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Photochemical synthesis of pyrano[2,3-d] pyrimidine scaffolds using photoexcited organic dye, Na₂ eosin Y as direct hydrogen atom transfer (HAT) photocatalyst via visible light-mediated under air atmosphere

Farzaneh Mohamadpour*

Abstract

The Knoevenagel-Michael cyclocondensation of barbituric acid/1,3-dimethylbarbituric acid, malononitrile, and arylaldehyde derivatives was used to construct a multicomponent green tandem method for the metal-free synthesis of pyrano[2,3-d]pyrimidine scaffolds. At room temperature in aqueous ethanol, photo-excited state functions generated from Na₂ eosin Y were employed as direct hydrogen atom transfer (HAT) catalysts by visible light mediated in the air atmosphere. This research looks towards expanding the use of a non-metallic organic dye that is both affordable and readily available. Because of its good yields, energy-effectiveness, high atom economy, time-saving qualities of the reaction, and operational simplicity, Na₂ eosin Y is photochemically produced with the least amount of a catalyst. As a result, various ecological and sustainable chemical properties are met. Surprisingly, such cyclization may be carried out on a gram scale, indicating the reaction's potential industrial application.

Keywords Na₂ eosin Y photoexcited, Visible light mediated, Photochemical synthesis, Pyranopyrimidines

Introduction

EY is a readily available non-metallic organic dye which has recently found widespread use due to its economic and ecological advantages over transition photocatalysts based on metal. At photoredox reactions mediated by eosin Y, the successfully oxidized/reduced intended substrates is often reliant on whether the substrates' prospective oxidability or reducibility is within the range of eosin Y (Fig. 1) [1–4].

*Correspondence: Farzaneh Mohamadpour mohamadpour.f.7@gmail.com School of Engineering, Apadana Institute of Higher Education, Shiraz, Iran The spectrum of photochemical processes induced by eosin Y has been constrained by the aforementioned electrochemical requirements. In contrast to other natural dyes, eosin Y has singular phenol and xanthene moieties as well as strong acidic characteristics, resulting in four different formulations. There is substantial proof that anionic variants EY show photoactivity in the bulk of other photoreaction investigations, whereas the neutral forms are thought to be inactive and useless in potentially relevant synthesis methods [5, 6]. Wang et al. [7] and Wu et al. [8] were recently inspired by the characteristics of eosin Y to lead the discovery of novel photoinduced eosin Y activation states. The researchers discovered that induced modes generated from neutral eosin Y may act as direct HAT catalysts and photoacids for stimulating



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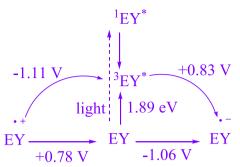


Fig. 1 Eosin Y's oxidative and reductive quenching cycles, as well as their associated potentials [1]

native Carbon Hydrogen bonds and glycals, respectively [1] (Fig. 2).

Hydrogen atom transfer (HAT) is a fundamental mechanism that may be involved in a variety of chemical, ecological, and biological systems. Direct HAT catalysis, assisted by quinone and benzophenone [9–11], was recently presented as a method to start C–H bonding in the presence of light.

Additionally, visible light radiation [12, 13] is a dependable method for green chemistry due to abundant energy resources, cheap price, and energy form in the synthesis of environmentally friendly organomolecules.

Pyranopyrimidines have been described with a variety of pharmacological properties as antihypertensive [14], cardiotonic [15], bronchiodilator [16], antibronchitic [17] and antitumor activities [18].

Numerous strategies are available [19–37]. Numerous instances occurred from these treatments. However, certain synthetic rules include limitations on the use of metal catalysts, severe reaction conditions, costly reagents, repetitive workup, low yield, prolonged reaction time, and environmental hazard.

Due to the aforementioned challenges and our concern for ecologically benign procedures, most scientists have been intrigued by the quest for easy, efficient, and environmentally safe methods that may enhance organic reactions under green conditions [38–40]. Considering

the above concerns and our desire to build pyrano[2,3-d]pyrimidine scaffolds production, it is critical to investigate environmentally safe catalysts under green conditions for the correct synthesizing of the nitrogen heterocyclic complexes. This research establishes a novel function for the utilization of a non-metallic in aforementioned photochemical synthesizing process. There is proof that photoinduced states generated from Na $_2$ eosin Y acts as a catalyst [41] for photochemical synthesizing through direct hydrogen atom transfer. This cyclocondensation at aqueous ethanol and room temperature and in an air environment is facilitated by visible light. This is a successful one-pot reaction carried out under very efficient, moderate, and simple conditions.

Experimental

General

All substances have their physical properties measured utilizing an Electro thermal 9100 equipment. Furthermore, the spectra were acquired utilizing nuclear magnetic resonance on a Bruker equipment (DRX-400 and DRX-300) with the solvent DMSO-d₆.

Under white LED (18 W) irradiation, a combination of aryl aldehyde derivatives (1, 1.0 mmol), malononitrile (2, 1.0 mmol), and barbituric acid/1,3-dimethylbarbituric acid (3, 1.0 mmol) in an H₂O/EtOH (2:1) (3 mL), was added Na₂ eosin Y (1 mol %) and it was stirred, at room temperature. TLC was used to monitor the reaction's progression, using *n*-hexane/ethyl acetate (3:1) as the eluent. After the reaction occurs, the obtained material was screened and washed with water, and the crude solid was crystallized again from ethanol to get the pure substance with no further purifying. We wanted to see if we could scale up to the level that pharmaceutical process R&D wants, even if we were able to synthesize the above molecules using gram-scale techniques. 50 mmol of 2-methoxybenzaldehyde, malononitrile, and barbituric acid were combined in an experiment under standard conditions. The large-scale reaction went well and concluded in 12 min, with the product collected using standard

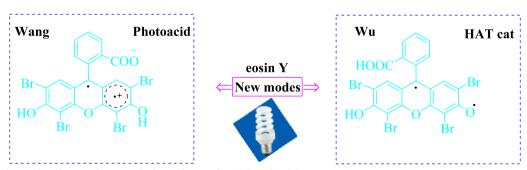


Fig. 2 The photoinduced eosin Y being studied as a photoacid or HAT catalyst [1]

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filtration. The ¹HNMR spectrum of this substance suggests that it is spectroscopically pure. After comparing spectroscopic data (¹HNMR), the products were categorized. Spectral files some of the known products are offered Supporting Information file.

Results and discussion

To begin, Table 1 summarizes the findings of an investigation into the reactivity of benzaldehyde, malononitrile, barbituric acid, EtOH/H2O (1:2) enhanced via irradiation at ambient temperature. With no photocatalyst, a 53% quantity of 4a was detected at room temperature for 25 min in EtOH/ H_2O (1:2). The process was facilitated by investigating a range of organophotocatalysts as rose bengal, erythrosin B, Na2 eosin Y, 9H-xanthen-9-one, rhodamine B, fluorescein, riboflavin and phenanthrenequinone (Fig. 3) under comparable conditions. The development of this phenomenon and the formation of the matching product 4a were seen satisfactorily in yields ranging from 42 to 94% (Table 1). As per our results, Na2 eosin Y performed better than other photocatalysts in this process. By adding 1 mol% Na₂ eosin Y, the yield was improved to 94% (Table 1, entry 4). Additionally, a poor product yield was observed in CH₂Cl₂, CH₃CN, CHCl₃, DMSO, DMF, toluene and THF (Table 2). By performing the reaction in $\rm H_2O$, EtOH, EtOAc, $\rm H_2O$ /EtOH, solvent-free, MeOH, were increased the rate and yield of the reaction. A huge improvement was observed in $\rm H_2O$ /EtOH (Table 2). The reaction went extremely well in $\rm H_2O$ /EtOH (2:1), yielding 94% under similar circumstances (Table 2, entry 3). The yield was tested using a variety of illumination, showing that it increased somewhat in response to white LED. The finding demonstrates the critical nature of $\rm Na_2$ eosin Y and visible light for the product to develop effectively. Additionally, optimum conditions were found by changing the white LED irradiation intensities. Greatest results were obtained when white 18W LED irradiation was used. As shown in Fig. 4 and Table 3 this method is applicable to a variety of substrates.

Figure 5 illustrates the outcomes of the manage tests performed to decide the mechanism underlying this 3-component visible light-driven response. Within the first stage of the Knoevenagel-Michael cyclocondensation procedure, arylidenemalononitrile (I), which is produced, is condensed with (II). Under the following conditions (Na $_2$ eosin Y in H $_2$ O/EtOH (2:1) and using white LED), malononitrile (2) and benzaldehyde (1) have been condensed to create arylidenemalononitrile (I) along with water removal. Then, the following reactions among the radical (II) and arylidenemalononitrile

Table 1 Photocatalyst optimization table

Entry	Photocatalyst	Solvent (3 mL)	Time (min)	Isolated Yields (%)
1	-	H ₂ O/EtOH (2:1)	25	53
2	Na ₂ eosin Y (0.2 mol%)	H ₂ O/EtOH (2:1)	10	71
3	Na ₂ eosin Y (0.5 mol%)	H ₂ O/EtOH (2:1)	10	85
4	Na ₂ eosin Y (1 mol%)	H ₂ O/EtOH (2:1)	10	94
5	Na ₂ eosin Y (1.5 mol%)	H ₂ O/EtOH (2:1)	10	94
6	Erythrosin B (1 mol%)	H ₂ O/EtOH (2:1)	10	42
7	Rose bengal (1 mol%)	H ₂ O/EtOH (2:1)	10	63
8	9H-Xanthen-9-one (1 mol%)	H ₂ O/EtOH (2:1)	10	52
9	Rhodamine B (1 mol%)	H ₂ O/EtOH (2:1)	10	75
10	Fluorescein (1 mol%)	H ₂ O/EtOH (2:1)	10	71
11	Riboflavin (1 mol%)	H ₂ O/EtOH (2:1)	10	68
12	Phenanthrenequinone (1 mol%)	H ₂ O/EtOH (2:1)	10	45

 $Reaction\ condition:\ malononitrile,\ benzaldehyde,\ barbituric\ acid,\ H_2O/EtOH,\ White\ LED\ (18\ W)\ with\ a\ variety\ of\ photocatalysts\ at\ room\ temperature$

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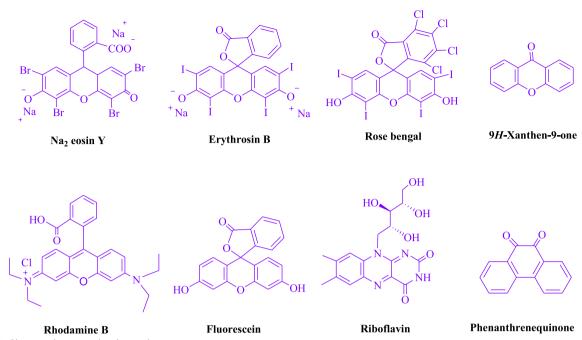


Fig. 3 Photocatalysts tested in this study

(I) led to the preferred product **4a** (94%). There was trace product **4a** created, even if the reaction was performed in general darkness. The effects of this experiment suggest that Fig. 6 offers a logical chemical pathway.

Figure 6 shows the suggested mechanism for synthesizing pyrano[2,3-d]pyrimidine scaffolds. With the use of visible light, malononitrile (2) is subjected tautomerization to give (A). Afterwards, (A) and aldehyde derivatives (1) react to form arylidenemalononitrile (B), undergoing an activation photochemically for the formation of a radical intermediate (C), in which visible light can partially affect with exerting extra energy to accelerate the reaction. As reported in previous studies [1, 4, 8], eosin Y-originated photoexcited modes can function as direct hydrogen atom transfer (HAT) catalysts for activating C-H bonds. The malononitrile radical is formed by the promotion of visible light triggered Na2 eosin Y* via a HAT procedure. Ground-state Na2 eosin Y and the intermediate (D) are regenerated by occurring reverse hydrogen atom transfer (RHAT) process between eosin Na₂ Y-H and radical adduct C. Then, malononitrile radical extracts a hydrogen atom from (E) to produce intermediate (F). Subsequently, intermediate (D) and (F) coalesce to generate (G) as Michael acceptor, additionally undergoing tautomerization and intramolecular cyclization for the product formation (4).

Table 4 compares the catalytic capability of a variety of catalysts mentioned in this article. It may find

a variety of uses, including the utilization of a little quantity of photocatalyst, a fast reaction time, and the absence of by-products when visible light irradiation is used. The atom-economic protocol is very successful at multigram scales and has significant industrial implications. These materials excel in terms of both efficiency and pureness. Table 4 also includes data on turnover number (TON) and turnover frequency (TOF). The higher the TON and TOF numerical values, the less catalyst is used and the greater the yield, and as the value rises, the catalyst becomes more effective.

Conclusion

In conclusion, the photoinduced states of Na_2 eosin Y-derived act as a direct hydrogen atom transfer (HAT) catalyst was used for photochemically synthesizing pyrano[2,3-d]pyrimidine scaffolds through the three-condensation domino response of aryl aldehydes, malononitrile and barbituric acid/1,3-dimethylbarbituric acid in aqueous ethanol via visible light-mediated at room temperature. This study provides a green methodology for photochemically synthesizing with the least catalyst, producing good results, speeding. up the process, and achieving a high atom economy utilizing a non-metallic organic dye available commercially and at a low cost, Na_2 eosin Y. This is a successful one-pot reaction that was carried out in a very efficient and straightforward manner.

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 Table 2
 Solvent and visible-light optimization table

Entry	Light Source	Solvent (3 mL)	Time (min)	Isolated Yields (%)
1	White light (18 W)	H ₂ O/EtOH (1:1)	10	83
2	White light (18 W)	H ₂ O/EtOH (1:2)	10	79
3	White light (18 W)	H ₂ O/EtOH (2:1)	10	94
4	White light (18 W)	EtOH	10	61
5	White light (18 W)	H ₂ O	10	74
6	White light (18 W)	MeOH	15	56
7	White light (18 W)	CH ₂ Cl ₂	30	26
8	White light (18 W)	CH ₃ CN	15	50
9	White light (18 W)	_	20	72
10	White light (18 W)	CHCl ₃	30	21
11	White light (18 W)	EtOAc	10	62
12	White light (18 W)	DMSO	25	47
13	White light (18 W)	DMF	40	25
14	White light (18 W)	Toluene	25	43
15	White light (18 W)	THF	40	28
16	White light (10 W)	H ₂ O/EtOH (2:1)	10	75
17	White light (12 W)	H ₂ O/EtOH (2:1)	10	82
18	White light (20 W)	H ₂ O/EtOH (2:1)	10	94
19	_	H ₂ O/EtOH (2:1)	25	< 5
20	Green light (18 W)	H ₂ O/EtOH (2:1)	10	83
21	Blue light (18 W)	H ₂ O/EtOH (2:1)	10	80

Reaction condition: malononitrile, benzaldehyde, barbituric acid, 1 mol% Na₂ eosin Y

Fig. 4 Synthesis of pyranopyrimidines

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Table 3 As a photocatalyst, photoexcited Na₂ eosin Y was used for the synthesis of pyranopyrimidines

Lit. 235-237 °C [24]

Lit. 158-160 °C [20]

Lit. 241-242 °C [20]

Lit. 211-212 °C [31]

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Table 3 (continued)

Table 3 (continued)					
H CN CN NH2	O NH ₂	Me O N O NH ₂	Me O N O NH ₂		
4m (5 min, 92%)	4n (5 min, 95%)	4o (7 min, 93%)	4p (10 min, 94%)		
Mp. 257–259 °C	Mp. 228–230 °C	Mp. 225–227 °C	Mp. 204–206 °C		
Lit. 256–260 °C [19]	Lit. 227–228 °C [31]	Lit. 226 °C [24]	Lit. 205–207 °C [24]		
O OH CN CN NH2	H N CN CN NH ₂	Br CN NH ₂	O NH ₂		
4q (12 min, 89%)	4r (8 min, 95%)	4s (15 min, 84%)	4t (15 min, 86%)		
Mp. 167–169 °C	Mp. 261–263 °C	Mp. 241–243 °C	Mp. 211–213 °C		
Lit. 169–170 °C [21]	Lit. 259–261 °C [22]	Lit. 240–245 °C [21]	Lit. 210–211 °C [25]		
O CI CN CN NH ₂	O CI CN CN NH2	OMe O NH2 H N O NH2			
4u (9 min, 87%)	4v (12 min, 91%)	4w (15 min, 92%)			
Mp. 212–214 ℃	Mp. 241–243 °C	Mp. 271–273 °C			
Lit. 211–214 °C [24]	Lit. 243–244 °C [31]	Lit. 272–274 °C [22]			

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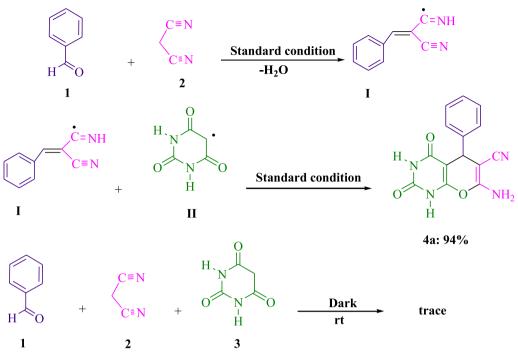


Fig. 5 The reactions of benzaldehyde (1, 1 mmol), malononitrile (2, 1 mmol), barbituric acid (3, 1 mmol), and are crucial control tests for comprehending the reactions' mechanism

Table 4 Comparative analysis of the catalytic properties of many of the catalysts mentioned in the text for producing 4a

Entry	Catalyst	Conditions	Time/Yield (%)	TON	TOF	References
1	[DABCO](SO ₃ H) ₂ (CI) ₂	H ₂ O, Reflux	10 min/86	17.2	1.7	[19]
2	$[DABCO](SO_3H)_2(HSO_2)_2$	H ₂ O, 90 °C	7 min/90	45	6.4	[19]
3	B(OH) ₃	THF/H ₂ O, Reflux	125 min/81	8.1	0.06	[24]
4	DBA	EtOH/H ₂ O, Reflux	58 min/94	4.7	0.08	[27]
5	Zn[(L)proline] ₂	EtOH, Reflux	60 min/85	5	0.08	[35]
6	theophylline	H ₂ O/EtOH, 50 °C	10 min/86	8.6	0.8	[36]
7	β-CD	H ₂ O, 80 °C	10 min/93	9.3	0.9	[37]
8	Na ₂ eosin Y	Visible light irradiation, $\rm H_2O/EtOH$ (2:1), rt	10 min/94	94	9.4	This study

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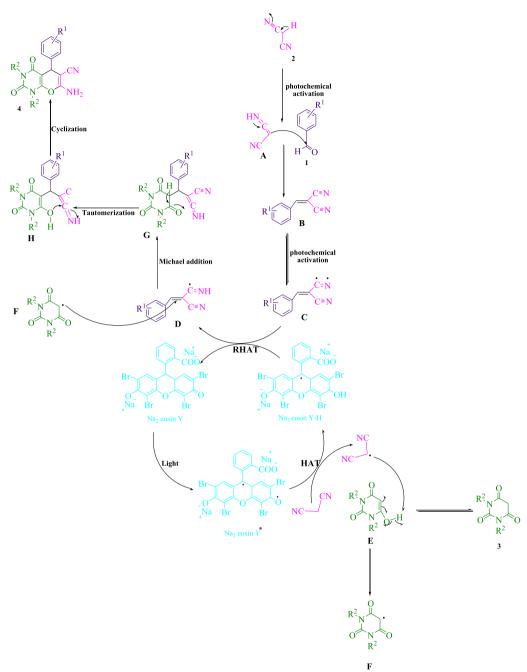


Fig. 6 Recommended mechanistic path

Supplementary Information

The online version contains supplementary material available at https://doi.org/10.1186/s13065-023-00912-7.

Additional file 1: Fig. S1. ¹HNMR spectrum of compound (300 MHz, DMSO-d₆) of **4d. Fig. S2.** ¹HNMR spectrum of compound (300 MHz, DMSO-d6) of **4e. Fig. S3.** ¹HNMR spectrum of compound (300 MHz, DMSO-d6) of **4m. Fig. S4.** ¹HNMR spectrum of compound (300 MHz, DMSO-d6) of **4v**.

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Author contributions

FM conceived and designed the experiments. FM conducted the experiments and interpreted the results. FM participated in analyzing the data and writing the paper. FM read and approved the final manuscript.

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Availability of data and materials

All data generated or analyzed during this study are included in this published. article.

Declarations

Ethics approval and consent to participate

Not applicable.

Consent for publication

Not applicable.

Competing interests

There is no conflict of interest to declare.

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