



Nd@C₃N₄-photoredox/chlorine dual catalyzed synthesis and evaluation of antitumor activities of 4-alkylated sulfonyl ketimines

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ABSTRACT

The first example of Nd@C₃N₄-photoredox/chlorine dual catalyzed alkylation with unactivated alkanes as the alkyl sources has been developed, which allows for the synthesis of various 4-alkylated cyclic sulfonyl ketimines. In this process, chlorine functions as both a redox and hydrogen atom transfer catalyst. The synergism of the reversible Nd²⁺/Nd³⁺ and Cl[·]/Cl⁻ redox pairs significantly enhances overall photocatalytic efficiency. The *in vitro* anticancer activity of 4-alkylated products was evaluated by using the CCK8 assay against both human choroidal melanoma (MUM-2B) and lung cancer (A549) cell. Compound **3da** showed approximately triple the potency of 5-fluorouracil.

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Semiconductor-based photoredox organic synthesis which uses light energy to facilitate chemical conversions in organic reactions, has received extensive attention from synthetic chemists due to its easy recyclability and excellent reusability [1,2]. Carbon nitride (C₃N₄) has garnered significant research interest as a cost-effective and readily accessible semiconductor photocatalyst for its high chemical stability and favorable photoelectrical properties [3,4]. Therefore, many efforts have been devoted to addressing its intrinsic limitations [5–12]. The introduction of the low-cost and abundant rare earth element neodymium (Nd) into C₃N₄ is considered as promising approach, which enhances the efficiency of photoinduced electron-hole pair separation and interfacial charge transfer due to the reversible shifting between Nd³⁺ and Nd²⁺ oxidation states, thereby boosting the overall photocatalytic performance [13,14]. Recently, Nd doped C₃N₄ (Nd@C₃N₄) has been applied in the photocatalytic degradation of organic dyes [15]. To the best of our knowledge, there have been no reports on the application of Nd@C₃N₄ in photoredox organic synthesis. Very recently, several intrinsic semiconductors have been applied in dual photoredox/redox catalysis for the efficient construction of high-value chemicals. However, no example of dual catalysis has been re-

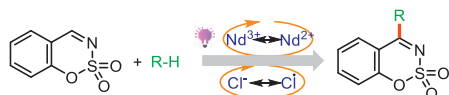
ported using high-performance extrinsic semiconductor photocatalyst.

Cyclic *N*-sulfonyl ketimines are highly significant structural subunits that are present in a large number of naturally occurring compounds, bioactive molecules and synthetic pharmaceuticals [16–18]. Furthermore, they are also versatile synthons and building blocks toward a wide variety of high-value compounds [19–24]. Consequently, their synthesis and functionalization have engrossed lots of interest from synthetic and pharmaceutical chemists. Among these cyclic sulfonyl ketimines derivatives, 4-alkylated cyclic sulfonyl ketimines are of particular interest, as they have shown valuable biological and pharmaceutical activities. Therefore, several synthetic protocols have been developed for such molecules [25–28]. However, these processes generally require stoichiometric chemical oxidants and large excess of alkylation reagents, which limit their applicability in large-scale synthesis.

The efficient and sustainable catalytic transformation of saturated C–H bonds of low-cost abundant unactivated alkanes into high value-added alkylated compounds has been a challenging task for synthetic chemists [29–31]. The photocatalytic hydrogen atom transfer (HAT) is an eco-friendly and energy-efficient strategy to achieve alkylation with unactivated alkane as the alkyl radical source [32,33]. Chlorine radical is a powerful HAT reagent, which can abstract hydrogen atoms from unactivated alkane to yield the

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Scheme 1. Nd@C₃N₄/TBACl dual catalysis.Table 1
Optimization of reaction conditions.^a

Entry	Variation from the standard reaction conditions	Yield (%) ^b
1	None	87
2	g-C ₃ N ₄ or Nd(NO ₃) ₃ ·H ₂ O was used	trace
3	Mixture of g-C ₃ N ₄ and Nd(NO ₃) ₃ ·H ₂ O was used	N.R.
4	2%, 4% or 8% Nd@C ₃ N ₄ was used	46, 68, 77
5	6% La@C ₃ N ₄ or 6% Yb@C ₃ N ₄ was used	35, 43
6	6% Fe@C ₃ N ₄ or 6% Co@C ₃ N ₄ was used	43
7	6% Cu@C ₃ N ₄ or 6% Ag@C ₃ N ₄ was used	trace
8	Without Nd@C ₃ N ₄ or visible light	N.R.
9	α-Fe ₂ O ₃ , WO ₃ or CdSe was used	trace, trace, trace
10	Ir(ppy) ₃ or 4CzIPN was used	trace, trace
11	Without TBACl	33
12	Acetone, THF, DMF or DMSO was used	18, trace, trace,
13	Blue LED or Green LED was used	38, trace
14	5 W LED instead of 10 W LED	48
15	Air instead of N ₂	N.R.

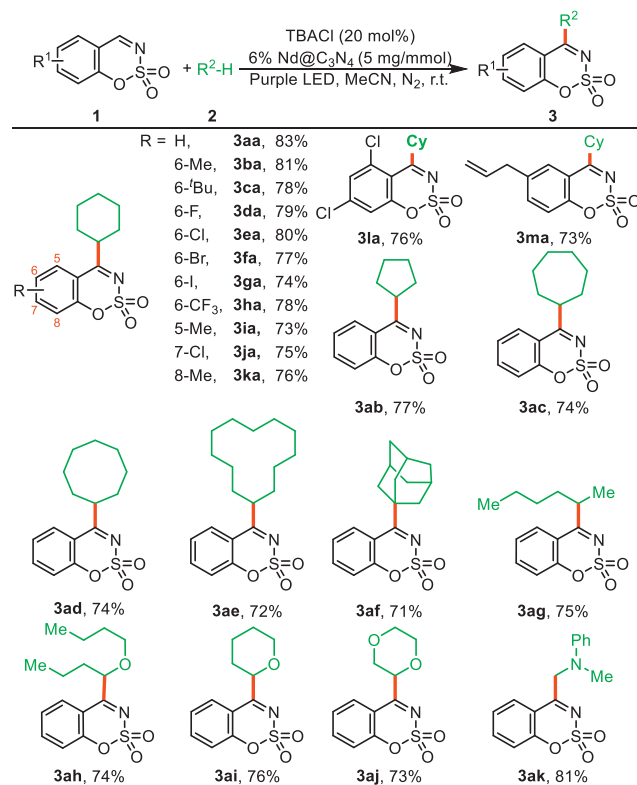
^a Conditions: **1a** (0.2 mmol), **2a** (10 equiv.), 6% Nd@C₃N₄ (10 mg), TBACl (20 mol%), MeCN (2 mL), purple LED (10 W), N₂, r.t., 24 h

^b Yield estimated by GC with dodecane as the internal reference.

corresponding carbon-centered radical. Chloride anion (Cl⁻) is considered as an ideal source for the generation of Cl[·] via single electron oxidation because it is innocuous and abundant in diverse salt forms. However, the single electron oxidation of Cl⁻ into Cl[·] is generally challenging due to the high redox potential ($E_{ox} = +2.03$ V vs. SCE). Recently, the photoinduced ligand-to-metal charge transfers (LMCT) [34,35] has emerged as a powerful protocol for the production of Cl[·] by the photolysis of transition metal chlorides [36–44]. However, a major drawback of these types of reactions is the inevitable residual transition metal in the final products, which greatly restricts their application in pharmaceutical synthesis. As a result, alternative photocatalytic HAT strategies that efficiently generate Cl[·] under transition metal-free and sustainable conditions should be highly desirable.

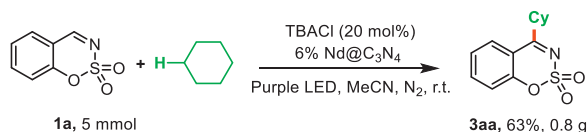
As part of our continuing studies in the area of green organic synthesis [45–51], we herein report the first Nd@C₃N₄-photoredox/chlorine dual catalyzed alkylation reaction with unactivated alkanes as the alkyl sources, by which various 4-alkylated cyclic sulfonyl ketimines were efficiently synthesized from cyclic *N*-sulfonyl ketimines and under visible light conditions (Scheme 1). In this reaction, Cl played a dual role also as a redox catalyst and a HAT catalyst. To the best of our knowledge, this is the first example of combining the reversible Nd³⁺/Nd²⁺ redox pair and the reversible Cl⁻/Cl[·] redox pair to achieve a huge improvement in the photocatalytic efficiency.

Initially, we investigated the reaction conditions employing *N*-sulfonyl ketimine (**1a**) and cyclohexane (**2a**) as the model substrates (Table 1). After systematic investigation of the reaction parameters, an 87% GC yield of the alkylated product **3aa** was achieved in the presence of 6% Nd@C₃N₄ as the heterogeneous photocatalyst and TBACl as the homogeneous HAT catalyst in MeCN with the irradiation of 10 W purple LED under nitrogen atmosphere at ambient temperature for 24 h (entry 1). The single g-C₃N₄, single Nd(NO₃)₃·H₂O and the physical mixture of g-C₃N₄ and Nd(NO₃)₃·H₂O either gave **3aa** in a trace yield or showed no

Scheme 2. Substrate scope. **1** (0.2 mmol), **2** (10 equiv.), 6% Nd@C₃N₄ (10 mg), TBACl (20 mol%), MeCN (2 mL), purple LED (10 W), N₂, r.t., 24 h; Isolated yields.

reactivity in this reaction (entries 2 and 3). These results indicated that Nd-doping has an improved photoelectrochemical property and significant enhancement in photoactivity. Changing the amount of incorporated Nd diminished the reaction yield, suggesting the photocatalytic activity of the Nd-doped g-C₃N₄ was highly dependent on the amount of incorporated Nd (entry 4). Performing the reaction with rare earth element-doped g-C₃N₄ gave a low yield of **3aa** (entry 5). With iron or copper family element-doped g-C₃N₄ as the photocatalyst, only a trace amount of **3aa** was detected (entries 6 and 7). Without a photocatalyst or visible light, the template reaction did not occur, suggesting that both photocatalyst and visible were necessary for this alkylation reaction (entry 8). Either no generation or trace amount of **3aa** was observed with intrinsic semiconductor photocatalyst (α-Fe₂O₃, WO₃ and CdSe, entry 9) and homogeneous photocatalyst Ir(ppy)₃ and 4CzIPN, entry 10). Only 33% yield of **3aa** was obtained in the absence of TBACl, highlighting the critical role of the HAT catalyst in promoting the homolytic alkyl C–H bond cleavage (entry 11). A series of solvents, including acetone, THF, DMF and DMSO, were also investigated, and these results showed that they were less effective than MeCN (entry 12). Carrying out this reaction with other LED sources, including blue LED and green LED, afforded **3aa** in 38% and trace yield, respectively (entry 13). The yield of target products decreased to 48% when the power of LEDs light was reduced to 5 W (entry 14). No reaction occurred under air atmosphere, suggesting that molecular oxygen suppressed this reaction (entry 15).

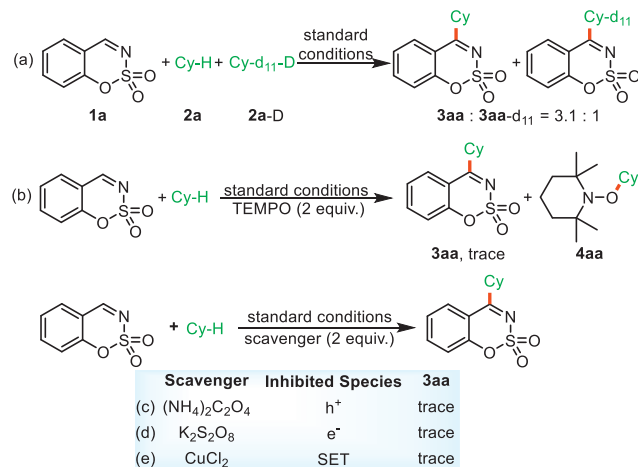
With the optimum reaction conditions in hand, we next sought to evaluate the scope of the reaction using a series of *N*-sulfonyl ketimines (**1**) and alkanes (**2**) (Scheme 2). No matter whether the phenyl ring of *N*-sulfonyl ketimine (**1**) was substituted with either an electron-donating, electron-withdrawing or sterically hindered group, all of them afforded the desired alkylated products in good yields (**3aa**, **3ca**–**3ka**). A series of useful scaffolds for late

Scheme 3. Large-scale synthesis of **3aa**.

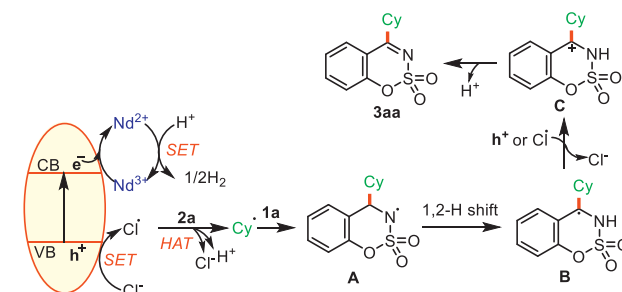
derivatization including methyl, *tert*-butyl, fluorine, chloro, bromo, iodine, and trifluoromethyl groups can be preserved. The existence of substituents—methyl or chloro at 5, 6, 7, and 8 positions of substrates **1** (**3ba**, **3ia–3ka**) did not significantly affect the C-4 alkylation outcome, generating desired products in high yields. Substrates **1** bearing the di-substituents at 5,7-position of the benzene ring was efficiently transformed into the corresponding products in a 76% yield (**3la**). Notably, alkene could be retained in this photocatalyzed reaction (**3ma**). Afterwards, the scope of this transformation with respect to other alkanes were also investigated. A series of cycloalkanes such as cyclopentane, cycloheptane, cyclooctane, cyclooctanes all proceeded well, yielding the products (**3ab–3ae**) in excellent yields. Additionally, bulky cycloalkanes adamantane (**3af**), as well as linear alkanes and ethers (**3ag** and **3ah**) were well tolerated in this semi-heterogeneous transformation. Furthermore, cyclic ethers such as tetrahydro-2*H*-pyran and 1,4-dioxane also reacted effectively with **1a** to produce the desired products (**3ai** and **3aj**) in 76% or 73% yields, respectively. No target product was detected when cyclohexanone was used as the substrate. *N,N*-Dimethylaniline was compatible with the reaction and afforded the C4-alkylated product in excellent yields (**3ak**).

From a practical perspective, the scalability of reactions and the reusability of photocatalysts are vital for the semi-heterogeneous catalytic systems. The scale-up synthesis was conducted on 5 mmol and 63% yield was obtained, demonstrating that the reaction could be scaled up effectively (Scheme 3). Additionally, the reusability and stability of the semiconductor photocatalyst were assessed via five recycling experiments in which the photocatalyst was successfully recovered and reused through simple centrifugal separation. The heterogeneous photocatalyst could retain good performance after five successive cycles with no significant loss in catalytic activity (Fig. 1a).

To gain deeper understanding of the Nd@C₃N₄/chlorine co-catalyzed semi-heterogeneous reaction, a variety of control experiments were conducted (Scheme 4). We started our investigations by studying kinetic isotope effects (KIE) of C(sp³)–H cleavage in this reaction in which a mixture of cyclohexane and its deuterated analogue cyclohexane-D in a 1:1 molar ratio was used, a distinguished kinetic isotope effect of ($k_H/k_D = 3.1$) was observed (Scheme 4a), indicating that the cleavage of the C–H bond in cyclohexane was the rate-limiting step in the reaction. The addition of radical inhibitors (TEMPO) in this photocatalytic transformation completely suppressed the generation of **3aa**, and cyclohexyl-TEMPO adduct **4aa** formed *in situ* was detected by GC–MS, suggesting the reaction involved a radical process and the generation of cyclohexyl radical (Scheme 4b). When the photogenerated



Scheme 4. Control experiments.



Scheme 5. Proposed reaction mechanism.

hole scavenger (h⁺) ammonium oxalate [NH₄]₂C₂O₄ or photogenerated electron scavenger (e⁻) K₂S₂O₈ was added to the photocatalytic reaction, the production of **3aa** was apparently quenched, providing strong evidence of the vital roles of electron and hole in this photocatalytic process (Schemes 4c and d). The reaction was completely hampered when the single electron transfer (SET) inhibitor CuCl₂ was added to the reaction, indicating a SET event was likely involved (Scheme 4e). Additionally, the "on/off" experiment demonstrated that light irradiation was essential for the reaction (Fig. 1b) and the low quantum yield ($\Phi = 0.3\%$) further ruled out the possibility of a radical chain mechanism involved in this process (Supporting information).

On the basis of the results obtained above and the literature [15,52], a probable mechanism was proposed as depicted in Scheme 5. Under the light irradiation, the composite photocatalysts Nd@C₃N₄ absorbed photons and generated photogenerated charge carriers. The introduction of Nd in intrinsic g-C₃N₄ rendered an impurity level, which could facilitate the separation of photogenerated electron-hole pairs. The photogenerated electrons (e⁻) reduced Nd³⁺ to Nd²⁺, which in turn promoted the reduction of protons (H⁺) to produce H₂ in a SET process. In parallel, chloride ions (Cl⁻) interacted with holes to form chlorine radicals in a SET event, which then engaged in a hydrogen atom transfer (HAT) process with cyclohexane **2a**, generating cyclohexyl radicals and releasing protons (H⁺). Afterwards, the cyclohexyl radical regio-selectively attacked C=N bond of *N*-sulfonyl ketimine to give intermediate **A**, followed by 1,2-H shift to yield a C-centered radical intermediate **B**. The intermediate **B** was subsequently oxidized by holes to form the cationic intermediate **C**. Finally, **C** underwent dehydrogenation and aromatization to produce the terminal product **3aa**.

The development of tumor resistance to existing chemotherapeutic agents puts forward a significant challenge in clinical can-

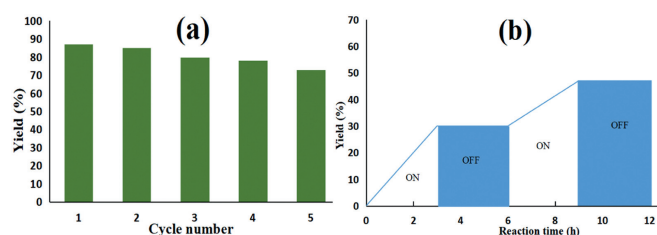


Fig. 1. Recycling experiments (a); "On/off" experiments (b).

Compound	Antiproliferation (IC ₅₀ μmol/L)	
	MUM-2B	A549
3da	1.74	3.77
3ha	1.93	3.95
3ja	4.24	4.87
3ag	3.45	5.12
3ah	3.65	5.79
5-FU	5.88	9.17

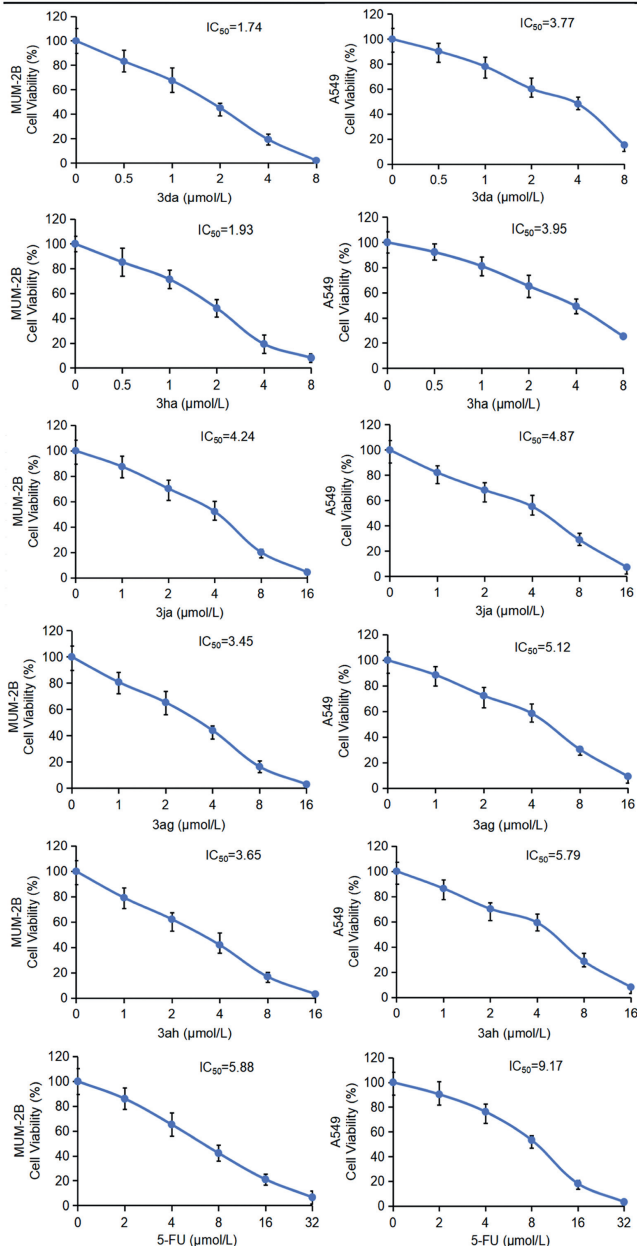


Fig. 2. Antitumor activities.

cer treatment. As a consequence, discovering new chemical compounds with antitumor effects is in high demand. The antitumor potential of synthesized compounds was assessed on human choroidal melanoma (MUM-2B cells) and lung cancer (A549 cells). Compound **3da** showed remarkable antitumor efficacy for these above-mentioned tumors, showing approximately triple the potency of the widely used antitumor drug 5-fluorouracil (5-FU) (Fig. 2). This report firstly observed antitumor activity in these novel compounds. We hope that further study of **3da** via our protocol will promote the development of new antitumor agents.

In summary, we have reported the first example of chlorine-mediated Nd@C₃N₄-photocatalyzed semi-heterogeneous synthesis of 4-alkylated *N*-sulfonyl ketimines and its antitumor activities research. With traceless and green photon as the energy source, Nd@C₃N₄ serves as a heterogeneous photocatalyst, while TBACl acts both as a redox and HAT catalyst. A variety of high-value alkylated *N*-sulfonyl ketimines were efficiently synthesized in moderate to high yields under mild conditions. Five successive reaction-separation cycles demonstrated the excellent recyclability of Nd@C₃N₄. Additionally, the high performance of Nd@C₃N₄ was validated in a gram-scale reaction, highlighting its potential for industrial applications. Mechanistic studies indicated that TBACl acted as both a redox catalyst to consume holes and a HAT catalyst to cleave C–H bonds in alkanes and the combination of the reversible Nd³⁺/Nd²⁺ and Cl[−]/Cl[•] redox pairs significantly enhanced photocatalytic efficiency. The antitumor activity experiment showed that such compounds had potential medicinal value. This semi-heterogeneous protocol provides new opportunities for organic synthesis of pharmaceutical drugs, showing potential industrial application value.

Declaration of competing interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

CRediT authorship contribution statement

Hong-Tao Ji: Methodology, Data curation. **Yu-Han Lu:** Investigation. **Yan-Ting Liu:** Investigation. **Yu-Lin Huang:** Investigation. **Jiang-Feng Tian:** Data curation. **Feng Liu:** Data curation. **Yan-Yan Zeng:** Data curation. **Hai-Yan Yang:** Data curation. **Yong-Hong Zhang:** Project administration, Methodology. **Wei-Min He:** Writing – review & editing, Writing – original draft.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.ccllet.2024.110568.

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