis of thiocyanates from the decarboxylative coupling of cinnamic acids with NH_4SCN via electrochemical protocol [39] (Scheme 1, eq c). However, to the best of our knowledge, there is no report in the literature

Scheme 1 Various synthetic routes to (*E*)-vinyl thiocyanates



Scheme 2 Control experiments



on the visible-light-mediated decarboxylative thiocyanation of cinnamic acids which can overcome the limitations of the previously reported methods. With this background in mind and in continuation of our efforts on visible light photoredox catalysis [40, 41], we have developed the synthesis of vinyl thiocyanates via a visible light induced photoredox catalysis reaction between cinnamic acids and potassium thiocyanates using Rose Bengal as a photoredox catalyst and Cs_2CO_3 as a base (Scheme 1, eq d). This method has advantages over the traditional transition-metal catalyzed cross-coupling reactions in terms of the stereoselectivity, simple operation and easily removable by-product CO_2.

2 Experimental Section

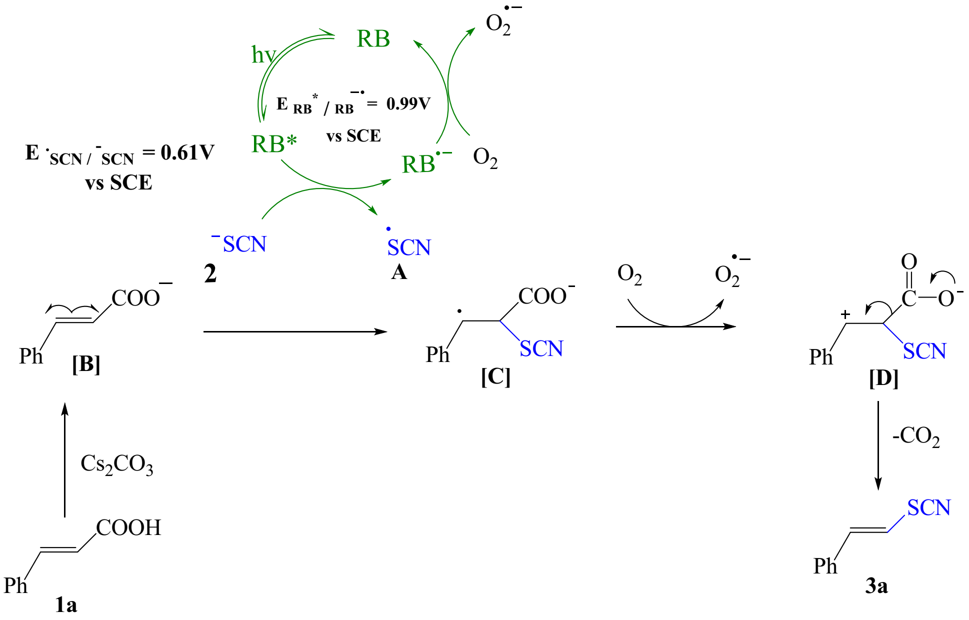
2.1 General Remarks

All chemicals were reagent grade and purchased from Aldrich, Alfa Aesar, Merck, Spectrochem and Qualigens and were used without purification. The reactions were monitored using pre-coated TLC plates of silica gel G/UV-254 of 0.25 mm thickness (Merck 60 F-254). NMR spectra were recorded on a BrukerAvance-II 400FT spectrometer at 400 MHz (^{1}H) and 100 MHz (^{13}C) in $CDCl_3$ using TMS as an internal reference. Mass spectra were recorded on a JEOL SX-102 (FAB) mass spectrometer at 70 eV. Mass Spectra (ESIMS) were obtained on Micromassquadro II spectrometer. Melting points were determined by open glass capillary method and were uncorrected.

3 General Experimental Procedure

In a flame-dried round bottom flask was equipped with a magnetic stirrer bar, cinnamic acid 1 (0.5 mmol, 1 equiv), KSCN 2 (1.5 mmol, 3 equiv), Rose Bengal (1 mol%, 0.005 equiv) and Cs_2CO_3 (1.5 mmol, 3 equiv) were added in 5 mL acetonitrile. The resulting mixture was stirred under irradiation with 23 W CFL at room temperature. Upon completion of the reaction (monitored by TLC) after 12 h, the reaction mixture was quenched with water and extracted with EtOAc (3 × 5 mL). The combined organic layers were dried over Na_2SO_4 and concentrated in vacuo and the crude product

Fig. 1 Probable mechanism



was purified by silica gel chromatography (EtOAc/*n*-hexane, 1:6) to afford an analytically pure sample of (*E*)-vinyl thiocyanates 3.

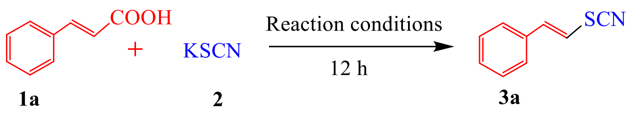
4 Result and Discussion

At the outset of our investigation, a model reaction was performed between cinnamic acid (1a) and potassium thiocyanate (2) using $Ru(bpy)_3Cl_2.6H_2O$ as a photocatalyst and Cs_2CO_3 as a base in THF under the irradiation of 23 W CFL. It was found that desired product (3a) was formed in 72% yield (Table 1 entry1). Encouraged by preliminary success, we screened the various reaction conditions. Initially, we used different photoredox catalysts such as $Ir(ppy)_3$, $Ru(bpy)_3(PF_6)_2$, Eosin-Y and Rose Bengal (Table 1 entries 2–5). Although $Ru(bpy)_3(PF_6)_2$ (Table 1 entry 3) was more effective than Rose Bengal (Table 1 entry 5) but we opted to use Rose Bengal as a catalyst due to its low cost and the transition-metal-free nature. Next, we focused our interest to optimize the solvent. A range of solvents were tested (Table 1 entries 6–9). When CH_3CN was used as the solvent, the yield of product increased to 74% (Table 1 entry 9 vs entry 5). Under neat condition, the desired product (3a) was obtained in 25% yield (Table 1 entry 10). Selection of a suitable base was another important factor which was the cornerstone of our protocol. A series of bases (Na_2CO_3, K_2CO_3, Na_3PO_4, DBU and NaOH) were screened (Table 1 entries 11–15) but Cs_2CO_3 remained the best choice (Table 1 entry 9). We noticed that on increasing the amount of two (1.2 mmol. to 1.5 mmol. or 2.4 equiv to 3.0 equiv), a respectable 79% yield of the product was recorded (Table 1 entry 16) but on further increasing the loading of 2 (1.75 mmol. or 3.5 equiv.) and Cs_2CO_3 (3.5 equiv), there was no significant change in the yield of 3a (Table 1 entry 17). However, the yield of 3a decreased on decreasing the amount of Cs_2CO_3 (2.5 equiv) (Table 1 entry 18). On switching from KSCN to NH_4SCN, again a decrement in the yield of 3a was observed (Table 1 entry 19). We next investigated the visible light source. In the presence of 18 W green LED and blue LED lamps, the desired product was isolated in trace and 20% yield respectively (Table 1 entries 20 and 21). No product formation could be detected in the dark (Table 1, entry 22).

After achieving these results in hand, we focused on investigating the scope of the reaction with respect to the cinnamic acids. As illustrated in Table 2, this reaction was compatible with many functional groups on cinnamic acids. Electron donating groups such as methyl and methoxy groups gave 58–82% yield of target molecules (Table 2, 3b-3f). It was noticed that electron withdrawing groups such as CHO, CN, $-CF_3$ and $-NO_2$ on the aromatic ring of cinnamic acids (Table 2, entries 3g, 3l, 3n and 3p) gave lesser yield of the decarboxylative product in comparison to the electron donating groups. A 12% yield of 3o was found when 3-(2-pyridyl) acrylic acid (1o) was used. The substrate 3, 3-Diphenylacrylic acid (1q) also gave low yield of the corresponding thiocyanation product (Table 2, 3q). Unfortunately, no product (3r) was obtained when (*E*)-4-phenylbut-2-enoic acid (1r) was used as the substrate.

Several control experiments were performed to shed light on the reaction pathway, the results of which are illustrated in **Scheme 2**. When 3 equivalents of TEMPO (a radical scavenger) were added to the reaction mixture, no yield of the

Table 1 Optimization of reaction conditions



Entry	Catalyst	Base	Solvent	Yield[b]
1	$Ru(bpy)_3Cl_2.6H_2O$	Cs_2CO_3	THF	72
2	$Ir(ppy)_3$	Cs_2CO_3	THF	30
3	$Ru(bpy)_3(PF_6)_2$	Cs_2CO_3	THF	75
4	Eosin Y	Cs_2CO_3	THF	40
5	Rose Bengal	Cs_2CO_3	THF	67
6	Rose Bengal	Cs_2CO_3	toluene	30
7	Rose Bengal	Cs_2CO_3	DMSO	16
8	Rose Bengal	Cs_2CO_3	EtOH	41
9	Rose Bengal	Cs_2CO_3	CH_3CN	74
10	Rose Bengal	Cs_2CO_3	Neat	25
11	Rose Bengal	Na_2CO_3	CH_3CN	trace
12	Rose Bengal	K_2CO_3	CH_3CN	n.d
13	Rose Bengal	Na_3PO_4	CH_3CN	10
14	Rose Bengal	DBU	CH_3CN	40
15	Rose Bengal	NaOH	CH_3CN	n.d
16	Rose Bengal	Cs_2CO_3	CH_3CN	79[c]
17	Rose Bengal	Cs_2CO_3	CH_3CN	80[d]
18	Rose Bengal	Cs_2CO_3	CH_3CN	68[e]
19	Rose Bengal	Cs_2CO_3	CH_3CN	25[f]
20	Rose Bengal	Cs_2CO_3	CH_3CN	n.d.[g]
21	Rose Bengal	Cs_2CO_3	CH_3CN	20[h]
22	Rose Bengal	Cs_2CO_3	CH_3CN	n.d[i]

[a] Reaction conditions: cinnamic acid (**1a**, 0.5 mmol, 1 eq.), KSCN (**2**, 1.2 mmol, 2.4 eq.), catalyst (1 mol %), solvent (5 mL), base (1.5 mmol, 3 eq.), open to air, irradiation under a 23 W CFL at room temperature for 12 h

[b] Isolated yield

[c] **2** (1.5 mmol. or 3.0 eq.)

[d] **2** (1.75 mmol. or 3.5 eq.) and Cs_2CO_3 (3.5 eq.)

[e] Cs_2CO_3 (2.5 eq.)

[f] NH_4SCN was used in place of KSCN

[g] 18 W green LED lamps

[h] 18 W blueLED lamps

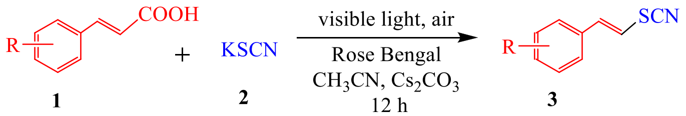
[i] In the dark. n.d. (Not detected)

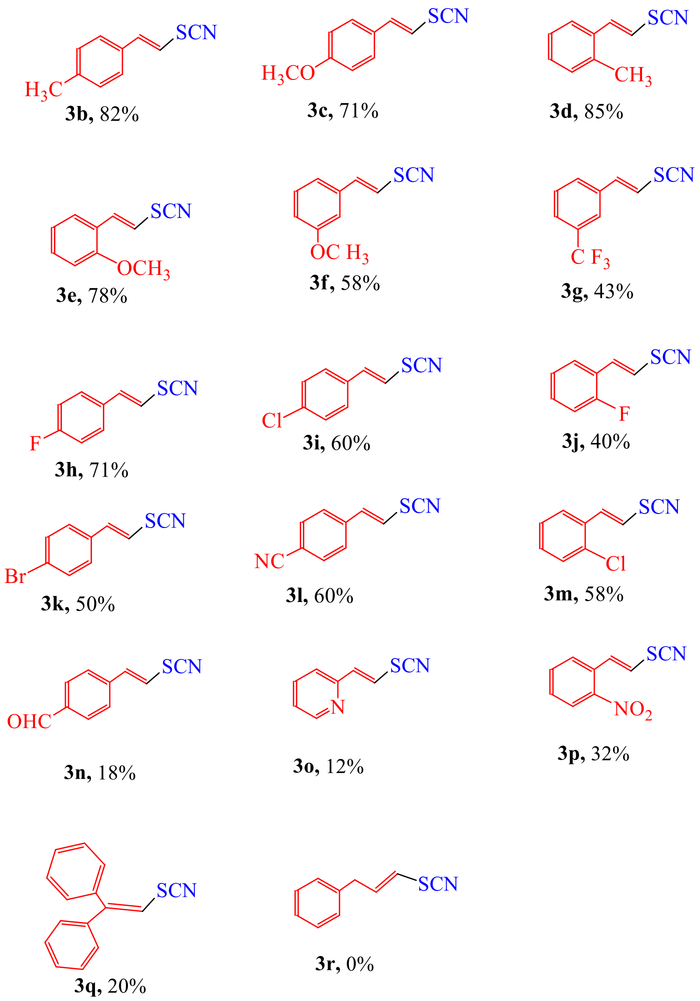
product was obtained which confirms that the reaction was proceeding through a radical pathway. In the absence of visible light, Cs_2CO_3, Rose Bengal and O_2, product formation was inhibited, thus verifying that reproducible construction of 3a involves all of these.

4.1 Mechanism

Based on above investigation and literature reports [42–44], a tentative mechanism has been proposed in Fig. 1. On exposure to visible light, a thiocyanate radical A is generated via single electron transfer to excited-state species Rose Bengal RB*, which is then converted into $RB^{\cdot -}$. The $RB^{\cdot -}$ further undergoes single electron transfer to molecular oxygen to complete the photoredox cycle. Simultaneously, base abstracts the proton from cinnamic acid 1a to form intermediate (B). Subsequently, radical A attacks intermediate (B) leading to the formation of intermediate (C) which is further oxidized to cationic intermediate (D) (via O_2 to $O_2^{\cdot -}$). Finally, intermediate (D) undergoes decarboxylation to yield the final product 3a. Detection of H_2O_2 by KI/starch indicator proves the generation of the superoxide radical anion during the reaction [45].

Table 2 Substrate Scope [a, b]





[a] Reaction conditions: cinnamic acids (1, 0.5 mmol, 1 eq.), KSCN (1.5 mmol, 3 eq.), Rose Bengal (1 mol %), CH_3CN (5 mL), Cs_2CO_3 (1.5 mmol, 3 eq.), open to air, irradiation under a 23 W CFL at room temperature for 12 h.

[b] Isolated yield.

5 Conclusion

In conclusion, we have developed a novel technology for vinyl thiocyanate formation via transition-metal-free visible light photoredox catalysis. We have synthesized a series of vinyl thiocyanates having numerous useful functionalities. They could be easily produced in good-to-excellent yields with only carbon dioxide as the byproduct under considerably mild reaction conditions. The established protocol, in contrast with the conventional methods, refrains from the use of a strong oxidant, transition-metal catalyst and high temperature. Moreover, this method provides a straightforward and environmentally benign process for the construction of vinyl thiocyanates from cinnamic acid and KSCN via decarboxylative radical pathway.

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Compliance with Ethical Standards

Conflicts of interest The authors declares that there is no conflicts of interest.

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