

# A Peerless Aproach: Organophotoredox/Cu(I) Catalyzed, Regioselective, Visible Light Facilitated, Click Synthesis of 1,2,3-Triazoles via Azide–Alkyne [3+2] Cycloaddition

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Received: 17 May 2017 / Accepted: 30 July 2017 © Springer Science+Business Media, LLC 2017

**Abstract** A visible light initiated, mild, one pot, multicomponent copper catalyzed azide alkyne [3+2] cycloaddition in the presence of organo photoredox catalyst Eosin Y using EtOH:H<sub>2</sub>O as reaction medium for the synthesis of substituted 1,2,3-triazoles is reported. A facile regioselective ring opening of epoxides followed by 1, 3 dipolar cycloaddition

with alkynes under CFL (Compact fluorescent light) irradiation as a source of visible light resulting in the formation of C–N bonds, is the characteristic feature of the present strategy. Scope of the present protocol is further extended by using benzyl chloride in place of epoxide.



**Electronic supplementary material** The online version of this article (doi:10.1007/s10562-017-2156-8) contains supplementary material, which is available to authorized users.

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Published online: 20 August 2017

**Keywords** Visible light  $\cdot$  [3+2] cycloaddition  $\cdot$  Eosin Y  $\cdot$  Regioselective  $\cdot$  1,2,3-Triazole

## **1** Introduction

Recently, the focus of the chemists is shifting towards synthetic routes employing green aspects [1-5]. Taking into consideration the increasing concern for the environment, the chemists are making a constant effort to adopt synthetic routes that discourage the involvement of hazardous chemicals. In this recent past, light has been utilized as one of the most efficient and prominent resources to achieve synthetically useful organic transformations [6–10]. A bulk of chemists are experimenting with visible light (generally CFLs and LEDs) because of its unique properties like easy availability, easy handling and serving as an eco-efficient, cost effective, environmentally benign and unending source of energy for the construction of organic molecules [11, 12]. It is able to transfer the activation energy to the reactants in chemical reactions. Thus visible light promoted catalysis has originated as one of the most sustainable tools in organic synthesis being accessible to a myriad of reactions [13–17]. However, some organic molecules are unable to absorb visible light [18]. To triumph over this barricade, chemists use photo sensitizers. In literature various methods have been revealed in which transition metals [19-22], nanoparticles [23-25] and certain organic dyes [26-29] are utilized as photocatalysts. Howbeit trasition metals like Ru(II) and Ir(II) complexes exhibit a number of impediments as they proclaim adverse inherent malignancy, high toxicity, low sustainability, short durability and expensiveness [30, 31]. Within this context, an organic dye, eosin Y (EY), is a fascinating alternative to transition metal photocatalysts and has lately been extensively used as a photoredox catalyst. It undergoes redox quenching to generate radical anion or radical cation and this process has been done via photoexcitation of eosin Y with simultaneous reductive or oxidative quenching through acceptance or donation of an electron [30].

Over the past few years, nitrogen heterocycles have gained substantial attention [32] (https://cen.acs.org/content/dam/ cen/supplements/CENsupplement092014.pdf, http://www. fiercepharma.com/special-report/top-20-generic-moleculesworldwide). In September 2014, C and EN Supplement presented top fifty drugs, most of them enclosed at least one nitrogen-containing heterocycle (https://cen.acs.org/content/dam/cen/supplements/CENsupplement092014.pdf). It is present in a range of natural products, biologically active structures and medicinally important compounds. 1,2,3-triazoles, five-membered nitrogen containing heterocycles, reveal diverse biological activities such as being anti-allergic [33], anti-HIV [34, 35], anti-microbial [36], anti-bacterial [37], anticancerous [38], antifungal [39], antimalarial [40], anti-inflammatory [41], antitubercular [42],  $\beta_3$ -adrenergic receptor agonist [43] and having antioxidant activity [44]. They also find industrial applications in optical brighteners, corrosion decelerating agents, dyes, agrochemicals, fungicides, herbicides and solar cells [45–48] (Fig. 1) [49–53].

Multicomponent reactions (MCRs) are highly costeffective, environmentally benign and absolutely appropriate for combinatorial library synthesis and have thus gained colossal advantages over conventional multi-step reaction sequences managing to diminish waste, time and cost successfully [54–58]. Huisgen 1,3-dipolar cycloaddition is one of the prominent reaction for the synthesis of 1,2,3-triazoles via copper-catalyzed azide-alkyne cycloaddition (CuAAC). Various methods for the synthesis of 1,2,3-triazoles in different reaction conditions have been developed [59-74]. Although the reported methods have several merits, however they were associated with some drawbacks like use of costly and bulky catalysts, non-reusability of catalyst, use of base, difficulty in the recovery of high boiling solvent, high temperature and use of excess amounts of reagents or catalysts which entails to design a more facile and eco-efficient protocol for the synthesis of 1,2,3-triazoles. In order to extend our work on visible light induced synthesis [75–78], here we have documented a visible light promoted Eosin Y and copper (I) iodide catalyzed [3+2] cycloaddition for the synthesis of 1,2,3-triazoles initiating from epoxides, sodium azide and terminal alkynes. The scope of the present protocol has been further increased by using benzyl chloride in place of epoxides (Scheme 1).

## 2 Results and Discussion

For the attainment of optimization, we took styrene oxide (1a), sodium azide (2) and phenyl acetylene (3a) as model substrates for the synthesis of 1,2,3-triazoles. During our initial attempts, we optimized the reaction conditions by using varied catalysts, photosensitizers and different sources as well as intensities of visible light (Table 1). Initially, we took styrene oxide (1a, 1.0 mmol), sodium azide (2, 1.0 mmol) and phenyl acetylene (3a, 1.0 mmol) under CFL (23 W) irradiation in the absence of catalyst at room temperature and found that product did not form after 24 h of CFL irradiation (Table 1, entry 1). After that, we performed our test reaction in the presence of catalysts. In the first instance, we used copper(II) chloride as a catalyst and afforded 41% yield of product within 8 h (Table 1, entry 2). Similarly when we used copper(I) oxide, it resulted in the product in 6 h with 50% yield (Table 1, entry 3). However when we used copper(I) iodide as a catalyst for the test reaction, surprisingly an improved result was obtained with 60% yield of the product in 5 h (Table 1, entry 4). On the basis of above investigations, we concluded that catalyst is mandatory for the reaction to proceed and copper(I) salts are suitable for the present transformation as it forms copper(I) phenylacetylide, which is the essential light absorbing species and increases the efficiency of reaction in presence of visible light [79-82]. Although the reactions proceeded well in the presence of copper(I) iodide, the yield and reaction time have not been much satisfactory. In order to further increase the effectiveness of the model reaction, we decided to make use of various types of photocatalysts. Within this context, we used a range of photocatalysts (Table 1, entries 5-9) and found that Eosin Y encouraged the reaction and furnished



Fig. 1 Some biologically important derivatives of 1,2,3-triazole



Scheme 1 Synthesis of 1,2,3-triazole

#### Table 1 Optimization of reaction conditions



Entry	Catalyst	Time (h)	Yield <sup>a</sup> (%)
1	None <sup>b</sup>	24.0	_c
2	CuCl <sub>2</sub> (20 mol%)	8.0	41
3	Cu <sub>2</sub> O (20 mol%)	6.0	50
4	CuI (20 mol%)	5.0	60
5	CuI (20 mol%) + Iodine (0.1 mol%)	4.5	71
6	CuI (20 mol%) + CAN <sup>d</sup> (0.1 mol%)	4.5	72
7	CuI (20 mol%) + RB <sup>e</sup> (0.1 mol%)	4.0	78
8	CuI (20 mol%) + $EB^{f}$ (0.1 mol%)	3.5	76
9	CuI (20 mol%) + Eosin Y (0.1 mol%)	3.5	82
10	CuI (10 mol%) + Eosin Y (0.1 mol%)	3.5	72
11	CuI (15 mol%)+Eosin Y (0.1 mol%)	3.5	75
12	CuI (30 mol%) + Eosin Y (0.1 mol%)	3.5	82
13	CuI (20 mol%) + Eosin Y (0.05 mol%)	3.5	78
14	CuI (20 mol%) + Eosin Y (0.2 mol%)	3.5	82
15	CuI + Eosin Y <sup>g</sup>	12.0	25
16	CuI + Eosin Y (18 W CFL)	3.5	75
17	CuI + Eosin Y (20 W CFL)	3.5	77
18	CuI + Eosin Y (23 W CFL)	3.5	82
19	CuI + Eosin Y (27 W CFL)	3.5	82
20	CuI + Eosin Y + TEMPO <sup>h</sup>	12.0	Trace

Reaction condition: 1a (1.0 mmol), 2 (1.0 mmol), 3a (1.0 mmol) in EtOH: H<sub>2</sub>O irradiated using CFL under open air at room temperature

<sup>a</sup>Yield of the product (%)

<sup>b</sup>Absence of catalysts

<sup>c</sup>Not detected

<sup>d</sup>Cerric Ammonium Nitrate

<sup>e</sup>Rose Bengal

<sup>f</sup>Eosin Blue

<sup>g</sup>Dark

<sup>h</sup>(2,2,6,6-Tetramethyl-piperidin-1-yl)oxyl

the products with 82% yields in 3.5 h (Table 1, entry 9). In the next endeavour, we focused towards the optimization of best catalyst loading for the present protocol (Table 1, entries 9–14) which led us to infer that the reaction works well with 20 mol% of Copper(I) iodide and 0.1 mol% of Eosin Y (Table 1, entry 9). After this we performed our model reaction with different intensities of light source (Table 1, entries 16–19) and observed that CFL (23W) is the ideal source of visible light for the present protocol (Table 1, entry 18). Moreover, the reaction was suppressed with TEMPO (2.0 mmol), clearly indicating that the reaction proceeds through a free radical pathway (Table 1, entry 20) [83–85].

 Table 2 Optimization of solvents for the synthesis of compound 4a



Entry	Solvents	Time (h)	Yield <sup>a</sup> (%)
1	DMF	24.0	Trace
2	THF	6.0	45
3	DMSO	10.0	21
4	DCM	10.0	26
5	DCE	24.0	Trace
6	CH <sub>3</sub> CN	10.0	49
7	MeOH	7.0	70
8	H <sub>2</sub> O	4.0	75
9	EtOH	4.5	71
10	EtOH: H <sub>2</sub> O (1: 1)	3.5	82
11	EtOH: H <sub>2</sub> O (2: 1)	3.5	78
12	EtOH: $H_2O(3:1)$	3.5	78
13	Neat	24.0	_b

Reaction condition: **1a** (1.0 mmol), **2** (1.0 mmol), **3a** (1.0 mmol) in solvent irradiated using CFL (23W) under open air at room temperature. <sup>a</sup>Yield of the product (%)

<sup>b</sup>Not detected.

After the finalization of the best catalytic system, we next screened different solvents to further optimize the reaction conditions (Table 2). Initially, we used DMF as a solvent and found that after 24 h of CFL irradiation the product was formed in traces (Table 2, entry 1). After that a batch of reactions with polar aprotic solvents (Table 2, entries 2-6) were performed but still neither yield nor time of reaction was satisfactory for the desired transformation. Further we used polar protic solvents (Table 2, entries 7-12) and discovered that the present strategy is effective with them. Among them EtOH:H<sub>2</sub>O (1:1) combination of solvent was observed as most appropriate for the required transformation (Table 2, entry 10). EtOH:H<sub>2</sub>O (1:1) solvent system might be effective because of greater solubility of reactants in this medium or due to an increase in the number of collisions between reactants which resulted in enhancement of their ground state energy and reaction rates [86-90]. A hypothesis is also associated with it; the transition states of present reaction could be stabilized by water as it has high static permittivity [91].

We also carried out the test reaction in neat condition but no result was obtained even 24 h of CFL irradiation (Table 2, entry 13). So the EtOH: $H_2O$  (1:1) mixed solvent system has been found to be most effective, convenient and efficient for the required transformation.

Once we had the optimal reaction conditions in hand, we next sought to extend this methodology with scope and limitations of the present synthetic strategy. In this regard, we used different derivatives of epoxide (1) as well as alkynes (3) (Table 3). It was observed that aryl substituted epoxides afforded the reactions more effectively in comparison to alkyl substituted epoxides. It was interestingly observed that in the case of aryl substituted epoxides a preferential attack on the more hindered carbon atom took place (Table 3, entries 1, 2, 3, 4 and 6) [59, 60, 63]. In contrast, alkyl substituted epoxides involved attack at less hindered carbon atom (Table 3, entries 8, 10, 11 and 12). Similar less hindered attack was found with glycidyl phenyl ether and isopropyl glycidyl ether 
 Table 3
 Substrate scope for the synthesis of compound 4



Entry	Epoxide (1)	Alkyne (3)	Product (4)	Yield <sup>a</sup> (%)	Time (h)	M. P. (°C)
7		Ja 3a	$ \begin{array}{c} OH \\ V \\ 4g \\ \end{array} $	71	5.0	62–64 °C [59]
8	le	3a		78	4.0	168–170 °C [63]
9	Cl If	Ja 3a		70	5.0	101–103 °C [67]
10	Jg Jg	Ja 3a		76	4.0	109–110 °C [59]
11	O Ih	Ja 3a		70	7.5	93–94 °C [67]
12	li 1i	3a		69	7.5	105–107 °C [63]

Table 3 (continued)

Reaction condition: 1 (1.0 mmol), 2 (1.0 mmol), 3 (1.0 mmol) in EtOH:  $H_2O$  irradiated using CFL (23W) under open air at room temperature <sup>a</sup>Yield of the product (%)

(Table 3, entries 5 and 7) [60]. As in epoxides, alkynes with aryl substitution react faster and more efficiently than those with alkyl substitution. Adopting the same

experimental route, we further extended the scope of this methodology for the synthesis of 1,2,3-triazole by replacing epoxide by Benzyl chloride (5). It resulted in 
 Table 4
 Substrate scope for the synthesis of compound 6





Reaction condition: **5** (1.0 mmol), **2** (1.0 mmol), **3** (1.0 mmol) in EtOH:  $H_2O$  irradiated using CFL (23W) under open air at room temperature <sup>a</sup>Yield of the product (%)

the formation of a new series of derivatives of 1,2,3-triazole (6) (Table 4, entries 1-10).

#### 2.1 Mechanism

A proposed mechanistic route, the cornerstone of literature survey [21, 79-82, 84, 92-100], for 1,2,3-triazoles has been postulated (Schemes 2, 3). On photo-absorption Eosin Y, a photoredox catalyst gets excited to its singlet state  $({}^{1}EY^{*})$  and is then converted to a more stable triplet state (<sup>3</sup>EY\*) via inter system crossing (ISC). After this it undergoes a single electron transfer (SET) and converts epoxide (1) into an arene radical cation (A) which further gets converted into its stable benzyl cation (A') form [92]. Next azide attacks as a nucleophile, at the same time Eosin Y undergoes a single electron transfer (SET) and (B) results into (C). After that (C) takes a proton from the solvent and converts into (D) which resulted in its active 1, 3 dipolar form (E). Simultaneously, phenyl acetylene reacts with copper (I) iodide to form a light absorbing species, copper (I) phenylacetylide (F). In last step (E) and (F) undergo the well known [3+2] cycloaddition which consequently leads to the formation of the desired scaffold, 1,2,3-triazole.

## 3 Conclusion

In conclusion, we have divulged the successful, visiblelight-promoted synthetic approach to accomplish a slick synthesis of 1,2,3-triazole, a biologically and pharmaceutically significant scaffold, via multicomponent [3+2]CuAAC using epoxide (1), sodium azide (2) and terminal alkyne (3) as reactants in presence of eosin Y, a photosensitizer and copper iodide. The present synthetic strategy has been pertained to the quick synthesis of 1,2,3-triazole using commercially available substrates, without employing harsh reaction conditions. The expansion of this methodology has been achieved by the use of a range of epoxides as well as phenylacetylene and benzyl chloride derivatives. The use of ubiquitous visible light makes the adopted procedure immensely green.



Scheme 2 Proposed mechanism for the synthesis of 1,2,3-triazole (4a)





Acknowledgements We sincerely thank SAIF, Punjab University, Chandigarh, for providing micro-analyses and spectra. The author A. Mishra and P. Rai are thankful to UGC, New Delhi for the award of Junior Research Fellowship (JRF) and Senior Research Fellowship (SRF), respectively.

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