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ROYAL SOCIETY OF CHEMISTRY

PAPER
Chaoqiu Chen, Yong Qin *et al.*
Highly dispersed Pt nanoparticles supported on carbon nanotubes produced by atomic layer deposition for hydrogen generation from hydrolysis of ammonia borane

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Article

ARS-TiO_2 photocatalyzed direct functionalization of sp2 C-H bonds toward thiocyanation and cyclization reactions under visible light

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ARS-TiO_2 photocatalyst has been prepared, by a simple method through stirring a mixture of ARS and TiO_2 at room temperature in the dark, to extend the photocatalytic response of titanium dioxide toward the visible light spectrum. The synergic effect of ARS and TiO_2 in the photocatalyst system has catalyzed direct C-H functionalization of sp2 C-H bonds toward thiocyanation and cyclization reactions. Several aromatic and heteroaromatic scaffolds (2-phenylamino-thiazole, phenol, aniline, indole and pyrrole derivatives) treated with thiocyanate anion at room temperature. Herein, the first report for thiocyanation of phenol and synthesis of 2-aminobenzothiazole derivatives under visible light has been developed.

Introduction

Organic dyes stand at the forefront of photoredox catalysis owning to the structural variation of light absorbing molecules as well as low cost and eco-friendliness[1]. These organic chromophores take part in photoinduced electron transfer (PET) processes in variant reactions[1]. However, organic dyes suffer from short excitated state life time[1, 2].

Although TiO_2 has been highlighted as salient photocatalyst due to low cost, operational simplicity, stable and nontoxic features as heterogeneous photocatalyst, but its photocatalytic application in organic reactions has been impeded[3, 4]. This limitation arises from not only high recombination rate of photoinduced electron and hole pairs but also nearly large band gap (3.2 eV for anatase and 3.02 eV for rutile) which absorbs mainly out of visible light region[5]. Hence, the photocatalyst must be utilized under ultraviolet (UV) irritation, resulted in direct activation of the chemical bonds or generation of a highly oxidative hole (h+) in valance band of TiO_2 which both cases lead to poor selectivity of the desired products[6, 7]. Among a great number of modifications for titanium dioxide, dye-sensitized TiO_2 compile some appealing properties[5, 8]. The combination of organic dyes and TiO_2 in dye-sensitized TiO_2 has bestowed mutual advantages for both of metal oxide and organic dye. Omitting highly oxidative hole (h+) in valance band of TiO_2 which results in lessening divergent pathways toward inconsistent products as well as rectifying excitation state life time for TiO_2 and organic dye are main advantages of this cooperation. More importantly, this photocatalyst system is applicable under visible light instead of ultra violet (UV) irradiation (Scheme 1a). Dye-sensitized semiconductors have widely been surveyed in dye-sensitized solar cells (DSSCs) devices[9]. However, application of dye-sensitized TiO_2 in the organic transformation mainly remains an elusive but very tempting destination[8, 10-19]. Besides, the application of all this class of photoredox catalyst have been limited in the transformation of functional groups such as reduction of nitrobenzenes and oxidation of amines[8, 10-16, 20].

Considering its potential in organic and bioorganic research trends, substituted arenes and heterocycles containing thiocyano are abundantly available in chemistry[21]. Thiocyano synthon has widely been utilized to convert to a broad scope of sulfur-containing functional groups namely thiocarbamates, thiophenols, sulfonic acids, sulfonyl cyanides, S-acetylates, thioesters and so forth[22]. Moreover, thiocyanates have been found as outstanding synthetic precursors to prepare biological and pharmaceutical activate organosulfur compounds such as thiazoles[22, 23], thiazinones[22, 23] and thiazolidine[24]. In this context, a vast variety of protocols have been developed to access straightforward thiocyanation of aromatic and heteroaromatic compounds. Most of these methods have been established on the basis of harsh reaction condition[25] (e.g. HIO_3/ NH_4SCN), corrosive halogen reagents[26] (e.g. hypervalent iodine reagents), large amount of strong oxidants[27, 28] (e.g. oxone and DDQ), using heavy metal[29] (e.g. $Pb(SCN)_2$). So, growing attention has been drawn on superior alternative protocols in thiocyanation reactions based on visible light[23, 30-33], available and economic catalyst[34] as well as mild and green reaction condition[35, 36] (e.g., NCS instead of hypervalent iodine reagents or $Cu(OTf)_2$ instead of heavy metals). However, introducing versatile, simple and

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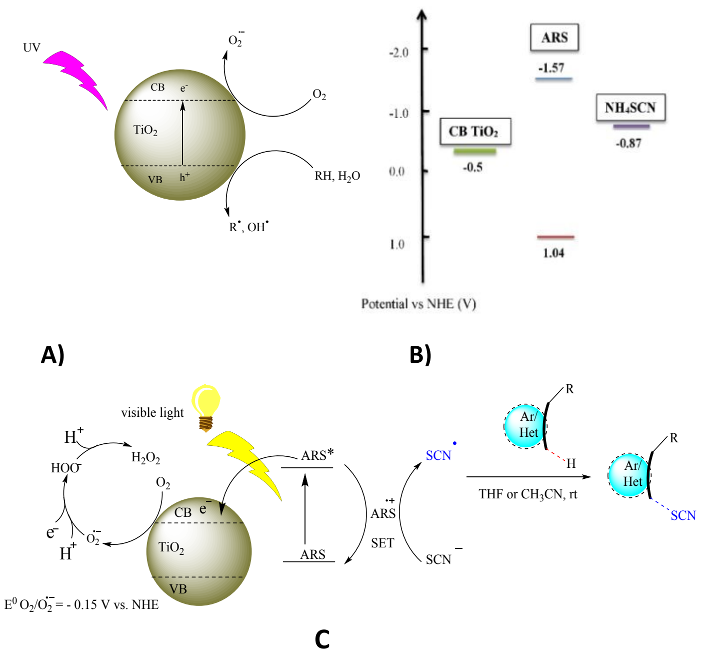
sustainable procedures in thiocyanation reactions are very desirable.

Quite recently, we introduced the application of dye-sensitized TiO_2 in C–C bond formation followed by cyclization reaction for the first time[37]. Herein, we speculated to examine the application of Alizarine Red S-TiO_2 (ARS-TiO_2) in C–S bond formation through direct thiocyanation reactions. On the basis of literature review[38], control experiments and our previous report[37], we proposed the reaction proceeds through PET process initiated by transfer an electron from the excited Alizarine Red S (ARS*) into conduction band (CB) of TiO_2. The resulting dye radical cation ($ARS^{\bullet+}$) comes back to ground state *via* single electron transfer (SET) process with thiocyanate anion which affords thiocyanate radical (Scheme 1c). These electron pathways are completely in accordance with the redox potentials of photocatalyst system and reagent (Scheme 1b). Subsequently, thiocyanate radical attacks the corresponding arene and heteroarene and affords target products. Moreover, generation of H_2O_2 has been verified using starch–iodine test (see SI).



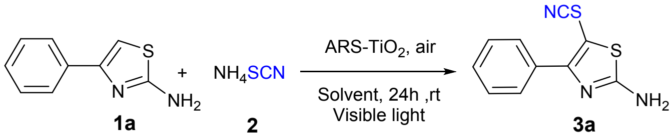
Scheme 1. (a) TiO_2 under UV irradiation; (b) Oxidation potentials of the ground and excited states of ARS, TiO_2 and NH_4SCN in CH_3CN[31, 37]; (c) application of ARS-TiO_2 in thiocyanation reaction through thiocyanate radical (E^0 $O_2/O_2^{-\bullet}$ [39]).

Results and discussion

To begin with, 2-phenylamino-thiazole was selected as model substrate. In general, thiazole scaffolds are present in a broad spectrum of biologically active and natural compounds. For example, abafungin, ritonavir, and sulfathiazole are some marketed drugs bearing thiazole fragments[40].

Three type of ARS-TiO_2 photocatalysts (0.48, 0.97 and 1.68 µmol g−1 of ARS on TiO_2) were prepared according to our previous report[37] and examined in the thiocyanation reaction under visible light at room temperature. The results have been summarized in Table 1. The reaction did not promote toward **3a** either in protic solvents (e.g. H_2O and EtOH) or $CHCl_3$ (entries 1-3). Gratifyingly, in CH_3CN 41% of target product was obtained, upon isolation (entry 4). The reaction yield had a remarkable enhancement up to 81% in THF (entry 5), but EtOAc and DMF turned out to be less effective media which afforded **3a** in 16% and 19%, respectively (entries 6,7). Afterward, we surveyed the light source impact in the reaction progress. Considering the λ max of ARS (420 nm)[37], the blue LED afforded the highest product yield rather than green and red LED (entries 8, 9). Notably, only 8% of corresponding product yielded in dark condition (entry 10). Next, 7.0 mg (1.2 g/L) of ARS-TiO_2 0.97 µmol g^{-1} and ARS-TiO_2 1.68 µmol g^{-1} were attempted in the reaction and we found that ARS-TiO_2 0.97 µmol g^{-1} containing ~ 58×10^{-10} mol ARS was the optimum photocatalyst for the reaction (entry 11). In this condition, the product yield increased up to 91%. Thereafter, three control experiment including inert atmosphere, absence of photocatalyst, and electron scavenger were accomplished (entries 12-14). Accordingly, the reaction did not proceed under Ar gas atmosphere as well as only trace amount of target product was obtained without use of the photocatalyst. In addition, in the presence of 3 equiv. of 2,2,6,6-tetramethylpiperidinyloxy (TEMPO) as electron scavenger, the reaction was completely quenched toward target product (entry 14). As expected, free titanium dioxide and ARS dye separately did not drive the reaction in considerable yields (entries 15, 16). Using the lower amount of NH_4SCN (1.5 mmol) resulted in less product yield (68%, entry 17) and using KSCN instead of NH_4SCN resulted in 64% of corresponding product (entry 18). As a consequence, light, dioxygen and photocatalyst are required to promote this reaction as well as the reaction probably progresses through SET pathway.

Table 1. Optimization of the reaction conditions[a]



Entry	Solvent	Light	Yield of 3a [%][b]

1	H_2O	15 W, Blue LED	0
2	EtOH	15 W, Blue LED	0
3	$CHCl_3$	15 W, Blue LED	6
4	CH_3CN	15 W, Blue LED	41
5	THF	15 W, Blue LED	82
6	EtOAc	15 W, Blue LED	16
7	DMF	15 W, Blue LED	19
8	THF	15W, Green LED	68
9	THF	15W, Red LED	45
10	THF	Dark	9
11	THF	15W, Blue LED	91[c], 91[d]
12[e]	THF	15 W, Blue LED	4
13[f]	THF	15 W, Blue LED	trace
14[g]	THF	15 W, Blue LED	0
15[h]	THF	15 W, Blue LED	18
16[i]	THF	15 W, Blue LED	22
17[j]	THF	15 W, Blue LED	68
18[k]	THF	15 W, Blue LED	64

[a]*Reaction conditions*: A mixture of **1a** (1 mmol), **2** (3 mmol) and photocatalyst (7.0 mg, 0.48 µmol g-1, containing ~ 33×10 $^{-10}$ mol ARS), solvent (6 mL), 15 W blue LED lamp (λ > 440 nm) open air for 24 h at room temperature. [b] Isolated yield. [c]7.0 mg ARS-TiO$_2$ 0.97 µmol g^{-1}, containing ~ 58×10 $^{-10}$ mol ARS. [d]7.0 mg ARS-TiO$_2$ 1.68 µmol g^{-1}, containing ~ 1.1×10 $^{-8}$ mol ARS. [e] 1 atm Ar balloon. [f]Reaction was performed in the absence of photocatalyst. [g]3 equiv. of TEMPO was added to the reaction. [h] 3 mol % ARS . [i] 3 mol % TiO$_2$ (P25). [j] 1.5 mmol NH$_4$SCN.[k] 3 mmol of KSCN instead of NH$_4$SCN.

Scheme 2. Screening the scope of 2-Amino-4-phenylthiazole derivatives in thiocyanation reaction [a,b]

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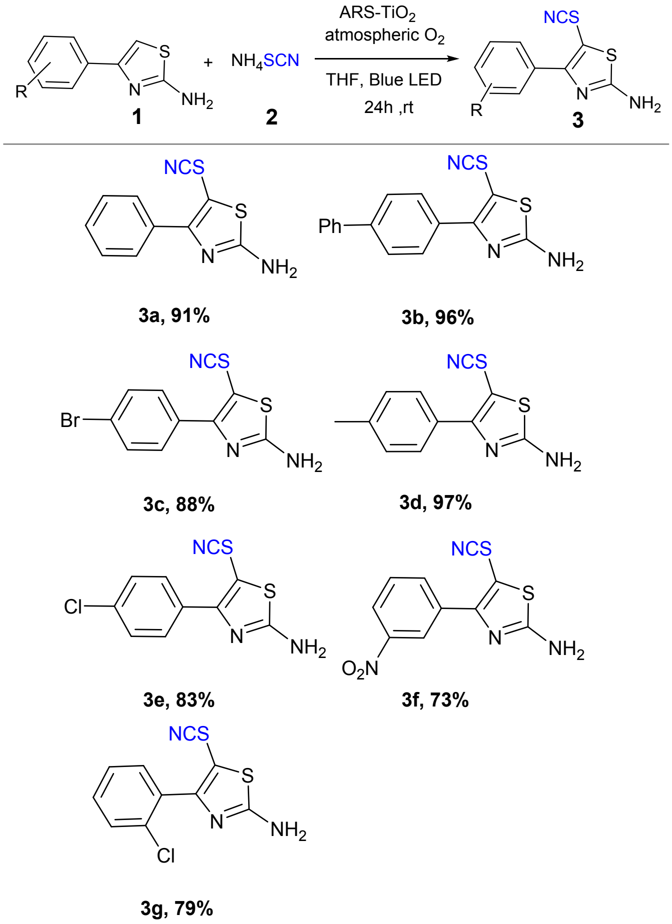

[a] *Reaction conditions:* A mixture of **1a** (1 mmol), **2** (3 mmol) and ARS-TiO$_2$ (7.0 mg, 0.97 µmol g−1) in THF (6 mL) was irradiated by 15 W blue LED lamp (λ > 440 nm) open air, 24 h, at room temperature. [b]Isolated yield.

Then, in order to realize the generality and scope of this reaction, we examined several 2-phenylamino-thiazoles containing electron withdrawing and pushing groups and the results were pictured in Scheme 2. Notably, electron eletron neutral and pushing groups afforded more efficiency than withdrawing groups (**3e**, **3f** and **3g**). Precursors bearing halogen groups such as chloro, and bromo could be tolerated under this protocol (**3c**, **3e** and **3g**), even strong electron withdrawing substituents, such as NO$_2$ group is active well under optimized condition (**3f**).

Afterward, we turned our attention to study the potential of this photocatalytic system in the thiocynation reaction of more heterocycle compounds (scheme 3). Firstly, we prepared 2-Amino-4-phenyloxazole as a chemical analog of 2-Amino-4-phenylthiazole. As shown, the photocatalytic reaction did not result in the corresponding product. It is possible the precursor is not electron rich enough to attack to thiocyanate radical and generate target product (**5**). However, we prepared 2-phenylimidazo[1,2-a]pyridine and the reaction condition afforded 89 % of corresponding product under the same optimized condition (**7**). Moreover, less electron rich heterocycle such as quinolone was inactive under this

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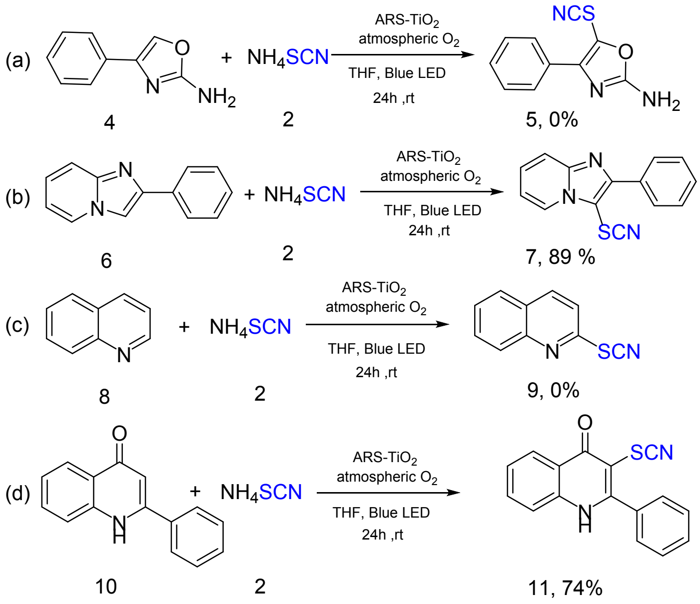
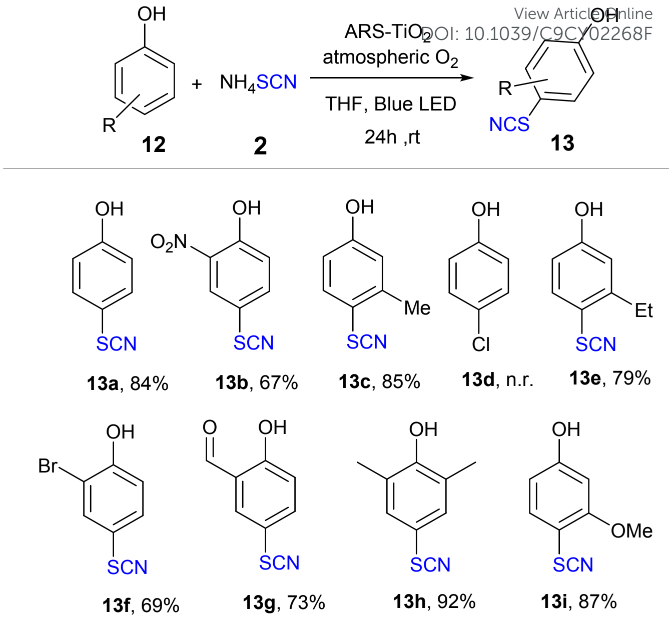
photocatalytic condition (**9**). Also, we tried thiocyano group on a biologically active compound[41-43], 2- phenylquinolin-4-one, and 74 % of the corresponding product was obtained under our condition (scheme 3d).

Scheme 3. Scope of the thiocyanation reaction of heterocyclic substrates containing nitrogen atom.



Encouraged by these results, we surveyed thiocyanation reaction of phenol using previous condition. To the best of our knowledge, there is no visible light-driven report in thiocyanation reaction of phenol derivatives. To our delight, the treatment of ammonium thiocyanate with phenol under this condition afforded 84% of corresponding product (**13a**) (Scheme 4). As shown in Scheme 4, this procedure is compatible with varied functional groups such as halogen, nitro, alkyl, methoxy, and aldehyde. It is also regioselective for thiocyanation at para position. Similar to thiocyanation reaction of 2-Amino-4-phenylthiazole derivatives, electron pushing groups have exhibited higher efficiency than electron withdrawing groups. Using 4 chlorophenol, as reagent, did not lead to any target product under this condition (**13d**). Notably, the hindrance effect of the reaction was completely negligible when the precursors with Me, MeO, and Et substitutions at meta position were attempted in the reaction (**13c**, **13e**, and **13i**).

Scheme 4 Screening the scope of phenol derivatives in thiocyanation reaction [a,b]



[a] *Reaction conditions:* A mixture of **12** (1 mmol), **2** (3 mmol) and ARS-TiO_2 (7.0 mg, 0.97 µmol g−1) in THF (6 mL) was irradiated by 15 W blue LED lamp (λ > 440 nm) open air, 24 h, and at room temperature. [b]Isolated yield.

Afterwards, aniline was examined under above-mentioned condition. It was found that THF yielded only 37% of target product (**15a**). To screen this reaction condition, several solvents, light and photocatalyst amount were assayed in thiocyanation reaction of aniline and results are summarized in Table S1 (See SI). CH_3CN turned out to be the best solvent in the reaction. Then, several structurally diverse anilines were analyzed in this condition and the results were illustrated in Scheme 5. The reactions proceeded to satisfactory yields. *N*-methylaniline and *N*-ethylaniline also afforded very good yields under optimized condition (**15b** and **15d**). Tertiary amines such as *N,N*-dimethylaniline (E_{ox} = + 0.96 V vs. NHE)[37] are good electron sacrificers to quench the photoexcited state of ARS-TiO_2 and afford amine radical cations. Hence, this class of tertiary amine is conqueror in competition with thiocyanate anion (E_{ox} = + 0.87 V vs. NHE) in PET process. Presumably, that is why treatment of ammonium thiocyanate with *N,N*-dimethylaniline and *N,N*-diethylaniline afforded lower yields and prolonged reaction time (**15f**, **15g**). Notably, thiocyanation reaction of diphenylamine selectively provide **15j** which -SCN have been placed at *para* position of one phenyl ring. The precursors containing chloro and methoxy substitutions at *meta* position were well tolerated the reaction condition. So, the reaction was not affected by steric hindrance at this position (**15c** and **15i**).

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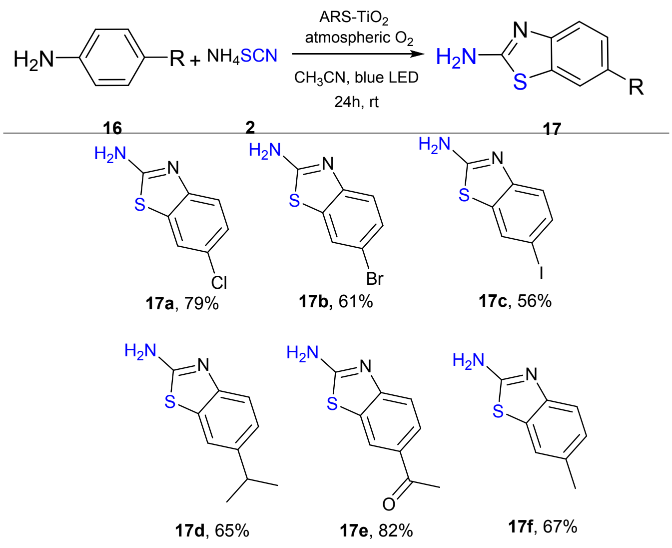
Interestingly, in case of occupation of *para* position, 2-aminobenzothiazole was the dominant product (**17**). As regards, investigation of generality scope of 2-aminobenzothiazole derivatives are shown in scheme 6. To the best of our knowledge, this is the first report of visible light-driven synthesis of 2-aminobenzothiazoles. This protocol also afforded target products **17a**-**17f** in good to excellent yields. Notably, the more electron withdrawing halogen at *para* position provided higher yields rather than iodide under photocatalytic condition (**17a**-**17c**).

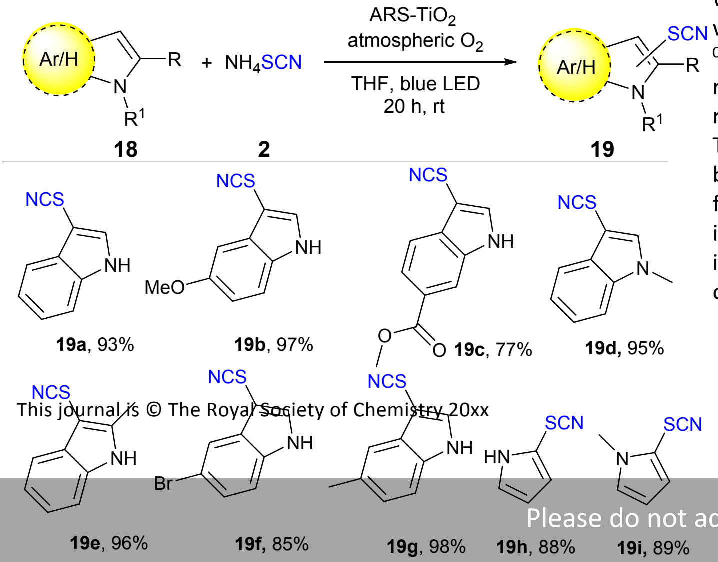
Scheme 6 Screening the scope of 2-aminobenzothiazole derivatives in the reaction [a,b]



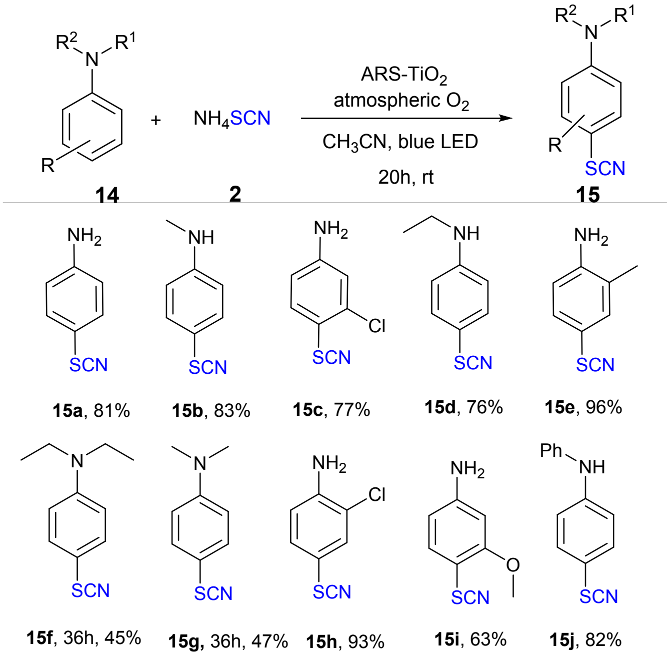
[a]*Reaction conditions:* A mixture of **16** (1 mmol), **2** (3 mmol) and ARS-TiO_2 (7.0 mg, 0.97 μmol g−1) in CH_3CN (6 mL) was irradiated by 15 W blue LED lamp (λ > 440 nm) open air, 24 h, at room temperature. [b]Isolated yield.

To continue substrate investigation in direct thiocyanation reaction, indoles and pyrroles were selected as two class of important heterocycles in organic and bioorganic chemistry[44]. These model reactions were performed under foregoing condition and good to excellent product yields were obtained in shorten time (Scheme 7). The photocatalytic condition handled precursors bearing varied functional groups even indole with C-2 steric hindrance (**19e**).

Scheme 7 Screening the scope of indole and pyrrole derivatives in thiocyanation reaction[a,b]



Scheme 5 Screening the scope of aniline derivatives in thiocyanation reaction [a,b]

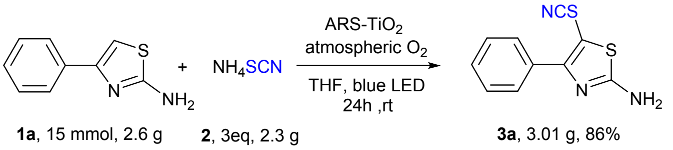


[a]*Reaction conditions:* A mixture of **14** (1 mmol), **2** (3 mmol) and ARS-TiO_2 (7.0 mg, 0.97 μmol g−1) in CH_3CN (6 mL) was irradiated by 15 W blue LED lamp (λ > 440 nm) open air, 20 h, at room temperature. [b]Isolated yield.

[a]*Reaction conditions:* A mixture of 6 (1 mmol), 2 (3 mmol) and ARS-TiO_2 (7.0 mg, 0.97 μmol g−1) in THF (6 mL) was irradiated by 15 W blue LED lamp (λ > 440 nm) open air, 20 h, at room temperature. [b]Isolated yield.

In adlast paragraph)dition, the reusability and stability survey of the photocatalyst system were performed under our optimized condition for **1a** substrate. When the reaction was completed, it was centrifuged to remove the photocatalyst. ASR-TiO_2 was washed three time with THF and dried on the vacuum oven at 30 ^{0}C. Similarly, the photocatalyst was utilized for second and third runs which provided 80 % and 61 % of target product (**3a**), respectively. The amounts of anchored ARS per unit gram of the TiO_2 powders were measured according to the reported method by Jang et al[17]. The results revealed that the amounts of ARS was found to be about 0.49 μmol g^{-1} and 0.26 μmol g^{-1} for ARS-TiO_2, in turn, after second and third runs. These drastically decreases in the amount of ARS on the TiO_2 might come from degradation or desorption of the anchored ARS. However, a comparison between FT-IR spectra of recovered ARS-TiO_2 (after third run) and untreated ARS-TiO_2 revealed no significant change in the absorption peaks of the remaining dye on the surface of titanium dioxide (See SI Figure S3). Moreover, the XRD patterns of photocatalysts, before and after recycling (after third run), exhibited no considerable change in the structure of TiO_2 during the reaction (See SI Figure S4).

Finally, we surveyed the possibility of a gram-scale thiocyanation reaction under our optimized condition. In this context, 3.01 g (86%) of target product (**3a**) was obtained, upon isolation which showed the merit of a practical procedure in thiocyanation reaction (scheme 8).



Scheme 8. Gram-scale thiocyanation of 2-phenylamino-thiazole.

Conclusions

In conclusion, we have developed a versatile, sustainable and regioselective thiocyanation protocol through direct C–S bond formation as well as the first visible light-driven synthesis of 2-aminobenzothiazole. A vast majority of important precursors treated with ammonium thiocyanate in satisfactory yields. Notably, the method is scalable and compatible with varied functional groups.

Experimental section

Preparation of ARS-TiO_2 photocatalyst.

At first, the TiO_2 powder was heated at 110 °C in air for 3 h to remove adsorbed water on the surface. Dyes-TiO_2 were prepared by a minor modified reported procedure[45]. Typically, three different molar concentration of ARS dye (0.1 mol/L, 0.2 mol/L and 0.4 mol/L) in ethanol were added to 1g of pre-heated nano TiO_2 (P25) and stirred at room temperature in the dark for 12 h. The solid was centrifuged and washed with ethanol five times and dried at 40 °C for 30 h on the vacuum oven and kept in the dark.

General procedure for thiocynation reactions.

A mixture of aromatic compounds and (1 mmol) and ammonium thiocyanate (3 mmol) in THF or CH_3CN (6 mL) placed in 10 mL round bottom flask, ARS-TiO_2 (7.0 mg, 0.97 μmol g−1) was added and the reaction was irradiated by 15 W blue LED Lamp (λ > 440 nm, distance app. 4.0 cm) in open air condition. The reaction was stirred for appropriate time at room temperature (25 °C) and monitored by TLC. After completion, the reaction mixture was centrifuged to separate the photocatalyst, then, the reaction was diluted with CH_2Cl_2 /EtOAc (15mL) and washed with brine. The combined organic phases were dried over $MgSO_4$ and the solvent was evaporated under reduced pressure to obtain the crude. Final product purification was done through silica gel column chromatography using petroleum ether/ethyl acetate.

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