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Visible-light Driven Eosin Y Catalyzed C(sp^2)-H Functionalization/C–O Bond Formation for Synthesis of Benzoxazoles

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Abstract: Visible light mediated synthesis of benzoxazoles from benzanilides under an air atmosphere at room temperature is reported using eosin Y as an organophotoredox catalyst by functionalization of C–H/C–O bond formation. This methodology accepts a broad range of functional groups and affords the benzoxazoles by transition-metal-free organic photoredox catalysis under very mild conditions.

Keywords: visible light, eosin Y, photocatalyst, heterocycle, benzoxazoles.

INTRODUCTION

THE functionalization of C–H bond has been considered as a sustainable and innovative method for molecular synthesis.[1] The formation of C–O bonds has received less attention from inert C–H bonds than the well-established C–C and C–N bond building methods.[2] This is caused in part by the absurdly high energy barrier between the frontier orbitals of the M–O HOMO and M–C LUMO atomic systems.[3] The C–H functionalization/C–O bond formation technique that is operationally straightforward, effective, and feasible is still very much preferred.[4]

The development of feasible and efficient methods for the synthesis of benzoxazoles is garnering a huge amount of attention. Among the most significant heterocyclic rings, benzoxazoles are widely present in natural products, advanced materials, pharmaceuticals, and biologically active compounds.[5–8] Because of the diverse range of pharmacological effects that benzoxazoles exhibit, such as antibacterial,[9] antifungal,[10] anticancer,[11] anti-inflammatory,[12] anti-mycobacterial,[13] antihistamine,[14] anti-parkinson,[15] inhibition of hepatitis C virus,[16] 5-HT3 antagonistic effect,[17] melatonin receptor antagonism,[18] amyloidogenesis inhibition[19] and Rho-kinase inhibition.[20] There are many commercially available drugs using benzoxazole as the main active component, including non-steroidal anti-inflammatory drug (NSAID) flunoxaprofen, benoxaprofen, antibiotic calcimycin, antibacterial boxazomycin B, muscle relaxant chloroxazone. Furthermore, the isolated benzoxazole alkaloids[21–25] from marine sponges or plants have strong medicinal activity. (Figure 1)

Among all of the various protocols to synthesise benzoxazole, direct C(sp^2)-H activation using UV or visible light to produce intramolecular C–O bonds have recently attracted increasing attention.[26–34] Because solar energy (visible light) is pure, simple to utilise, and an endless energy source, there are many opportunities for the creation of environmentally beneficial and sustainable protocols for organic synthesis.[35–40] The conversion of solar energy into chemical energy for chemical reactions is the focus of several trailblazing researchers,[41,42] who have also developed a promising method for using photoredox catalysts to start single electron transfer (SET) processes.[43–45] Recently, a superior alternative to transition metal[46,47] photoredox catalysts, especially metal-free organic dyes such as eosin Y, fluorescein, rose bengal, nile red, perylene

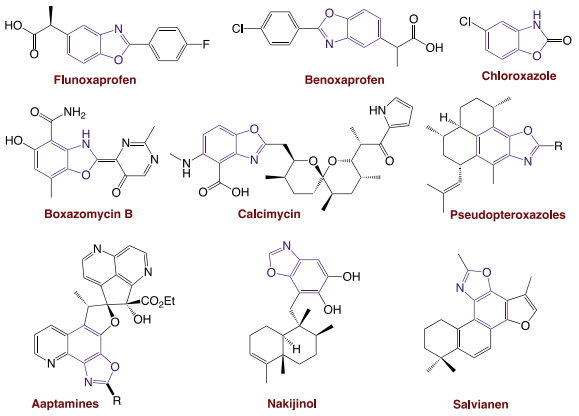


Figure 1. Chemical structure of some biologically important benzoxazole drugs.

and rhodamine B have been used as economically and ecologically superior surrogates to carry out organic transformations. In the present study, we devoted to further explore the employment of visible-light photoredox catalyst in the cyclization of benzanilides in a sustainable approach. In continuation of our work on development of environmentally benign synthesis[48] herein we proposed the application of a natural organic dye eosin Y, as photocatalyst using visible blue light to promote the transition-metal complexes and base free chemical reaction under mild conditions. (Scheme 1).

EXPERIMENTAL

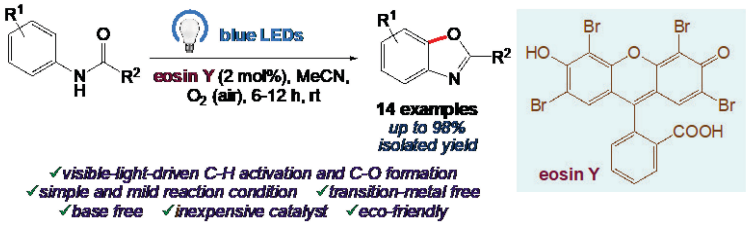
All materials used are commercially available and were purchased from Sigma-Aldrich and used without any additional purification. Melting points were determined by open glass capillary method and are uncorrected. All chemicals used were reagent grade and were used as received. ^{1}H NMR and ^{13}C NMR spectra were recorded on a Bruker AVANCE DPX (400 MHz and 75 MHz) FT spectrometer in $CDCl_3$ using TMS as an internal reference (chemical shift in δ ppm).

General Procedure for the Preparation of Products (2a–n)

The reactions were carried out in a 10 mL glass vial, equipped with a rubber septum and a magnetic stirrer. Substituted benzanilides (**1a**–**n**, 0.2 mmol), and Eosin Y (2 mol%) were dissolved in acetonitrile (4 mL) and the mixture was irradiated with a 3 W blue-LED (467 nm) under an air atmosphere at rt for 6-12h. After completion of the reaction (monitored by TLC), the reaction crude was placed into a separatory funnel, water (5 mL) was added and the mixture was extracted with EtOAc (3 × 5 mL). The organic layer was separated, and the aqueous layer was extracted with ethyl acetate (2 × 15 mL). The combined organic extract was dried over anhydrous $MgSO_4$, filtered and evaporated under reduced pressure. Finally, the products (**2a**–**n**) were isolated by column chromatography (silica gel, eluent: 8 : 2 hexanes/ethyl acetate).

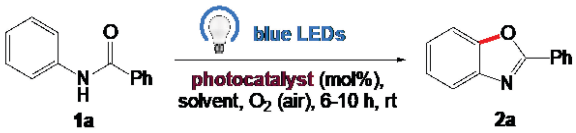
RESULTS AND DISCUSSION

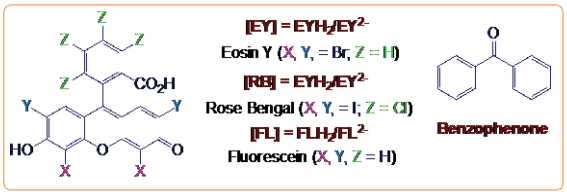
In this current study, we focused on extending our understanding to exploit aromatic C–H oxidation reactions of benzanilides in a sustainable manner. In the absence of transition-metal complexes and bases, we proposed the utilization of natural organic dye, Eosin Y as a photocatalyst using visible blue light to expedite the process. In order to work out the envisaged protocol, a key reaction was conducted with benzanilides **1(a–n)** in MeCN containing 2 mol% of eosin Y under an air atmosphere (without air bubbling) by irradiation with visible light (blue LEDs 3 W) at rt. The reaction delivered the desired substituted benzoxazoles **2(a–n)** in 98 % isolated yield after 6h (Table 1, entry 1). Following this experiment, a series of control experiments were performed, which indicates that an organic dye is essential to give the desired product with high yield (98 %) (Table 1, entry 1) and Eosin Y was found to be the best organic dye (Table 1, entry 1 versus 2, 3, 4). There was no product formation or it was formed in traces in the absence (−) of any one of the reagents/catalyst (Table 1, entries 11−13). The reaction did not proceed satisfactorily when a household 20 W fluorescent lamp was used instead of blue LEDs (Table 1, entries 1 versus 10). In the absence of any gas or under a nitrogen atmosphere no product formation was detected (Table 1, entry 11). These results establish that visible light, photocatalyst and air all are essential (+) for the reaction and support the photocatalytic model of the reaction.



Scheme 1. Synthesis of benzoxazoles.

Table 1. Optimization of reaction conditions[a].



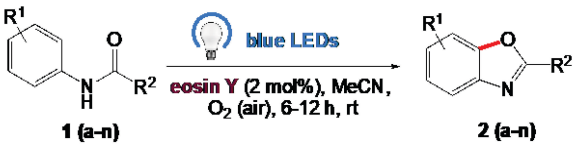


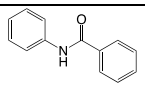
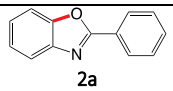
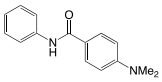
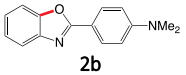
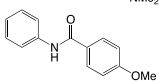
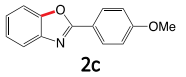
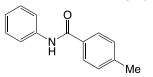
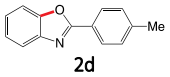
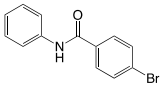
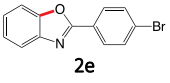
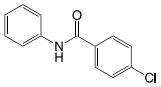
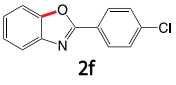
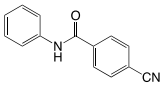
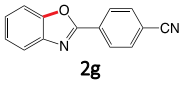
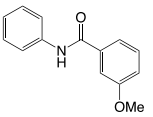
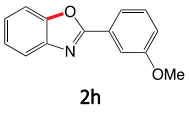
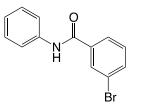
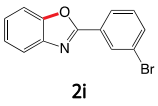
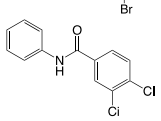
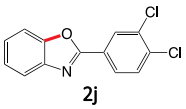
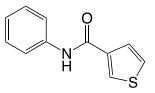
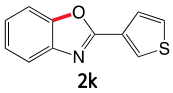
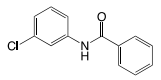
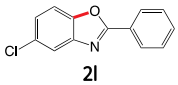
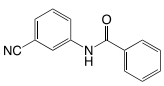
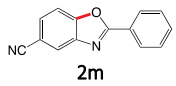
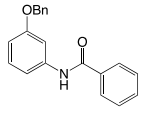
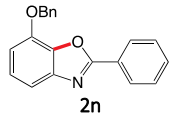
Entry	Photo-catalyst	Catalyst loading / mol %	Solvent	Atmos-phere	Time / h	Yield[b] / %
1	Eosin Y	2	MeCN	O_2	6	98
2	Rose Bengal	2	MeCN	O_2	6	87
3	Fluorescein	2	MeCN	O_2	6	83
4	Benzophenone	2	MeCN	O_2	6	78
5	Eosin Y	2	EtOH	O_2	6	66
6	Eosin Y	2	DMSO	O_2	6	68
7	Eosin Y	2	$CHCl_3$	O_2	6	61
8	Eosin Y	1	MeCN	O_2	6	69
9	Eosin Y	3	MeCN	O_2	6	98
10	Eosin Y	2	MeCN	O_2	6	48[c]
11	Eosin Y	2	MeCN	N_2	10	n.d[d]
12	Eosin Y	2	MeCN	O_2	10	Trace[e]
13	–	–	MeCN	O_2	10	n.d.[f]

[a] Reaction conditions: benzanilides (1.0 mmol), eosin Y (2.0 mol%), MeCN (3.0 mL), blue LEDs 3 W, irradiation under an air atmosphere at rt.
[b] Isolated yield of the product (2a).
[c] The reaction was carried out using 20 W CFL (compact fluorescent lamp).
[d] Reaction was performed under N_2 atmosphere.
[e] Reaction was performed in the dark.
[f] Reaction was carried out without catalyst.

Next, the reaction conditions were optimized with respect to solvents and the catalyst used in the reaction. In all the tested solvents (MeCN, EtOH, DMSO and $CHCl_3$) the yield of **2(a–n)** was > 60 % (Table 1), which indicates that the reaction is not very sensitive to reaction media. MeCN was the best solvent in terms of the reaction time and yield (Table 1, entry 1), hence it was used throughout the synthesis. When the amount of the catalyst was decreased from 2 mol % to 1 mol %, the yield of **2(a–n)** considerably reduced (Table 1, entry 8), but the use of 3 mol % of the catalyst did not affect the yield (Table 1, entry 9). Benzoxazoles exhibit various biological activities and has greater synthetic utility in medicinal chemistry. The use of novel one-pot visible light irradiated synthesis using eosin Y as an organophotoredox catalyst fulfil the basic need of green chemistry.

Table 2. Visible-light photocatalysed synthesis of benzoxazoles.



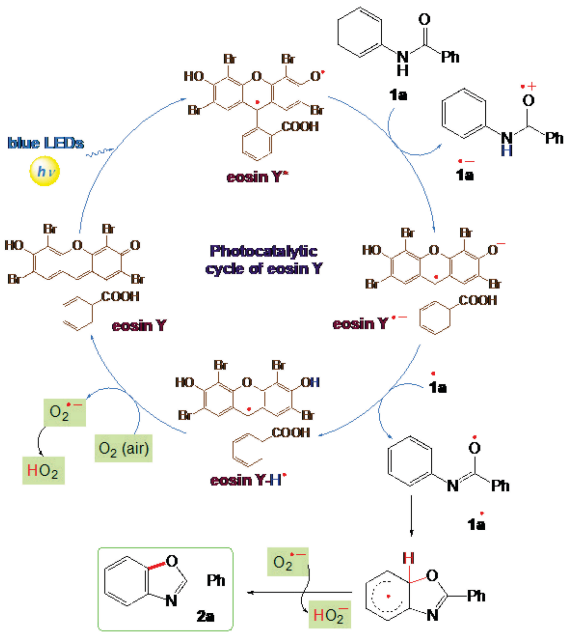
Entry	Substrate	Product	Time / h	Yield / %
1		 2a	6	98
2		 2b	10	65
3		 2c	8	82
4		 2d	9	70
5		 2e	7	93
6		 2f	7	94
7		 2g	8	85
8		 2h	8	78
9		 2i	6	90
10		 2j	6	96
11		 2k	10	65
12		 2l	12	48
13		 2m	12	55
14		 2n	10	67

Under the established reaction conditions in hand, the reaction was tried in a one-pot procedure starting

directly from benzanilides **1(a–n)** to give the desired product **2(a–n)** as depicted in Scheme 2. To our delight, it worked well and a number of substituted benzoxazoles were successfully synthesized starting directly from starting material. (Table 2).

This clearly shows that the reaction is very mild and tolerates considerable functional group variations like, OMe, Me, Cl, Br, CN and OBn (benzyloxy) in the substrate **1(a–n)**, which results the desired product **2(a–n)** in good to excellent yields (48–98 %). Different para-substitutions at the oxygen substituted-phenyl moiety were checked, and excellent results were achieved with *p*-Cl and *p*-CN substituents (Table 2). Furthermore, good to acceptable yields were attained with the electron-donating substituent *p*-OMe, and the effect of meta-substitutions was also examined in substrates (Table 2).

On the basis of the above observations and the literature precedents, a plausible mechanism involving photoredox catalysis for the oxidative cyclization of benzanilides is depicted in Scheme 2. On absorption of visible light, singlet and/or triplet excited states of Eosin Y, the benzanilides can act as a quencher by an electron-transfer reaction, giving rise to the corresponding benzanilide radical cation (1a•+) together with the radical anion Eosin Y•–. The important difference between Eosin Y and other catalysts can explain the lack of needing a base in our protocol. Moreover, it can also explain the reactivity of the substrate with strong electron-withdrawing substituent. The deprotonation of the substrate occurs first with other photocatalysts, where the presence of a base is necessary, and the reaction does not proceed. Deprotonation of the benzanilide radical cation 1a•+ gives the oxygen radical that after cyclization and further rearomatization by any oxidant species affords phenylbenzoxazole. O_2•– is necessary to close the catalytic cycle and return Eosin Y to its ground state.



Scheme 2. Plausible mechanism involving photoredox catalysis for the oxidative cyclization of benzanilides.

CONCLUSION

In conclusion, we have developed an efficient photochemical organocatalysed protocol for the synthesis of substituted benzoxazoles via C–H functionalization, directly from benzanilides in a one-pot procedure by using inexpensive eosin Y as a powerful organophotoredox catalyst at rt. The reaction involves visible light, and O_2 (air) as valuable reagents. Moreover, the use of Eosin Y as photocatalyst exhibits an important advancement over transition-metal catalysis and other organic photocatalysts that does not require the use of strong bases. The present methodology also offers many advantages of green chemistry such as high atom economy, reduced reaction time, one-pot consolidated procedure and high efficiency that could be advantageous for pharmaceutical uses

Supplementary Information. Supporting information (^{1}H and ^{13}C NMR spectra for all synthesized compounds) to the paper is attached to the electronic version of the article at: https://doi.org/10.5562/cca3927.

PDF files with attached documents are best viewed with Adobe Acrobat Reader which is free and can be downloaded from Adobe's web site.

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