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ARTICLE

Visible-light-initiated, metal-free photocatalyzed synthesis of tosyl hydrazone

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Herein, we report a novel visible-light-induced, photoredox-catalyzed, eco-friendly one-pot method for the synthesis of tosyl hydrazone using the organic dye rose bengal, since its synthesis under visible light irradiation had not been reported before. The reaction exhibited a wide substrate scope, strong tolerance to various functional groups, enabling the synthesis and modification of pharmaceutical compounds. The proposed mechanism is supported by both experimental and density functional theory (DFT) calcultions.

Introduction

Tosyl hydrazone/Sulfonyl hydrazones are commonly found as structural units in natural products, many of which exhibit significant biological properties, including insecticidal activity, enzyme inhibition, anti-oxidant, anti-alzheimer, anti-bacterial, anti-fungal, anti-depressant, and anti-cancer activities[1–15]. In addition, sulfonyl hydrazones display significant chemical diversity and act as crucial intermediates in a wide range of organic transformations[16,17] and can be used as an alternative precursor for hazardous diazo compounds[18], carbene sources[19,20], and umpolung alkylation[21].

Due to their significant roles in chemistry and biology, sulfonyl hydrazones have been extensively studied and synthesized through various methods. Conventionally, this transformation involves the coupling of aldehydes with activated sulfonyl derivatives, typically sulfonyl chlorides[4,22,23]. While current strategies for synthesizing sulfonyl hydrazones can be effective, they often involve multiple steps, narrow substrate scopes, and the use of additives. To overcome these challenges, Yang et al. and Singh et al. reported one-pot approaches for the synthesis of sulfonyl hydrazones using sulfonyl hydrazides as precursor via a grinding method[24], and by employing a gold-catalyst[25] respectively, this method also suffers from limited substrate compatibility. Because these reactions still involve the utilization of unfavourable conditions, they are still insufficient for green and sustainable chemistry, despite their effectiveness and generality. Therefore, finding a straightforward, efficient, cost-effective, and ecologically friendly method to produce sulfonyl hydrazones is still highly desired.

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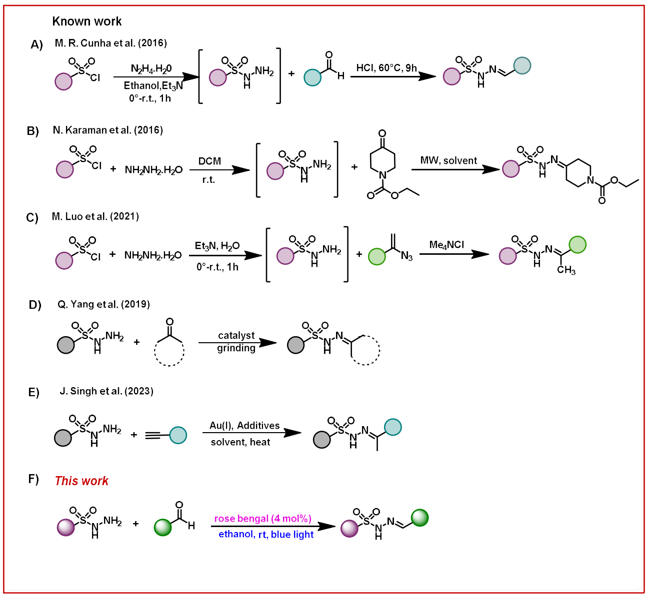
In recent years, visible light photoredox catalysis has emerged as a green and effective synthetic approach[26–29], particularly with the advancement of organic dyes as metal-free photocatalysts characterized by highly oxidative excited states[30,31]. Rose bengal, a metal-free photocatalyst possessing remarkable photoredox properties, serves as a inexpensive and readily accessible catalyst. It has evolved into an excellent catalyst for organic molecule synthesis and directed functionalization.[32,33]

The growing interest in using organic dyes, rose bengal, activated by photoenergy, has opened up promising avenues for developing environmentally benign and efficient synthetic methods. Inspired by recent advancements in sustainable chemistry and our ongoing efforts in photoinduced organic transformations[34,35], we have developed first time a green, efficient, mild, and metal-free protocol for the synthesis of tosyl hydrazones (Scheme 1).

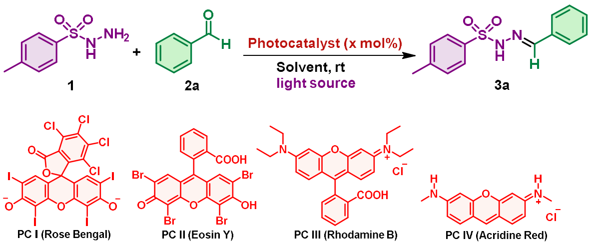


Scheme 1: Latest strategies for sulfonyl hydrazone synthesis

Results and discussion

To support our hypothesis, we started our investigations with the optimization of the reaction conditions for the synthesis of tosyl hydrazones (Table 1). Our studies were initiated to find the model reaction between *p*-toluenesulfonyl hydrazide **(1)** and benzaldehyde **(2a)** under visible-light-irradiation of a 40W Kessil lamp (440nm), screening photocatalyst i.e., rose bengal and ethanol as solvent for 24 hours, the transformation produced the desired product **(3a)** in 89% yield (Table 1, entry 1). The influence of different photocatalysts was evaluated by substituting PC I with metal-free alternatives PC II, PC III, and PC IV, which resulted in a decreased yield of the product (Table 1, entry 2). Further screening of various solvents (Table 1, entries 3-8) concluded that ethanol is a suitable solvent for the reaction medium for the present photocatalytic transformation (Table 1, entry 5). Variation in rose bengal loading revealed that 4 mol% remained the optimal catalyst amount (Table 1, entry 10). A decrease in product yield was observed upon changing the light source (Table 1, entry 11). Both the light source and photocatalyst were found to be indispensable for the reaction, as no product formation was observed in the absence of either light or the photocatalyst (Table 1, entries 12 and 13). Notably, variations in reaction time had some significant impact on the yield of the desired product (Table 1, entry 14). Detailed optimization procedures are described in the Supporting Information (see SI Pg. S5-S6).

Table 1 Optimization of the reaction conditions[(a)]



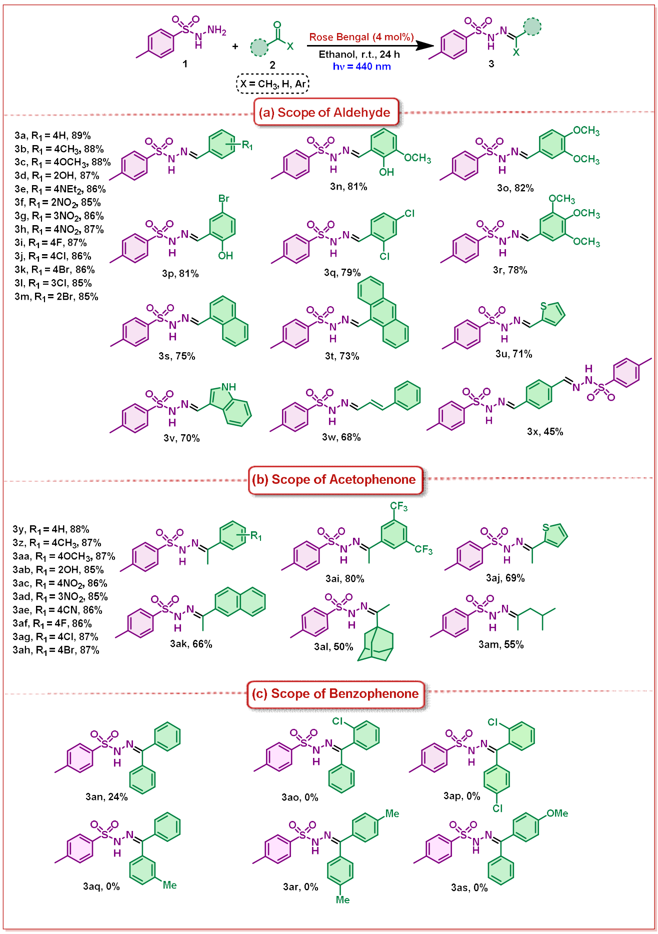
Entry	Deviation from standard condition	Yield[(b)] (%)
1	None [(a)]	89
2	PC II, PC III, PC IV instead of PC I	trace
3	Methanol instead of ethanol	30
4	H_2O instead of ethanol	49
5	Ethanol instead of MeCN	52
6	DMF instead of ethanol	25
7	DMSO instead of ethanol	29
8	Non-polar solvents	11-32
10	1, 2, 3, 4 mol% of rose bengal	62-89
11	Different LEDs instead of blue light	70-80
12	Without PC	nr
13	In dark	nr
14	16, 18, 20 h instead of 24 h	62-82

[(a)]Reaction conditions: *p*-toluene sulfonyl hydrazide **1** (0.5 mmol), benzaldehyde **2a** (0.5 mmol), photocatalyst (x mol %), solvent (7 mL).

[(b)]Isolated yield

With the optimized conditions established, we next investigated the generality of our model reaction with a wide range of aldehyde and ketone derivatives as shown in (Scheme 2).

We initially explored a range of aromatic aldehydes, and the results demonstrated that this methodology has broad functional group compatibility. Aromatic aldehydes bearing diverse electronic and steric features on their aryl rings were well tolerated, affording the desired products in good to excellent yields (Scheme 2a). Moreover, the position of substituents on the aromatic ring whether *ortho*, *meta*, or *para* exerted minimal influence on the reaction efficiency, as evidenced by the consistently high yield of products **(3b–3m)**. Notably, the reaction exhibited excellent tolerance toward both electron-donating groups, such as methyl **(3b)** and methoxy **(3c)**, hydroxyl **(3d)**, and electron-withdrawing groups, including fluoro **(3i)**, chloro **(3j, 3l)**, bromo **(3k, 3m)**, and nitro **(3f-3h)**. Disubstituted and trisubstituted aldehydes bearing both electron-donating and electron-withdrawing groups successfully furnished the corresponding tosyl hydrazones in 78–81% yields **(3n–3r)**. Next, we turned our attention to polycyclic aldehydes, which afforded the desired product in comparatively lower yields, 75-73% **(3s, 3t)**, possibly due to steric hindrance imposed by the fused ring system. Meanwhile, heterocyclic aldehydes containing indole and thiophene ring systems afforded the corresponding products in moderate yields of 70–71% **(3u, 3v)**. Notably, the conjugated aldehyde afforded a lower yield of 68% **(3w)**, while the dicarboxaldehyde gave a significantly reduced yield of 45% **(3x)**, likely due to both formyl groups reacting with **1**.



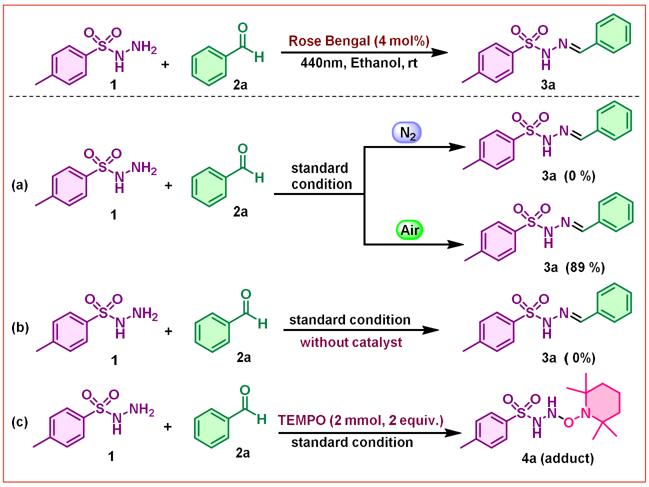
Scheme 2: Substrate generality of aldehydes and ketones(a)

(a)Reaction condition : *p*-toluene sulfonyl hydrazide (2 mmol), aldehydes/ketones (2 mmol), rose bengal (4 mol %), solvent (20 mL), 440nm (24 h) under open air at room temperature.

Encouraged by the positive outcomes observed in the substrate scope study of aldehydes, we next explored the reaction with various ketone derivatives. The reaction tolerated a wide range of substituents like halogens **(3af-3ah)** and other functional groups such as methyl **(3z)**, methoxy **(3aa)**, hydroxy **(3ab)**, nitro **(3ac, 3ad)**, and cyano **(3ae)** were well tolerated at the *ortho*, *meta*, and *para* positions, affording the desired products in yields of 85% to 88%. Next, we examined disubstituted ketones, which afforded the product in 80% yield **(3ai)**. The thiophene-containing derivative gave a moderate yield of 69% **(3aj)**, while the naphthalene-based substrate also provided a moderate yield of 66% **(3ak)**. Our protocol was also compatible with aliphatic ketones, both cyclic and acyclic, resulting in decreased yield of 50% **(3al)** and 55% **(3am)**, respectively.

Encouraged by the positive results obtained with acetophenone derivatives (Scheme 2b), we turned our attention to benzophenone derivatives to further evaluate the scope of the method. However, benzophenone itself afforded a low yield of 24% **(3an)**, and its substituted derivatives failed to produce the desired products **(3ao–3as)**, likely due to steric hindrance from the bulky aryl substituents (Scheme 2c).

To gain insight into the reaction mechanism, a series of control experiments was performed. Under standard conditions, when **1** and **2a** were reacted in a nitrogen (inert) atmosphere, the desired product **3a** was not formed which clearly shows that the reaction will proceed under open environment. Hence, performing the reaction in open air resulted in maximum yield, indicating that atmospheric oxygen is essential for the reaction (Scheme 3a). When the same reaction conditions were applied without the photocatalyst, the desired product 3a was not formed indicating the necessity of photocatalyst in the reaction condition (Scheme 3b). Next, we perform a reaction between 1 and 2a in the presence of 2 mol% of 2,2,6,6-tetramethylpiperidinyloxy (TEMPO) under standard conditions; the desired product 3a was not formed (Scheme 3c). Instead, TEMPO adduct **(4a)** was detected by high-resolution mass spectrometry (HRMS). This result implied that the *p*-toluene sulfonyl hydrazide radical **C** (Scheme 5) acts as the key reactive intermediate in the reaction.



Scheme 3: Control experiments

To gain deeper insight into the mechanism of the coupling reaction, we carried out a series of mechanistic investigations (Scheme 4). UV/visible spectroscopy was conducted on the individual reactants, and the reaction mixture (Scheme 4a). The findings showed that the reactants did not absorb in the visible region, whereas the reaction mixture containing **1**, **2a** and rose bengal exhibited absorbance within this range.

Subsequently, we performed a series of fluorescence quenching experiments, which showed that excited rose bengal (RB*) was effectively quenched by **1** but not by **2a** alone (Scheme 4c, d). A linear relationship was observed between the extent of photocatalyst quenching and the concentration of **1** clearly demonstrating a direct interaction between excited rose bengal and **1** but with **2a** no such interaction has been observed (Scheme 4e). Moreover, intermediate **C** was found to be the effective quencher of the excited state of **RB**. In addition, light/dark cycle experiment revealed that the reaction was paused in the absence of visible light and resumed upon re-irradiation, confirming the necessity of continuous light throughout the process (Scheme 4b). Furthermore, cyclic voltammatry (CV) analysis of **1**, **2a**, and **RB** was performed to determine their redox potentials. The results revealed that **RB** exhibited the highest redox potential **(+0.86 V)**, indicating its ability to readily accept electrons and undergo reduction.

In contrast, **1** showed the lowest redox potential **(+0.83 V)**, while **2a** displayed a slightly higher value **(+0.85 V)**. Therefore, among **1** and **2a**, reactant **1** is more prone to electron loss. These findings confirm that electron transfer occurs from **1** to **RB**, supporting the proposed single electron transfer **(SET)** mechanism. 10

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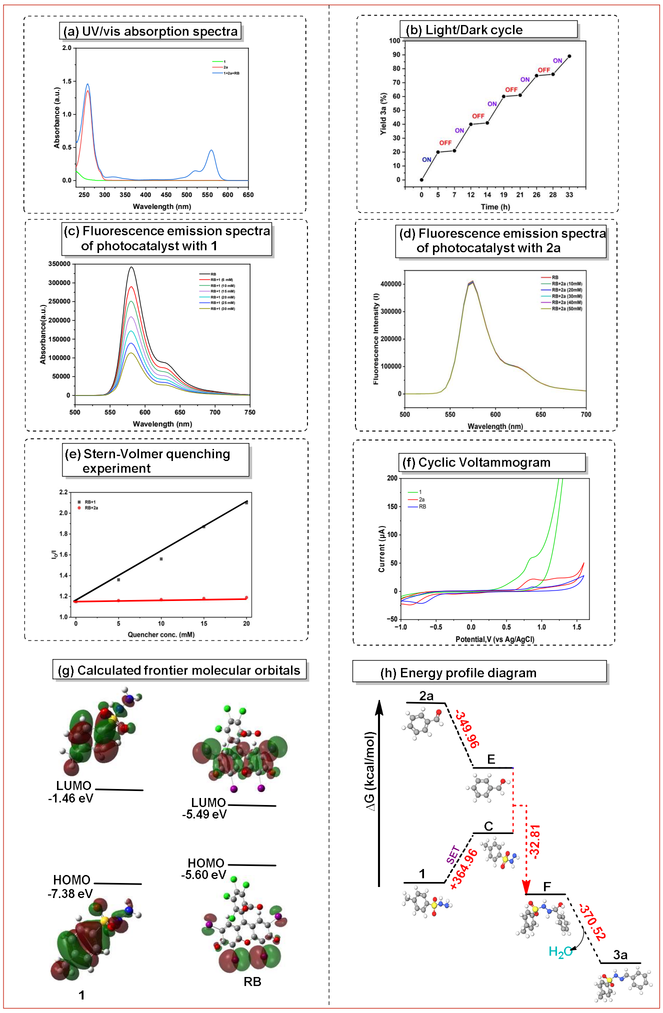
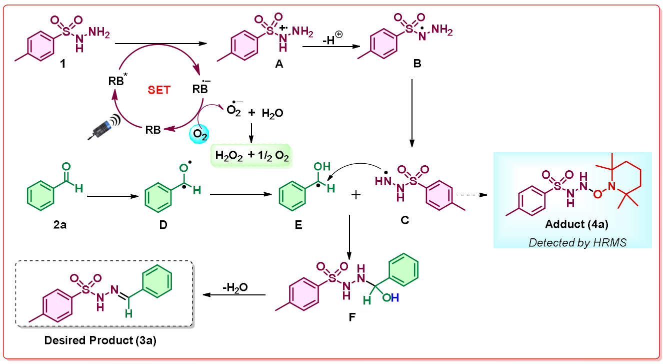


Scheme 4: Mechanistic investigations

10 28 Additionally, to confirm the spontaneity of the reaction and to gain insight into the reaction mechanism, Density Functional Theory (DFT) calculations were performed using Gaussian software to determine the Gibbs free energy changes associated with the reaction pathway. The calculated free energy (thermal+electronic) energy difference between reactant **1** and intermediate **C** was found to be **+364.96 kcal/mol**, indicating that the formation of the radical species **C** from **1** is a non-spontaneous process. Therefore, the use of a photocatalyst (RB) is essential to facilitate this transformation. In contrast, the conversion of reactant **2a** into species **E** exhibited a negative free energy (thermal+electronic) energy difference **-349.96 kcal/mol**, suggesting that this step is spontaneous and proceeds readily under 5 visible-light-irradiation, leading to the formation of intermediate **D** and eventually into **E**. Furthermore, DFT results confirm that the coupling of species **E** and **C** is also a spontaneous process with an energy difference of **-32.81 kcal/mol**. Finally, the formation of the product **3a**, accompanied by the elimination of a water molecule, was found to be highly favourable, with a negative energy difference of **-370.52 kcal/mol** (for detailed energy profile diagram see Scheme 4h). We have also calculated the HOMO–LUMO energy gap of reactant **1** and **RB** to evaluate whether the reaction proceeds via a single electron transfer (SET) mechanism. The results revealed that the LUMO of **RB** lies at a lower energy **(-5.49 eV)** level than the HOMO of **1 (-7.38 eV),** indicating that an electron transfer from 1a to RB is energetically favourable, leading to the formation of the radical cation **C** confirms SET mechanism (see Scheme 4g).

Based on control experiment and mechanistic investigations we have proposed the following reaction mechanism, as shown in (Scheme 5). At the beginning, the photocatalyst **RB** was excited to its excited state **RB*** under visible light irradiation. The RB* then reduced 1a into radical cation **(A)** *via* a single electron transfer (SET) process, and the reduced RB returns to its ground state by donating its electron to atmospheric O_2, which is subsequently converted into a superoxide ion[36]. This superoxide ion then reacts with H_2O to produce hydrogen peroxide and an O_2 molecule. The species **(A)**, upon deprotonation, is converted into radical **(B)**. Through rearrangement, radical **(B)** converts into radical **(C)**. Conversely, benzaldehyde **(2a)** absorbs visible light, generating biradical **(D)**. This species **(D)** subsequently abstracts a proton to produce intermediate **(E)**. Finally, radical **(C)** then couples with intermediate **(E)**, producing the target compound **3a** with the elimination of water.



Scheme 5: Plausible mechanism for the synthesis of tosyl hydrazone

Conclusions

In conclusion, we have established for the first time a visible-light-driven method for synthesizing tosyl hydrazones *via* metal-free photoredox catalysis in an environmentally friendly solvent. This approach enables the efficient preparation of a broad range of tosyl hydrazones in good to excellent yields. The method is cost-effective, atom-efficient, and eco-friendly. Notably, it introduces a novel photoredox-catalyzed C–N bond formation between sulfonyl hydrazine and carbonyl compounds (aldehydes/ketones) to yield the corresponding tosyl hydrazones, offering significant potential for applications in organic synthesis. Furthermore, this mechanistically unique photoredox process opens new avenues for developing innovative transformations in photoredox catalysis.

Data availability

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

Conflicts of interest

There are no conflicts to declare.

Acknowledgments

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