



Photocatalytic One-Pot Synthesis of Quinazolinone Under Ambient Conditions

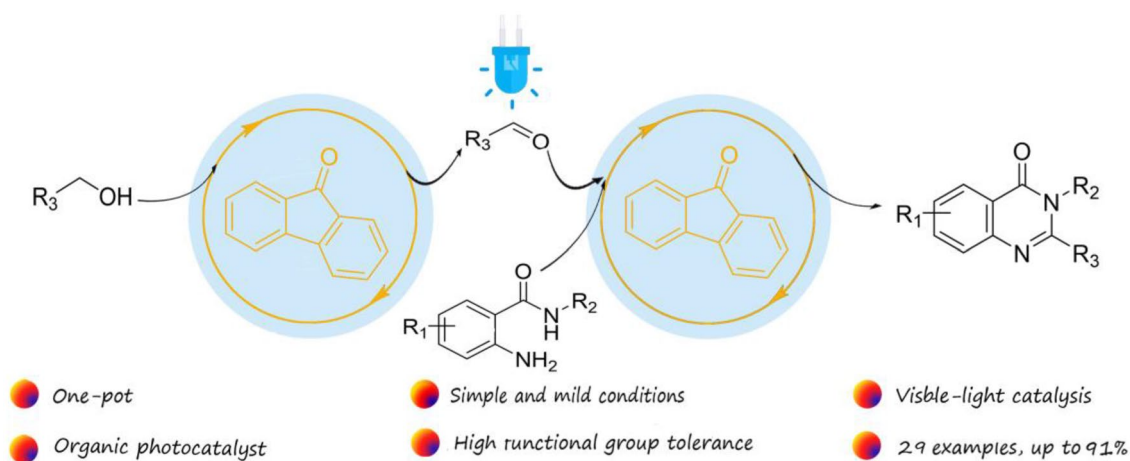
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Abstract

A novel photocatalytic cyclization reaction was developed for the synthesis of quinazolinones from *o*-aminobenzamides and *in-situ* generated aldehydes from alcohols using 9-fluorenone as the photocatalyst through a "one-pot" process. Furthermore, alcohols are perfect alternatives to aldehydes due to some unique advantages, such as being green, less toxic, available, and economical. The present protocol showed good tolerance for various substrates and could afford a range of quinazolinones (29 examples) up to 91% under ambient conditions.

Graphical Abstract



Keywords Visible-light-catalyzed · Photochemical catalysis · One-Pot · Cyclization · Quinazolinones

1 Introduction

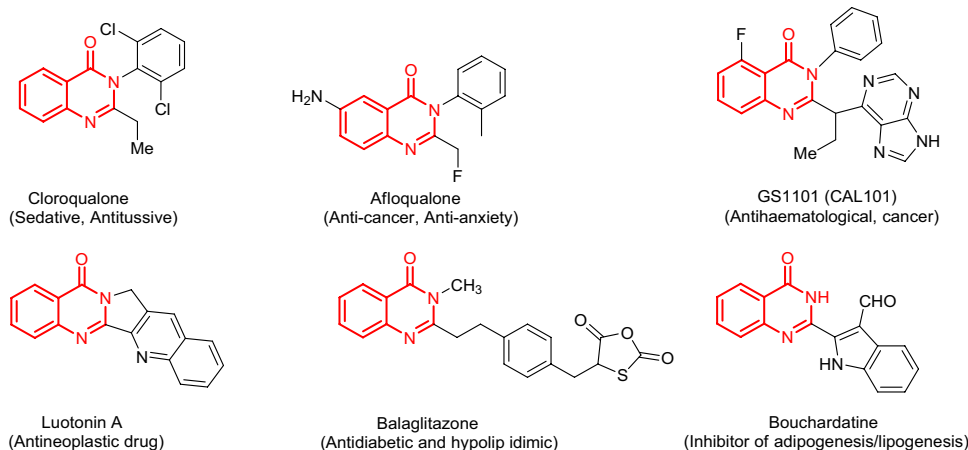
N-containing heteroaromatic compounds are important structures found in natural products, pharmaceutically important molecules, and organic functional materials [1–3]. Among them, quinazolinone and its derivatives have received considerable attention in recent years, because they exhibit significant pharmacological and biological activities, such as antibacterial, antineoplastic, anti-inflammatory, anti-convulsant, antimalarial, anti-asthmatic, anti-Alzheimer, and anticancer (Scheme 1) [4–7]. In addition, quinazolinones play an important role in organic reactions due to their

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Scheme 1 Examples of synthetic and natural quinazolinones

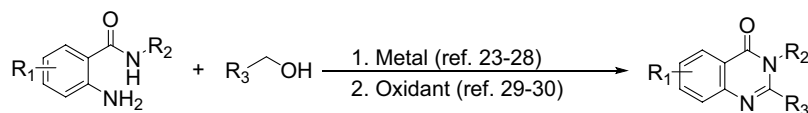


excellent characteristics. Hence, the excellent characteristics of quinazolinone derivatives have promoted extensive studies of their synthesis, and a number of synthetic methods have been developed over the past few decades [8–12]. Despite these significant advances, the most classical and general approaches still utilized *o*-aminobenzamides and aldehydes as the substrates to form quinazolinones followed by the oxidation of the resulting amination intermediates in the laboratory and industry [13–16]. Most of these processes generally require the use of stoichiometric of non-renewable oxidants (KMnO_4 [17], DDQ [18], and CuCl_2 [19]) and metal catalysts [20–22]. However, aldehydes were required as the reagents to generate quinazolinones with good yields in the above reactions, which were significantly toxic and sensitive to unavoidably occurring self-aldol side reactions. Therefore, the development of greener and more harmless material instead of aldehydes for the synthesis of

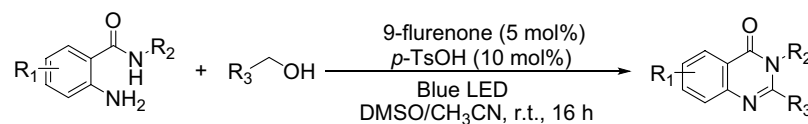
quinazolinones is highly desirable. As a class of compounds, alcohols are perfect alternatives to aldehydes because they are greener, more available, more economical, more stable, and less toxic than aldehydes, which are smoothly oxidized to aldehydes followed by the condensation with *o*-aminobenzamides forming quinazolinones. In recent years, various metal catalysts or oxidants (such as Ir [23], Pd [24], Ru [25], Mn [26], Fe [27], Cu [28], DMSO [29], TBHP [30]) have been used to prepare quinazolinones (Scheme 2). Although these protocols have certain disadvantages, these condensations require the use of metal catalysts or stoichiometric quantities of toxic oxidants. Therefore, it is imperative to develop a more practical, green, and efficient approach to constructing quinazolinones.

Recently, visible-light catalysis has attracted widespread research interest in light degradation [31, 32] and organic synthesis owing to the inherent green, mild, and character of

(a) Previous works

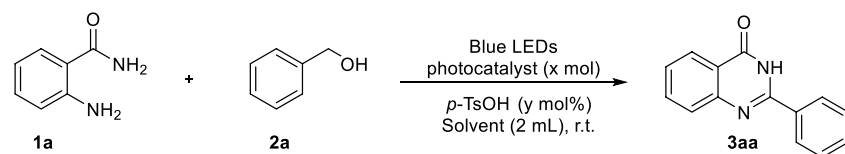


(b) This work



- One-pot
- Visible-light catalysis
- Organic photocatalyst
- Simple and mild condition
- High functional group tolerance
- 29 examples, up to 91% yield

Scheme 2 Reported methods for the synthesis of quinazolinones

Table 1 Condition optimization for the synthesis of quinazolinones

Entry	Photocatalyst (x mol%)	<i>p</i> -TsOH (y mol%)	Solvent	Time (h)	3aa (%) ^a
1	Eosin Y (2)	10	CH ₃ CN	10	34
2	Fluorescein (2)	10	CH ₃ CN	10	35
3	Pyrenedione (2)	10	CH ₃ CN	10	31
4	9-fluorenone (2)	10	CH ₃ CN	10	43
5	9-fluorenone (2)	0	CH ₃ CN	10	22
6	9-fluorenone (2)	10	PhMe	10	Trace
7	9-fluorenone (2)	10	THF	10	4
8	9-fluorenone (2)	10	DMF	10	7
9	9-fluorenone (2)	10	CH ₃ CN + DMSO ^b	10	52
10	9-fluorenone (5)	10	CH ₃ CN + DMSO ^b	10	78
11	9-fluorenone (5)	10	CH₃CN + DMSO^b	16	91
12	9-fluorenone (5)	10	CH ₃ CN + DMSO ^b	20	92

Reaction conditions: **1a** (0.2 mmol), **2a** (0.24 mmol), photocatalyst (x mol%) and *p*-TsOH (y mol%) in solvent (2.0 mL) with 10 W blue LEDs at room temperature for 16 h

^aIsolated yields

^bCH₃CN/DMSO (v/v)=9: 1

light [33–47]. Many kinds of organic transformations have already been accomplished with good to excellent yields under ambient conditions by using transition-metal catalysts, such as Aza-Henry reaction, oxidative addition, and cross-coupling reaction [48–51]. However, compared to transition metal catalysts, organic dyes, and small organic molecules, as photosensitizers, have recently aroused growing interest due to their high efficiency of visible-light absorption, enhanced stability, and easy modification for visible light catalysis [52–57]. Advantageously, these organic molecules show unique reactivity and unparalleled selectivity in organic reactions, and the structures of these molecules can be easily optimized for obtaining the desired products. Especially, 9-fluorenone, as a commercially available and cheap metal-free photocatalyst, can activate O₂ molecule to transform it into reactive oxygen species (ROS) such as superoxide anion radical, hydrogen peroxide, singlet oxygen and hydroxy radical which are the key oxidants in many organic reactions [58–60].

Based on all this information and our own interest to explore metal-free catalysis, we develop an efficient, highly atom economical, and environmentally friendly one-pot

strategy for constructing quinazolinones from alcohols with *o*-aminobenzamides in good yields using 9-fluorenone as the photocatalyst by irradiation of visible light under ambient conditions.

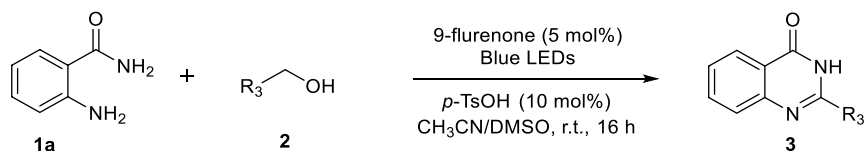
2 Experiment

2.1 Materials

All reagent-grade chemicals were obtained from commercial suppliers and were used as received unless otherwise noted (Table S1, Support Information). DMSO (anhydrous, ≥ 99.9%) and CH₃CN (anhydrous, ≥ 99.9%) were purchased from Sigma Aldrich.

2.2 General Procedure for the Synthesis of Quinazolinones Using Alcohol

O-aminobenzamides (0.2 mmol), alcohols (0.24 mmol), 9-fluorenone (0.01 mmol, 5 mol %), *p*-TsOH (0.02 mmol, 10 mol %), CH₃CN (1.8 mL), and DMSO (0.2 mL) were

Table 2 Scope of the alcohols

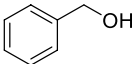
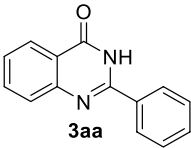
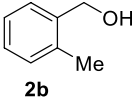
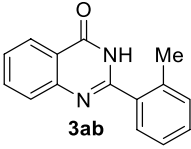
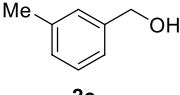
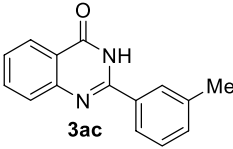
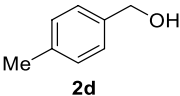
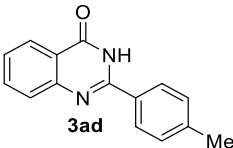
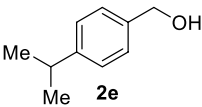
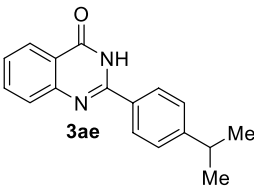
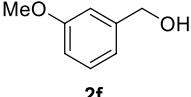
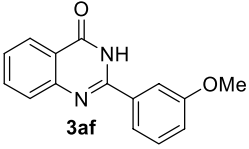
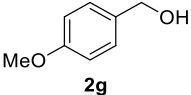
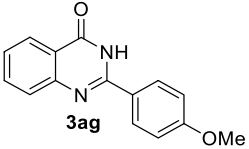
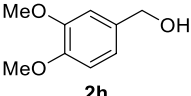
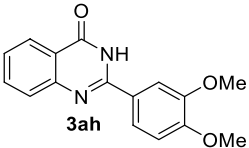
Entry	Substrate	Product	Yield (%) ^a	M.P. [References]
1	 2a	 3aa	91	237–238 °C [67]
2	 2b	 3ab	87	222–224 °C [67]
3	 2c	 3ac	89	210–212 °C [29]
4	 2d	 3ad	89	244–245 °C [67]
5	 2e	 3ae	88	211–212 °C [67]
6	 2f	 3af	87	245–247 °C [67]
7	 2g	 3ag	88	245–247 °C [68]
8	 2h	 3ah	87	241–243 °C [69]

Table 2 (continued)

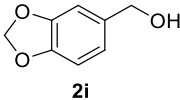
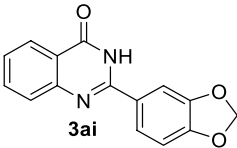
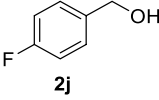
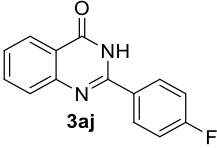
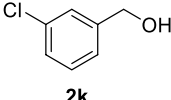
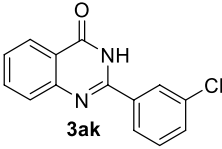
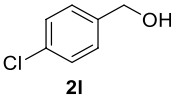
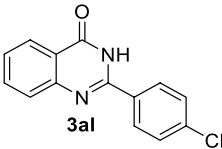
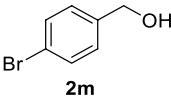
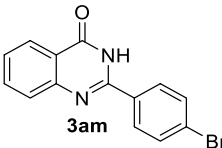
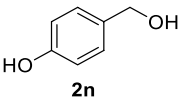
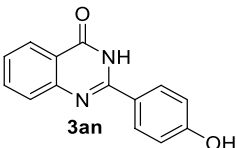
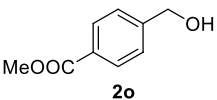
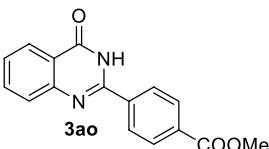
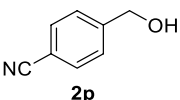
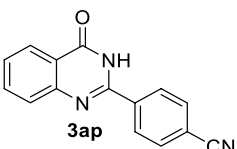
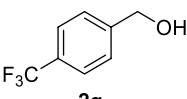
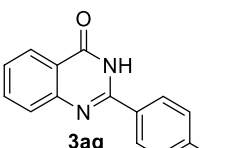
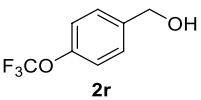
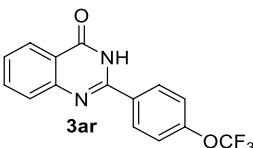
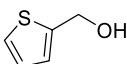
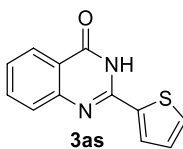
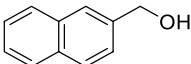
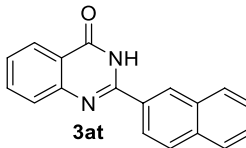
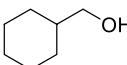
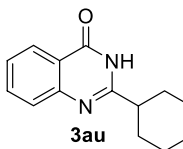
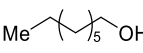
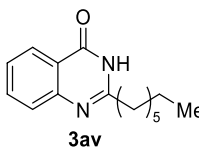
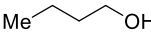
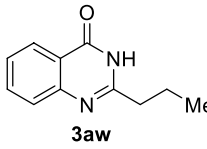
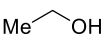
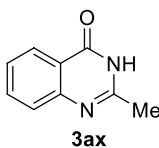
Entry	Substrate	Product	Yield (%) ^a	M.P. [References]
9	 2i	 3ai	87	276–279 °C [70]
10	 2j	 3aj	85	258–259 °C [29]
11	 2k	 3ak	86	295–296 °C [67]
12	 2l	 3al	89	300–301 °C [67]
13	 2m	 3am	82	296–297 °C [71]
14	 2n	 3an	89	> 300 °C [72]
15	 2o	 3ao	85	275–277 °C [73]
16	 2p	 3ap	88	281–282 °C [74]
17	 2q	 3aq	84	308–310 °C [67]
18	 2r	 3ar	85	269–270 °C [75]

Table 2 (continued)

Entry	Substrate	Product	Yield (%) ^a	M.P. [References]
19	 2s	 3as	80	272–273 °C [76]
20	 2t	 3at	87	212–214 °C [67]
21 ^b	 2u	 3au	45	229–231 °C [77]
22 ^b	 2v	 3av	32	140–142 °C [78]
23 ^b	 2w	 3aw	42	200–201 °C [30]
24 ^b	 2x	 3ax	N.D	

Reaction conditions: **1a** (0.2 mmol), **2** (0.24 mmol), 9-fluorenone (5 mol%) and *p*-TsOH (10 mol%) in the mixed solvent of CH₃CN (1.8 mL) and DMSO (0.2 mL) with 10 W blue LEDs at room temperature for 16 h

^aIsolated yield, N.D. = not detected

^bReaction for 24 h

added to a 10 mL flat quartz glass jar and placed in a photocatalytic parallel reactor. The container was placed under the 10 W blue LEDs lamp at room temperature for 16 h. After completion of the reaction, 100 mL of distilled water is added to the mixture. Then, the mixture was extracted with ethyl acetate (50 mL×3), dried over anhydrous sodium sulfate, filtered, and the solvent was rotary evaporated to obtain a crude product. The produce was obtained by column chromatography on silica gel and was identified by NMR analyses. All analytical data of the known compounds are consistent with those reported in the literatures.

2.3 Gram-Scale Synthesis of **3aa**

O-aminobenzamides (8 mmol), benzyl alcohol (9.6 mmol), 9-fluorenone (5 mol%), *p*-TsOH (10 mol%), CH₃CN (9 mL), and DMSO (1 mL) were added into a 25 mL flat quartz glass jar with a stirrer under 10 W blue LEDs at room temperature for 16 h. After completion, 500 mL of distilled water was added to the mixture. The mixture was extracted with ethyl acetate (100 mL×3), dried over anhydrous sodium sulfate, and filtered. The mixture was concentrated in vacuo and

purified by flash column chromatography with hexanes/ethyl acetate to afford the product **3aa**.

2.4 Product Analysis

Melting points of all products were measured on an RY-1 micro melt apparatus. Proton nuclear magnetic resonance (^1H NMR) spectra were recorded using a 400 spectrometer at 400 MHz. Chemical shifts in ^1H NMR spectra are reported in parts per million (ppm) on the scale from an internal standard of DMSO- d_6 (2.50 ppm). Coupling constant J values are reported in hertz (Hz), and the corresponding representation of splitting mode is as follows: s, singlet; d, doublet; t, triplet; m, multiplet; b, broad. Carbon-13 nuclear magnetic resonance (^{13}C NMR) spectra were recorded at 100 MHz using a 400 spectrometer. Chemical shifts are reported in delta (δ) units and the ppm from the center of the peak of DMSO- d_6 (39.520 ppm). ^{13}C NMR spectra were routinely run with broadband decoupling.

3 Results and Discussions

3.1 Optimization of Reaction Conditions

To optimize the reaction conditions, we initially chose *o*-aminobenzamide (**1a**) to react with benzyl alcohol (**2a**) as a model reaction. To achieve a green reaction outcome, we chose a series of organic dyes (eosin Y, fluorescein, perylene, and 9-fluorenone as the photocatalysts (2 mol%) to give the target product under 10 W blue LEDs in the air within 10 h under ambient conditions. During these preliminary tests, 9-fluorenone showed reasonable activity in the preparation of quinazolinone (**3aa**) in the presence of 10 mol% *p*-TsOH (Table 1, entries 1–4). It was found that the efficiency of the reaction was reduced markedly in the absence of *p*-TsOH (Table 1, entry 5). Next, we examined different solvents. The use of other nonpolar and polar aprotic solvents such as toluene, THF, and DMF resulted in the formation of the desired product in $\leq 7\%$ yields (Table 1, entries 6–8). According to previous research, the lifetime of the excited state of 9-fluorenone could be increased with an additional stabilizing effect of DMSO [61]. To our delight, a further improved yield (52%) was achieved in the mixture of CH_3CN and DMSO (Table 1, entry 9). In addition, the

results showed that the catalyst dosage and extending reaction time were advantageous to improve the yield of **3aa** (Table 1, entries 10–12). In addition, the effect of the dosage of 9-fluorenone on the yield of the target product **3aa** and the methods of improving photocatalytic activity were further studied [31, 32, 62–66]. In summary, the reaction works best using 5 mol% 9-fluorenone and 10 mol% *p*-TsOH in the mixed solvent of CH_3CN and DMSO for 16 h under air and blue LEDs at room temperature (Table 1, entry 11).

3.2 Synthesis of Quinazolinone.

After optimization of the reaction conditions and finding the best photocatalyst, we become interested in exploring the scope of the reactions and the results are listed in Table 2. Benzyl alcohols bearing one or two electron-donating groups, such as methyl (**2b**, **2c**, and **2d**), isopropyl (**2e**), methoxy (**2f** and **2g**), dimethoxy (**2h**), and methyl-enedioxy (**2i**) were converted to the corresponding products (**3ab–3ai**) in 87–89% yields. In the case of halide-substituted benzyl alcohol, the corresponding desired products (**3aj–3am**) were obtained in good to excellent yields. No cleavage of halogen atoms was observed when halide-substituted alcohols were utilized. In addition, 4-hydroxybenzyl alcohol (**2n**) and methyl 4-(hydroxymethyl) benzoate (**2o**) were also suitable substrates, which gave the corresponding products **3an** and **3ao** in 89% and 85% yields, respectively. In this case, benzyl alcohols with a strong electron-withdrawing group, such as cyano (**2p**), trifluoromethyl (**2q**), and trifluoromethoxy (**2r**) were used as substrates, the desired products **3ap–3ar** could be obtained in 84–88% yields. Also, heteroatom-containing and fused-ring primary alcohols (**2s** and **2t**) reacted with **1a** to provide the corresponding products (**3as** and **3at**) in 80% and 87% yields. Finally, aliphatic alcohols such as cyclohexanemethanol (**2u**), 1-octanol (**2v**), and 1-butanol (**2w**) were tested successively, and the desired products **3au–3aw** were obtained in 32–45% yields. Unfortunately, when ethanol (**2x**) was chosen as the substrate, the target product was not obtained.

Encouraged by the above results, we then extended this method to different *o*-aminobenzamides (**1**). As can be seen in Table 3, the electronic properties of the substituents on the phenyl group of *o*-aminobenzamides did not

Scheme 3 Gram-scale synthesis of **3aa**

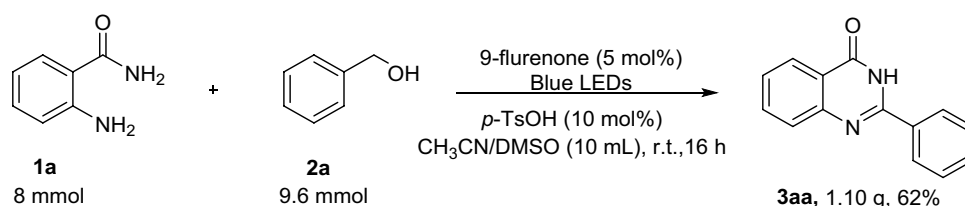
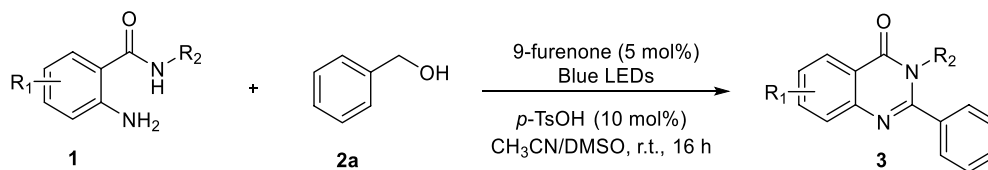


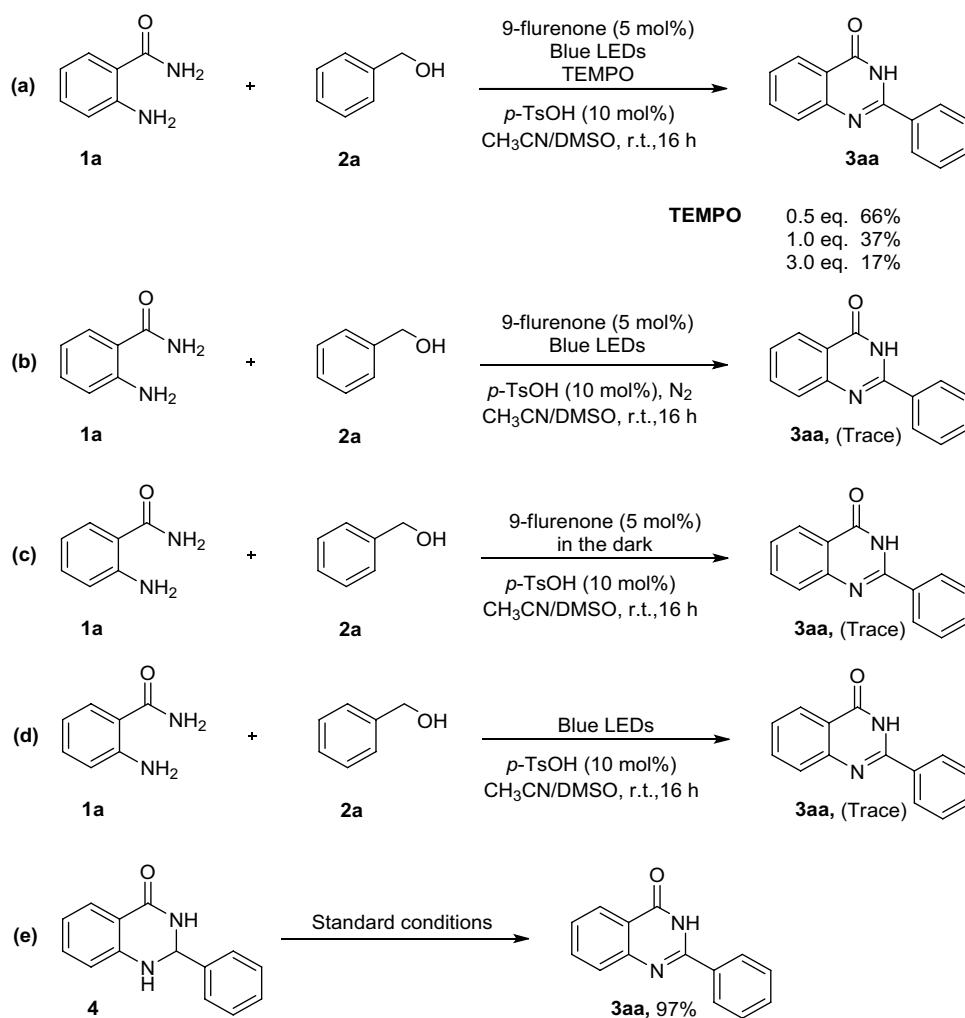
Table 3 Scope of the *o*-aminobenzamides

Entry	Substrate	Product	Yield (%) ^a	M.P [References]
1			84	238–238 °C [25]
2			82	278–279 °C [25]
3			85	279–287 °C [67]
4			81	285–286 °C [79]
5			85	112–114 °C [80]
6 ^b			78	158–159 °C [81]

Reaction conditions: **1** (0.2 mmol), **2a** (0.24 mmol), 9-fluorenone (5 mol%), *p*-TsOH (10 mol%) in the mixed solvent of CH₃CN (1.8 mL) and DMSO (0.2 mL) with 10 W blue LEDs at room temperature for 16 h

^aIsolated yield

^b**1g** (0.1 mmol), **2a** (0.12 mmol), reaction for 24 h

Scheme 4 Controlled experiments

have a significant impact on the reaction outcome, and the desired products (**3ba–3ea**) could be obtained in 81–85% yields. To our delight, the *N*-substituted *o*-aminobenzamides **1f** and **1g** could also react with **2a** to satisfactorily generate the desired products **3fa** and **3ga** in 85% and 78% yields, respectively.

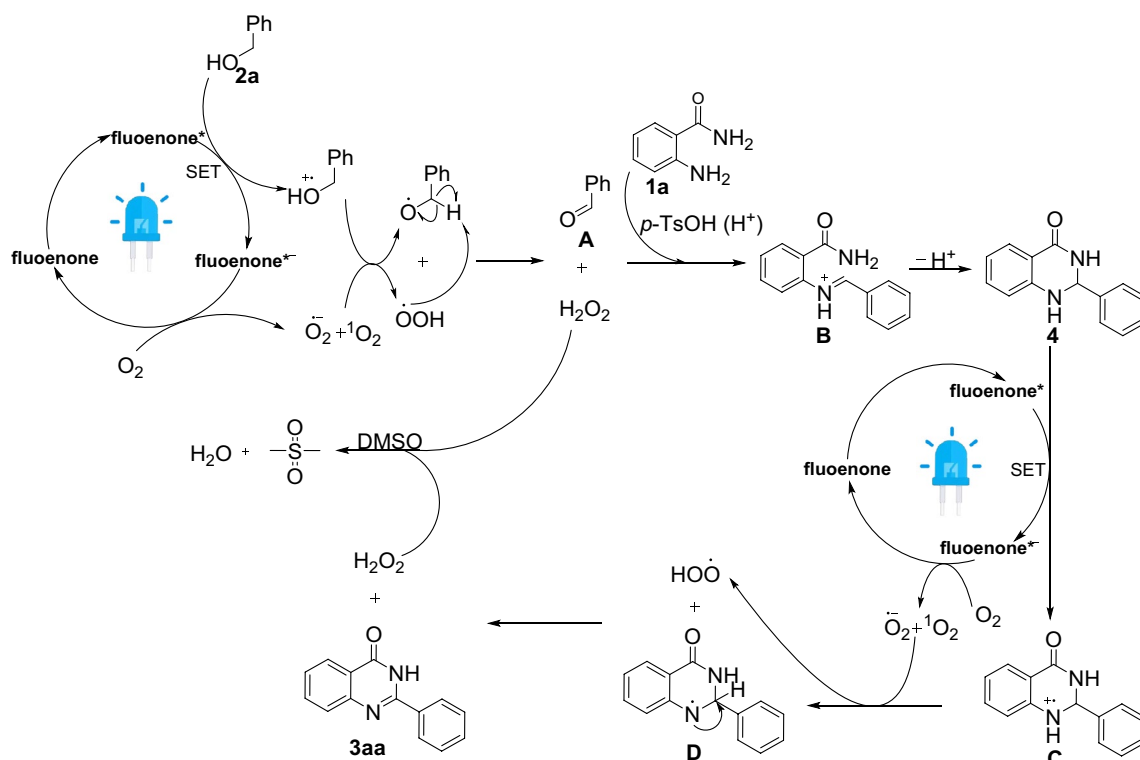
Further, to test the utility of this photochemical strategy, an 8 mmol scale-up reaction was conducted under optimized reaction conditions (Scheme 3). To our delight, the reaction of *o*-aminobenzamide (**1a**) and benzyl alcohol (**2a**) was also performed well under blue LEDs, giving the product **3aa** in 62% yield (1.10 g). Therefore, the catalytic system works well for the synthesis of quinazolinones on a gram scale.

3.3 Controlled Experiment

Through the UV–visible diffuse reflectance spectra (UV–vis DRS), the absorption profiles of the 9-fluorenone have wide absorption in the visible light region [82, 83] (Fig. S3, Supporting Information). Next, the recyclability of the

optimized catalyst (9-fluorenone) for the model reaction was investigated (Fig. S4, Support Information). After each recycling, the catalyst was recovered by column chromatography, and applied to the next cycle. The activity of the catalyst decreased a little during the five cycles.

To shed light on the mechanism of this visible-light-induced reaction for the preparation of quinazolinones from primary alcohols and *o*-aminobenzamides, several control experiments were conducted (Scheme 4). When various amounts of 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO) as radical trapping agent was added to the photocatalytic reaction under an identical reaction conditions and the yield of (**3aa**) significantly decreased upon increasing the amount of TEMPO, implying that the radical pathway may be involved (Scheme 4a). The reaction of *o*-aminobenzamide **1a** and benzyl alcohol **2a**, in the absence of light or O_2 , was performed under optimal conditions, with no product (**3aa**) was observed, suggesting the essentiality of light and O_2 in such a transformation (Scheme 4b and c). Equally unsurprisingly, when **1a** was treated with **2a** without photocatalyst, nearly no product was observed for the standard



Scheme 5 The possible reaction mechanism

reaction (Scheme 4d). In addition, the reaction of intermediate **4** could proceed smoothly to furnish the desired product **3aa** in 97% yield under the optimized reaction conditions, which strongly demonstrated the formation of intermediate **4** in the reaction. Moreover, we carried out a set of electron paramagnetic resonance (EPR) experiments. The ESR experiments verified that both superoxide radical anion and singlet oxygen radical were included in the reaction (Fig. S6, Support Information). These experimental results clearly revealed that the route was involved in the reaction.

3.4 Reaction Mechanism

On the basis of the experimental results and literature references [84, 85], a possible mechanism for the photocatalytic reaction of the condensation of *o*-aminobenzamide (**1a**) with benzyl alcohol (**2a**) to quinazolinone (**3aa**) is suggested in Scheme 5. Initially, 9-fluorenone* was produced by 9-fluorenone under visible light conditions. Benzyl alcohol was converted to activated benzyl alcohol with the action of 9-fluorenone* via single electron transfer (SET) producing 9-fluorenone*⁻. Accompanied by the oxidation of 9-fluorenone*⁻ under O₂ in the air, 9-fluorenone was generated by releasing singlet oxygen radical and superoxide radical anion. Next, the activated benzyl alcohol reacted with the superoxide radical anion to generate peroxide radical and

further abstraction of one more hydrogen atom by the peroxide radical generated benzaldehyde (**A**) and H₂O₂. Then, H₂O₂ was reacted with DMSO to generate dimethyl sulfone and H₂O. Intermediate **4** was formed by the condensation between *o*-aminobenzamide (**1a**) and benzaldehyde (**A**) through a stepwise acid-promoted cyclization in the presence of *p*-TsOH. Intermediate **C** was generated through single electron transfer (SET) from the reaction of 9-fluorenone* and **4**. The superoxide radical anion from O₂ was reacted with **C** to produce intermediate **D** and the peroxide radical. Finally, the peroxide radical abstracted one hydrogen atom from **D** to give the target product (**3aa**) by producing H₂O₂, which was transformed to dimethyl sulfone in the presence of DMSO.

4 Conclusions

In conclusion, we have developed a novel and efficient method for the synthesis of quinazolinones from *o*-aminobenzamides and primary alcohols using 9-fluorenone as the cheap and high active photocatalyst under irradiation of visible light. Based on this approach, various multi-substituted quinazolinones were easily synthesized in good yields under mild reaction conditions. Moreover, it could achieve the gram-scale transformation in a satisfactory yield, which

might indicate that this strategy has more applications in the future. Further study on the synthesis of other heterocyclic structures by photoredox catalysis is currently underway in our laboratory.

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