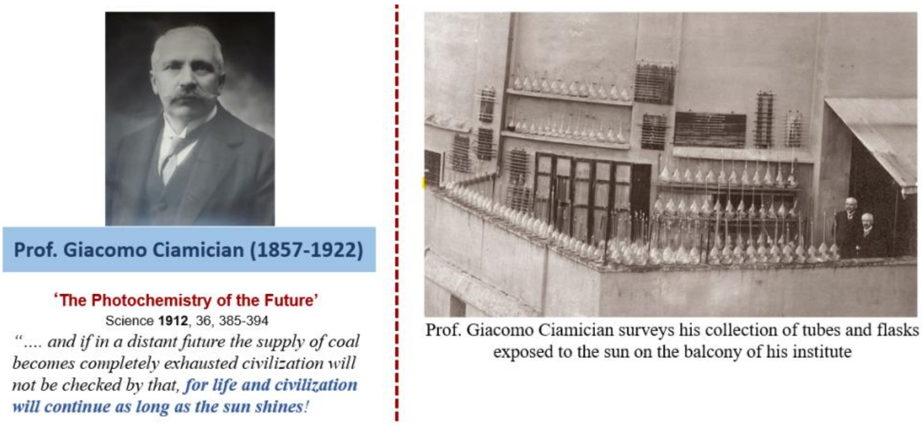
1.1 Introduction

Present-day society has its roots deeply embedded in the utilization of fossil fuels, which enable essential functions like transportation, heavy industry, building heating, and electricity generation. However, the combustion of fossil fuels impairs critical issues such as global warming, resource depletion, and environmental degradation. Despite technological advancements enhancing their efficiency, fossil fuels remain unsustainable as they are finite resources [1-3]. Coal, oil, and natural gas deposits are constrained and non-renewable sources of energy and cannot be replenished once depleted. If the current pace of energy consumption continues, it is expected that oil and natural gas supplies will decline dramatically over the next century. While coal reserves are more abundant, their utilization exacerbates environmental problems like global warming [4]. The future imperative lies in embracing alternative energy sources to meet the escalating energy demands of industrialized nations and developing economies [5]. Energy conservation and more efficient utilization are also crucial approaches to address the energy crisis. Effective mitigation of fossil fuel related consequences necessitates concerted international efforts to enact changes in behavior, policy, and consumption [6-8]. The Paris Agreement, a collective initiative aimed at combating climate change, sets motivated targets to limit global temperature rise to 1.5°C. Each nation has assorted policies for achieving these objectives, but prioritizing renewable energy development is paramount to lessen pressure on modern industries. Nature has consistently served as an unparalleled source of inspiration for chemists, driving the development of innovative reactions aimed at achieving more effectual and selective formation of organic molecules.

Solar energy stands out among various renewable energy sources due to its exceptional attributes, including its long lifespan, limitless, potent, and pollution-free nature, accessibility on-site, and numerous other advantages [9-11]. This is attributed to the fact that the daily solar radiation reaching earth surpasses the amount required to fulfill the entire world's annual energy needs. Recent years have witnessed a shift towards utilizing visible light photons as an environment-friendly energy source for activating reactants and intermediates in sustainable synthesis. As a traceless source of energy for activating substrates and intermediates, visible light offers additional benefits such as enhanced atom economy and green catalysis. Moreover, it serves as a paradigm for designing organic molecules [12-16].



*"…. and if in a distant future the supply of coal becomes completely exhausted civilization will not be checked by that, **for life and civilization will continue as long as the sun shines!***

Prof. Giacomo Ciamician surveys his collection of tubes and flasks exposed to the sun on the balcony of his institute

Figure 1.1 A picture showing the balcony of Prof. Giacomo Ciamician's lab where he used to perform his experiments (Ref- 17)

Over a century ago, Ciamician, who served as a chemistry professor at the Bologna University in Italy from 1889–1922, laid the foundation for harnessing sunlight as a renewable energy source in molecular construction (Figure 1.1). His inspiration stemmed from observing the ability of plants to harness solar energy, leading to his pioneering contributions to the field of photochemistry. Ciamician's deep recognition of the significance of sunshine is exemplified by his profound statement, "*there is another agent that has a major effect on the activities of life and that deserves to be extensively investigated: that is light*" [17-19].

He enthusiastically encouraged harnessing the daily solar energy received by earth to replace fossil fuel like coal. He accurately predicted the development of fuels through simulated photochemical processes, commonly known as artificial photosynthesis, which remains a primary focus of modern chemistry research. His words reflect a deep appreciation for the potency of sunlight: *"...and if in a distant future the supply of coal becomes completely exhausted civilization will not be checked by that, for life and civilization will continue as long as the sun shines!"* [19].

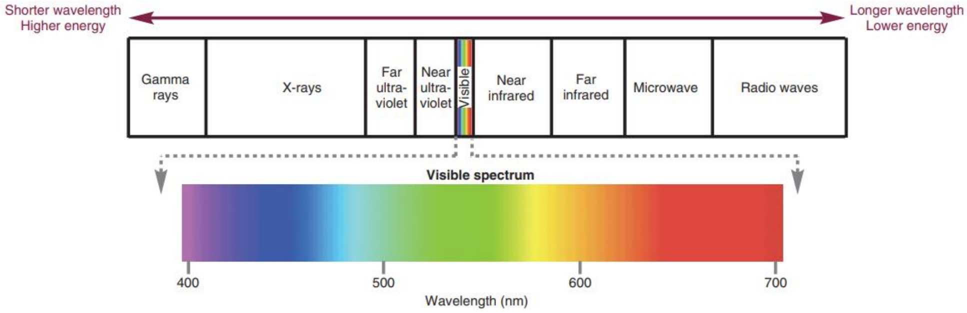


Figure 1.2 Electromagnetic spectrum (Image- Internet source)

The solar spectrum received on Earth’s surface is predominantly in visible and near-infrared range, whereas near ultraviolet contributes minimally (Figure 1.2). Given its vast natural prevalence, harnessing visible light as a source of energy in synthesis holds significant appeal. Unlike UV light, the management of visible light is straightforward, safe, and has promising prospects for industrial-scale applications [20,21].

During his investigations, Ciamician faced an experimental obstacle: the necessity of a reliable light source for conducting photochemical studies. In modern research, scientists utilize robust halogen, mercury, and tungsten lamps equipped with light filters, enabling them to select nearly monochromatic light beams or LEDs (Figure 1.3). Laser sources, both pulsed and continuous, are increasingly prevalent in photochemical laboratories. However, at the dawn of the 20th century, options were limited; mercury, halogen, LED, or laser light sources were non-existent, and tungsten lamps emitted dim, reddish light insufficient for inducing photochemical reactions. Consequently, the balconies of Ciamician's institute served as his laboratory's most suitable location, as sunlight remained the sole useful light source for his photochemical investigations (Figure 1.1).

Figure 1.3 (a) LEDs with precise wavelengths (b) Reaction setup in our lab

The remarkable advancement of visible light LED technology has manufactured LEDs with precise wavelengths (thin emission bands) and adjustable intensities, easily modulated by altering the current. This advancement enables more precise photochemical reactions through selective excitation of the photocatalyst, thereby minimizing the excitation of non-targeted functional groups or substrates and reducing unwanted side-product formation. Visible light photons serve as a cost-effective light source and can now be readily generated at lower energy costs using energy-efficient high-power LED technology [22,23].

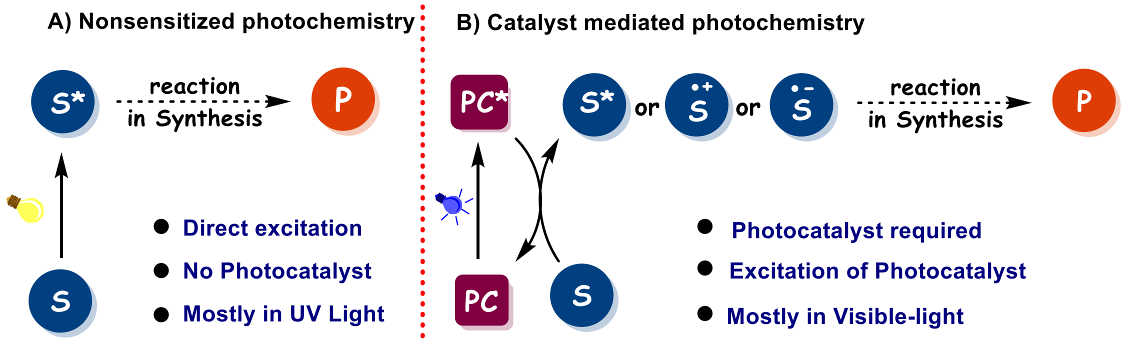
1.2 Mechanistic pathways of photochemical reactions

The field of photochemistry explores the chemical processes prompted by light absorption. Nonetheless, the advancement of photochemistry has encountered two primary challenges. Firstly, it comprises precisely regulating and manipulating solar radiation, essential for efficient photochemical reactions. The development and effectiveness of visible light-initiated reactions over time have been intertwined with the evolution of cost-effective and potent lighting tools. Secondly, visible light photons possess relatively low energy compared to other regions of the electromagnetic spectrum, as described by the equation of photon energy ($E = hc/\lambda$, where h represents Planck's constant, c symbolizes the speed of light, and λ denotes the wavelength). For a photochemical reaction to be initiated, an organic molecule must absorb a visible light photon with adequate energy to promote an electron from the ground state to an excited state [12-14, 24].

Various species can capture the photon's energy and shift into an electrically excited state, thus initiating a chemical reaction (Scheme 1.1). There are two primary methods to initiate chemical reactions [25-28]:

i) Direct excitation of the reactants or reaction intermediates (direct photochemistry).

ii) Utilization of colored photocatalysts to absorb light and subsequently activate non-absorbing substrates.



Scheme 1.1 (a) Direct photochemistry, and (b) Photocatalyzed mechanism

Visible light photocatalysis, despite relying on catalytic action in photons, is defined as a chemical process facilitated by a catalytic component known as the photocatalyst (PC). These photocatalysts absorb visible light within the range of 380–780 nm and participate in the conversion of organic molecules. They serve as intermediaries between visible-light photons having lower energy and more energy-requiring organic molecules. Furthermore, they can interact with organic molecules through single-electron transfer or energy transfer mechanisms by raising the energy level in their excited state. This generates reactive intermediates, including radicals and radical ions, which drive organic conversions and convert visible light into chemical energy [10, 29-32].

An optimal photocatalyst should possess several key properties:

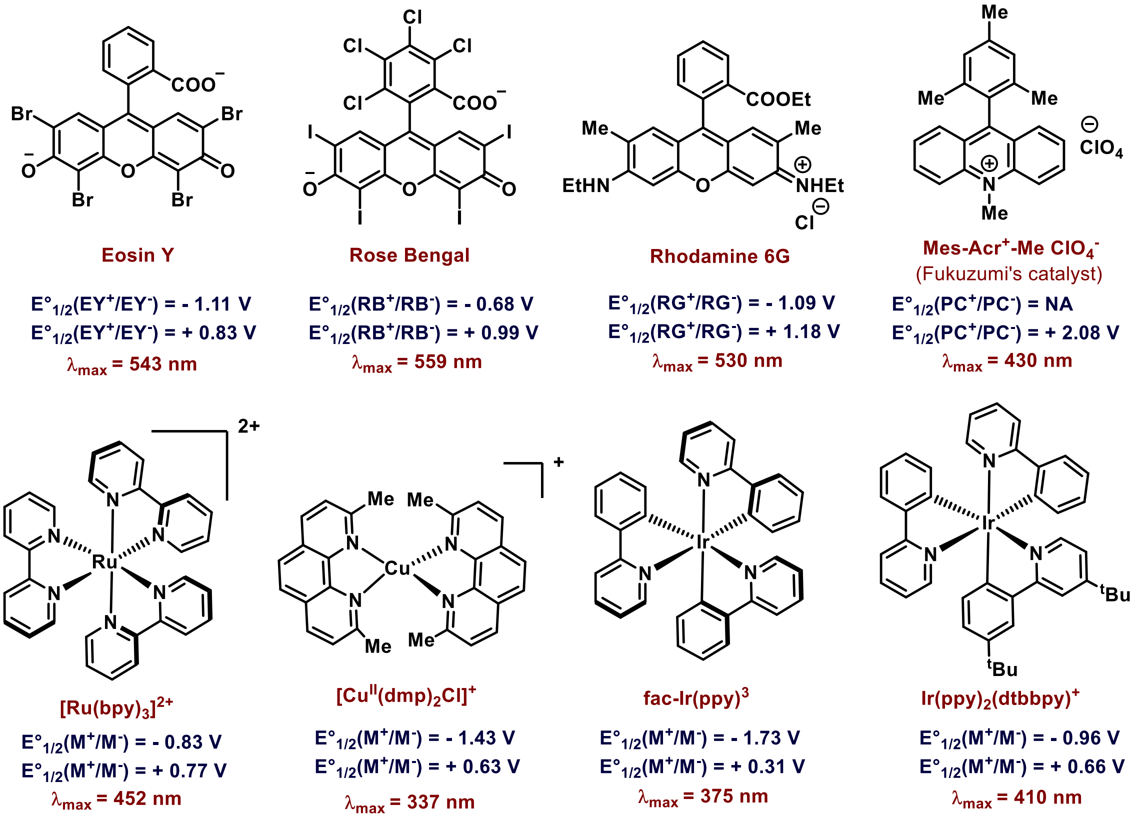
i) significant absorption in the visible spectrum (>400 nm),

ii) an excited-state lifetime preferably longer than one nanosecond to facilitate efficient bimolecular energy transfer/ electron transfer,

iii) stability of the excited state in the reaction mixture, and

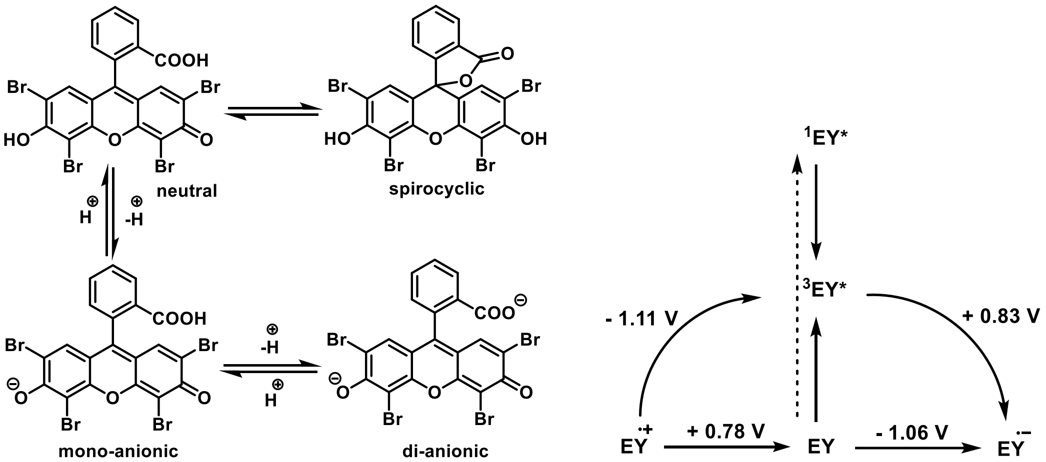
iv) strong reduction and/or oxidation potential compared to their ground states for single-electron-transfer reactions with suitable organic moieties.

The structures of some photocatalysts utilized in visible-light-mediated photochemical reactions are illustrated in Scheme 1.2. The commonly employed photocatalysts are valuable metal complexes, characterized by a central metal coordinated with organic ligands that serve as electron acceptors. Despite the advancement of metal-based photocatalysts, visible light photocatalysis is increasingly exploring organic photocatalysts due to their availability, low cost, reduced toxicity, and biocompatibility. A few well-known examples of transition metal photocatalysts are $[Ru(bpy)_3]^{2+}$, $[Cu(dap)_2]^+$, fac-Ir(ppy)$_3$, Ir(ppy)$_2$(dtbbpy)$^+$ and that of organic photocatalysts are pyrylium and acridinium salts, cyanoarenes, and organic dyes such as rose bengal, rhodamine 6G, fluorescein, Fukuzumi's catalyst, rhodamine, and eosin Y [29-38].



Scheme 1.2 Example of some photoredox catalysts**.**

A metal-free and easily available organic dye, eosin Y, has emerged as a preferable choice over transition-metal-based photocatalysts due to its economic and ecological advantages. Unlike other organic dyes, eosin Y features distinctive xanthene and phenol moieties, along with notable acid-base properties, resulting in four distinct structures: spirocyclic, monoanionic, neutral, and di-anionic forms (Scheme 1.3a). Redox potentials of the excited state of eosin Y can be estimated by taking the energy of the triplet excited state and the standard redox potentials of the ground state, which are found via cyclic voltammetry (Scheme 1.3b). Also, the excited eosin Y absorbs light at wavelengths of 329 nm and 543 nm, while eosin Y-H absorbs at 366 nm, as determined by transient absorbance tests. A process that requires both light and a catalyst (which is not consumed) is referred to as a "photocatalytic reaction" [39-42].



Scheme 1.3 a) Different forms of eosin Y depending on pH,
b) Excited states of eosin Y

The photoexcited molecules interact with other reaction species through various mechanisms: photoinduced electron transfer (photoredox catalysis), hydrogen atom transfer (HAT), energy transfer (photosensitization), and electron-donor-acceptor (EDA) complex.

1.2.1 Photoinduced electron transfer (photoredox catalysis)

The interaction between the excited-state photocatalyst (PC*) and an electron-donor (D) or electron-acceptor (A) initiates a redox process termed photoinduced electron transfer (PET). This PET process, also referred to as photoredox catalysis, has developed in the last decade as a potent tool in synthetic organic chemistry. By converting visible light energy into a chemical potential, various organic transformations can be initiated, enabling the development of novel synthetic methodologies. PET promotes an electron from the excited photocatalyst (PC*) via a single electron transfer (SET) process to the organic molecule or sacrificial species, or conversely, promoting the catalytic formation of more reactive radical intermediates under minimal conditions [32,37,43-46].

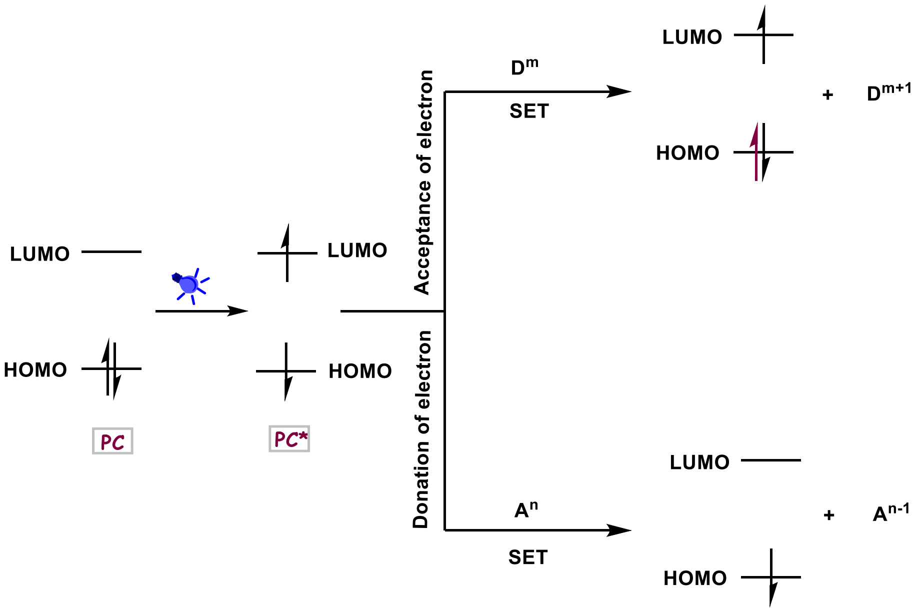
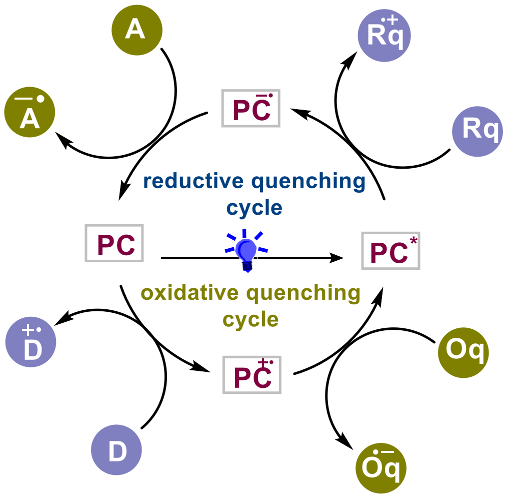


Figure 1.4 Photoinduced electron transfer mechanism

Upon photoexcitation of the catalyst, an electronic transition occurs from the highest occupied molecular orbital (HOMO) to the lowest unoccupied molecular orbital (LUMO), creating a vacancy in the HOMO and thereby increasing the electron affinity of the photocatalyst, facilitating electron donation from donor (D^m). Simultaneously, an electron resides in the higher-energy LUMO, reducing the ionization potential and facilitating electron acceptance to acceptor (A^m). This phenomenon elucidates the promising redox characteristics of the excited state compared to the ground state (Figure 1.4).

Based on the redox potential of the activated photocatalyst, two important catalytic cycles can transpire: oxidative and reductive quenching cycles (Scheme 1.4).



Scheme 1.4 Mechanism of single electron transfer**.**

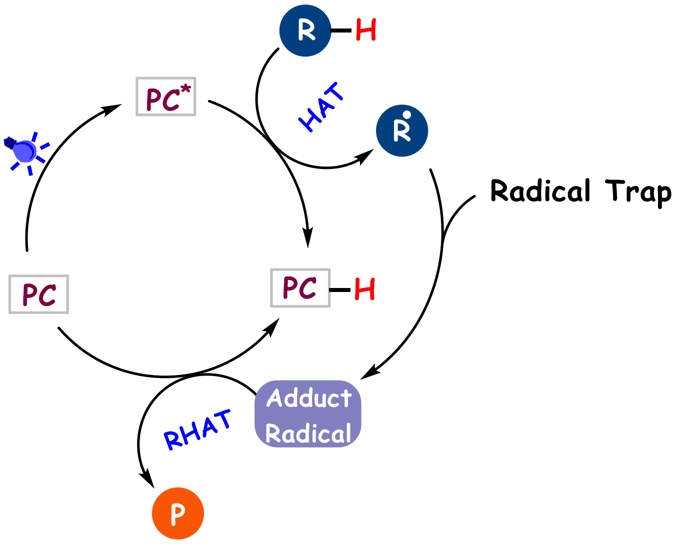
When exposed to an appropriate wavelength of light, a photocatalyst (PC) can be excited to produce an excited species (PC*) with an electron of higher energy. This electron can then be transferred to a quencher (Oq), resulting in the formation of a radical cation ($PC^{\bullet+}$). The quencher subsequently generates a stable anionic radical ($Oq^{\bullet-}$) after undergoing one-electron reduction, which undergoes further reductive transformation. By absorbing an electron from a donor molecule (D), the photocatalyst's cationic radical species ($PC^{\bullet+}$) returns to its ground state and gets ready for the subsequent cycle (oxidative quenching cycle). Conversely, in the reductive quenching cycle, the excited photocatalyst (PC*) receives an electron from the donor molecule and gives it to an acceptor molecule (A), thus returning to the ground state and completing the catalytic cycle. These redox events may also involve a concerted proton transfer known as proton-coupled electron transfer (PCET) [28,33,47-49].

Understanding the redox potentials of the molecules involved in a photoredox process is crucial to estimating if a targeted single electron transfer (SET) will be thermodynamically feasible. In a reductive quenching cycle, where the excited state photocatalyst is quenched by the donor, a photocatalyst having an excited-state reduction potential greater than the oxidation potential of the donating molecule should be selected. Conversely, in an oxidative quenching cycle, a photocatalyst having an excited-state oxidation potential lesser than the reduction potential of the accepting molecule should be chosen to achieve thermodynamically favorable SET [28].

1.2.2 Hydrogen Atom Transfer (HAT)

Hydrogen atom transfer (HAT) involves the simultaneous movement of a proton and an electron between two molecules in a single kinetic step (Scheme 1.5) [50]. This fundamental chemical transformation plays a pivotal role in various reactions, including hydrocarbon combustion and aerobic oxidations, as well as influencing atmospheric processes. In biology, numerous metalloenzymes rely on HAT mechanisms, and extensive research has focused on understanding their role in eliminating the harmful effects of in-vivo generated reactive oxygen species (ROS) and the function of antioxidants [51-53]. In organic synthesis, HAT offers unique opportunities by enabling the straightforward activation of aliphatic R-H bonds, often with high selectivity, without requiring the introduction of directing groups into substrates [54].

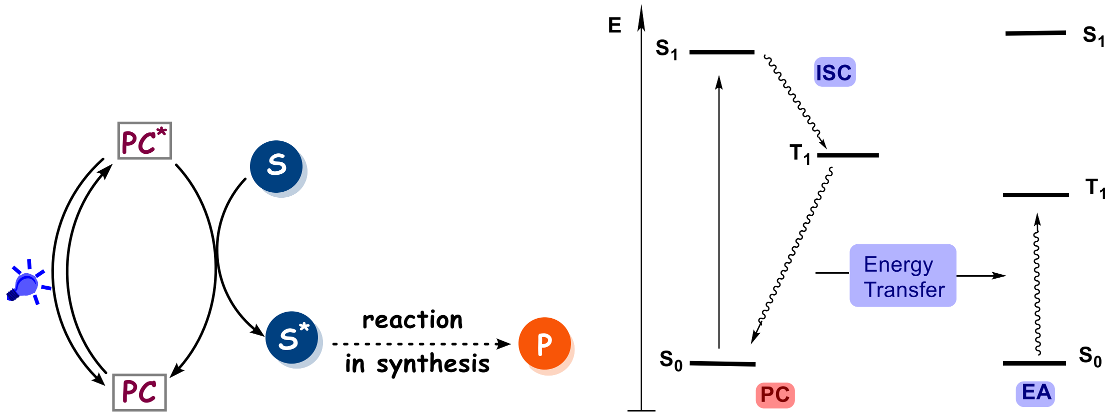
The HAT process produces carbon radicals by selectively cleaving (homolytic cleavage) C(sp^3)–H bonds, in a variety of hydrogen donors, including alkanes, ethers, amides, and aldehydes, using the energy of photon as a catalyst. These radical intermediates serve as reactive species for the formation of carbon-carbon and carbon-heteroatom bonds through reactions with specific reactants. In the process, the excited state of the photocatalyst (PC*) directly abstracts a hydrogen atom from a substrate R-H in a direct HAT process to generate an alkyl radical. The catalytic cycle is completed through a reverse-HAT step to one of the intermediates generated during the process [55-58].



Scheme 1.5 Mechanism of HAT process

1.2.3 Energy Transfer (photosensitization)

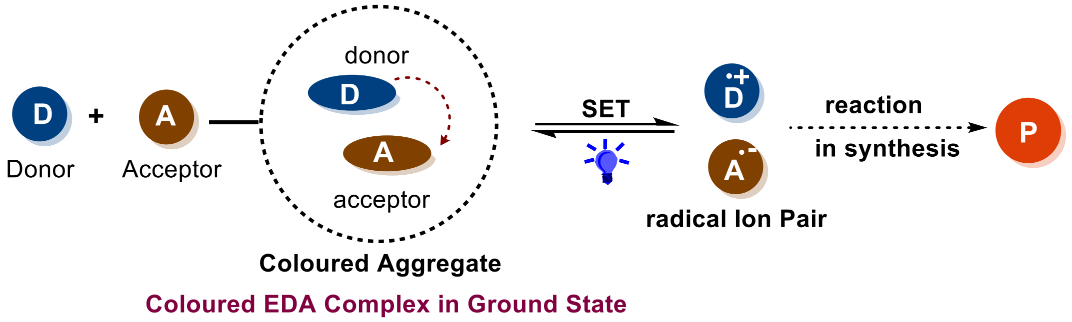
It is essential to differentiate between electron transfer and energy transfer mechanisms in discussions regarding visible-light-mediated photocatalysis. Electron transfer photocatalysis utilizes the strong redox potential of an excited state molecule to initiate single electron transfer (SET), which has gathered important consideration in recent years. On the other hand, energy transfer is formally described as “the photophysical method wherein energy from one chemical entity (the donor or photocatalyst) is transferred to another molecular entity (the acceptor S), raising it to a higher energy level and regenerating the electronic ground state of the donor” (Scheme 1.6). Many organic moieties do not undergo SET by photoredox catalysis as they exhibit comparatively high oxidation or reduction potentials that are not compatible with the excited state of photocatalysts. To surmount this challenge, a strategy involves using excited photocatalysts to transform organic molecules into their triplet states through energy transfer, a process recognized as photosensitization [30,31,59-61]. Upon photon absorption, the ground singlet state (S_0) photocatalyst firstly excites to its lowest singlet excited state (S_1) and consequently attains its lowest-energy triplet state (T_1), having a longer lifetime, through intersystem crossing (ISC). Subsequently, the triplet state photocatalyst (the donor, PC*) decays to its ground singlet state from its triplet state, promoting the singlet ground state of the organic molecule (S, the acceptor) to its lowest-energy triplet state (T_1) via an energy transfer process.



Scheme 1.6 Photochemistry of Energy Transfer pathway

1.2.4 Electron-Donor-Acceptor (EDA) Complexes

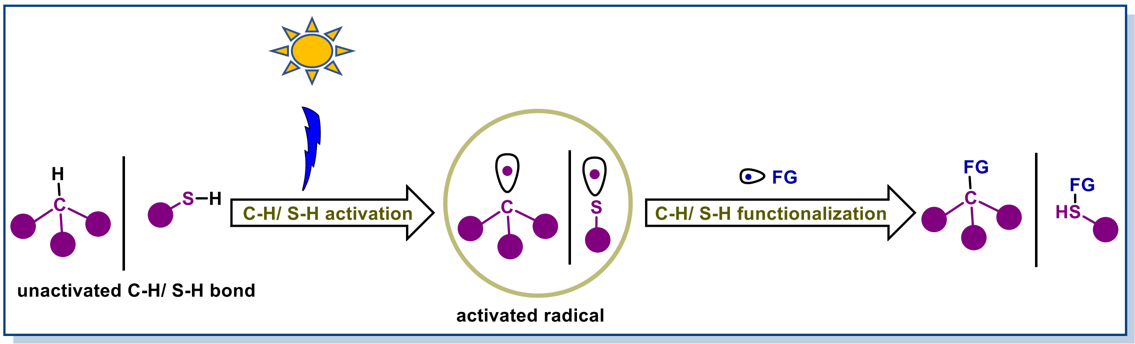
In recent times, synthetic chemists have recognized the importance of a photochemical method distinct from photoredox catalysis, as it operates without the need for an external photoredox catalyst. This method relies on the interaction between an electron-acceptor molecule (A) and an electron-donor moiety (D), typically Lewis acids and bases, respectively, resulting in the generation of a new molecular complex in the ground state termed as an electron-donor-acceptor (EDA) complex (Scheme 1.7). Although the individual constituents A and D do not absorb visible light, the formed EDA complex does. Upon excitation by light under mild conditions, an intramolecular single electron transfer (SET) from donor to acceptor occurs, leading to the formation of a radical ion pair which further leads to the formation of the desired product. This process activates widely accessible substrates, resulting in the production of reactive radicals under extremely mild reaction conditions [62-66].



Scheme 1.7 Catalyst-free mechanism *via* EDA complex

1.3 C-H/S-H Functionalization

At the start of the century, activating C-H/S-H bonds i.e C-H/S-H functionalization, posed a significant challenge due to the low reactivity of these bonds. In today's context, this area has emerged as one of the extensively investigated areas for the synthetic chemists. This includes both the breadth of feasible transformations and the variety of catalytic methods facilitating C−H/S-H functionalization. The term "functionalization" refers to a procedure that involves C–H/S-H bond activation followed by C–H/S-H transformation, which reduces the number of steps in multi-step synthesis [67-69]. This method reduces the requirement for drawn-out, labor-intensive protection-deprotection processes and also lowers the likelihood that undesirable by-products would emerge (Scheme 1.8).



Scheme 1.8 Photo-Induced C-H/S-H functionalization

Selective functionalization of C-H/S-H bonds has been achieved by a variety of catalytic techniques, including the use of transition metals, enzyme systems, photochemical methods, electrochemical approaches, and photoredox systems. The field of photoredox catalysis for C-H/S-H bond functionalization is rapidly expanding and intersecting with various scientific disciplines, including renewable energy and chemical feedstock applications, novel reaction discovery, natural product synthesis, materials science, and biological applications. Organo-photoredox catalysts possess a broader redox window compared to their inorganic/organometallic counterparts. This characteristic could be crucial for achieving specific types of transformations or accessing substrates in particular reaction that are not attainable using metal-based systems [70-72]. The concept of photo-induced C-H/S-H functionalization can be employed for the oxidative coupling reactions.

1.4 Visible-light-induced oxidative coupling reactions

The selective creation of bonds between carbon-carbon or carbon-heteroatom centers stands as a primary challenge in synthetic organic chemistry. Cross-coupling reactions catalyzed by transition metal emerged as a robust method for enhancing molecular complexity during the last decades of the previous century [73]. This technique involves coupling two distinct moieties: one having a more electrophilic center (linked to an electronegative atom) and the other featuring a more nucleophilic center (linked to an electropositive atom).

However, traditional cross-coupling reactions between electrophiles and nucleophiles face two main limitations. The primary limitation includes the often necessary prefunctionalization of substrates. Also, electropositive atoms bonded to nucleophilic carbons, such as silicon, boron, zinc, or tin, are relatively scarce in organic chemistry. These atoms typically form byproducts that are challenging to separate. Consequently, the poor atom economy associated with traditional cross-coupling methods does not align well with the principles of "green chemistry," which seek to minimize environmental impact and promote sustainability in processes [74].

An alternative approach for overcoming these challenges utilizing the oxidative coupling of two nucleophiles has been discovered, where the utilization of hydrocarbons as nucleophiles is believed by many to be an optimal approach (Figure 1.5). These nucleophilic centers commonly feature hydrogen attachments, eliminating the necessity for prefunctionalization, while ensuring electro neutrality in the process typically mandates an external (or internal) oxidant [75-77].

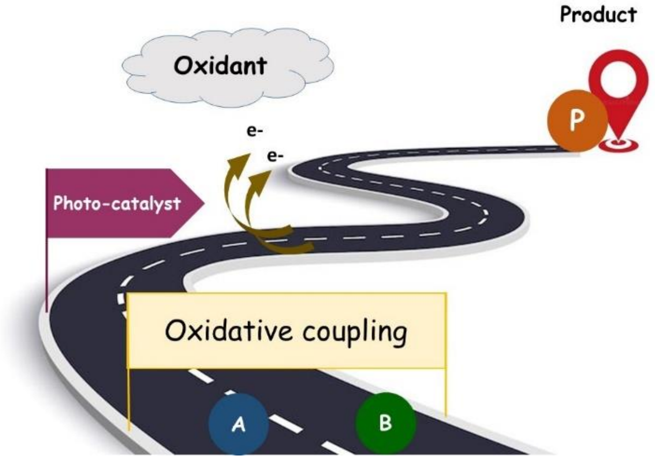


Figure 1.5 Photoinduced oxidative coupling reactions using an oxidant

Photoinduced catalysis has emerged as a technically appealing and energy-efficient platform for oxidative coupling reactions. Unlike cross-coupling reactions, oxidative coupling involves the oxidation of both reactants during the reaction, requiring an external oxidant to complete the catalytic cycle. The nature of the external oxidant shows a vital role in the reaction mechanism, with two important types identified: metal-based oxidants and metal-free oxidants (Figure 1.6).

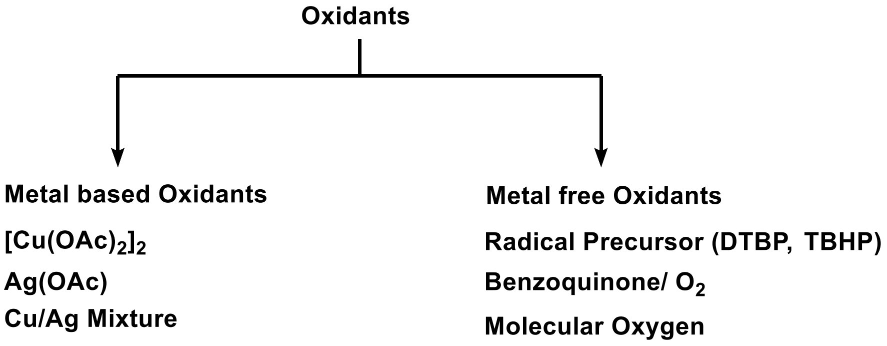
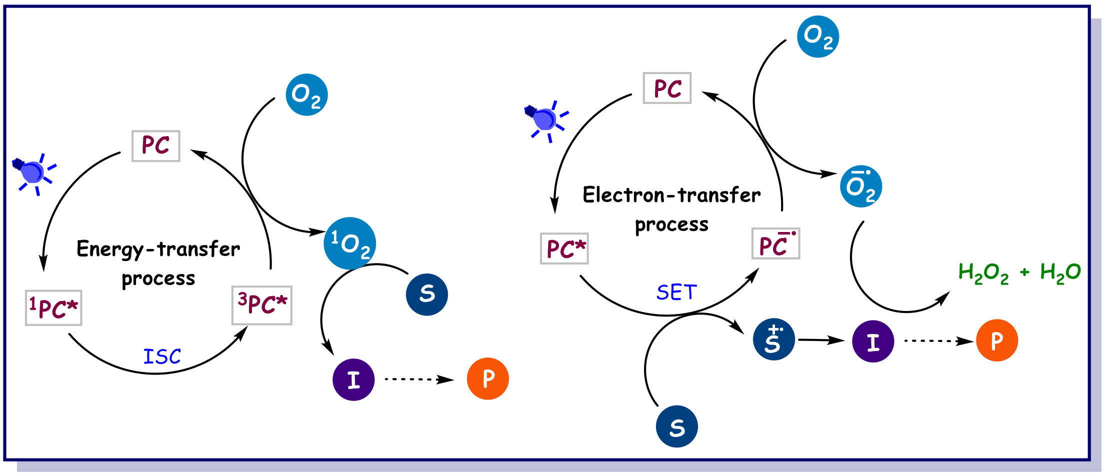


Figure 1.6 Types of oxidants for photochemical reactions

The requirement for an external oxidant is generally seen as a drawback, as the by-products formed by their reduction must be removed, except in cases where molecular oxygen can be utilized, generating only water as a by-product. Molecular oxygen stands out as a cost-effective, environmentally friendly, and abundant oxidant. With the assistance of a photoexcited catalyst, molecular oxygen can be excited to produce either singlet oxygen or superoxide radical anion through an energy transfer or electron-transfer process (Scheme 1.8). Both singlet oxygen and superoxide radical anion are reactive oxygen species (ROS) capable of readily reacting with the substrates or the active species generated during the reaction [14,78-81].



Scheme 1.9 Pathways depicting photoredox-catalyzed aerobic oxidation

While transition-metal catalyzed oxidative cross-coupling reactions have seen notable advancements, strategies rooted in visible-light-initiated photoredox catalysis have emerged as effective pathways for creating new chemical bonds between two nucleophilic centers in recent years [82-84]. The mild and environmentally benign features of visible-light-induced single electron transfer processes are making them increasingly popular for oxidative cross-coupling reactions (C-C, C-N, C-S, C-O, S-N, S-O coupling, etc.).

1.4.1 Photoredox catalyzed C-C coupling reactions

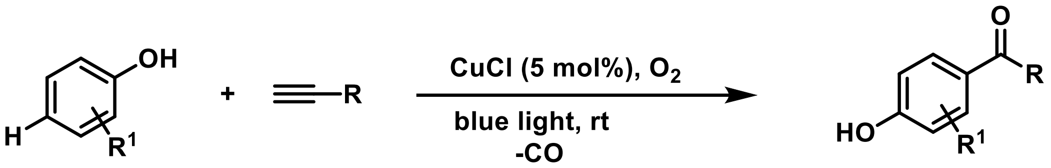
The essence of chemical synthesis lies in building chemical bonds, with carbon–carbon (C–C) bonds serving as crucial structural elements in numerous bioactive compounds, natural products, and functional materials. Therefore, the creation of these bonds stands as one of the fundamental processes in synthetic organic chemistry. Photoinduced C–C coupling reactions have gained prominence due to their remarkable ability to form carbon–carbon bonds, facilitating the efficient synthesis of complex molecules from basic starting materials. Thus, visible-light-initiated C-C coupling has emerged as an indispensable, economical, and eco-friendly tool in chemical synthesis [42,85-89].

Herein, we have summarized recent developments in visible-light-mediated aerobic oxidative C-C coupling reactions.

A new photosensitizer, remazol brilliant blue R (RBBR), was employed by Prof. Leow's group to develop a mild photocatalytic method for hydroperoxyarylation of styrenes using air as an oxidant [90].

Scheme 1.10 Hydroperoxyarylation of styrenes

Kuo Chu Hwang et.al. reported a visible-light-initiated direct oxidative C-C coupling of phenols and terminal alkynes utilizing copper as a photoredox catalyst. Using cleavage of the C-C triple bond, this technique enables the regioselective synthesis of functionalized aryl and alkyl ketones [91].

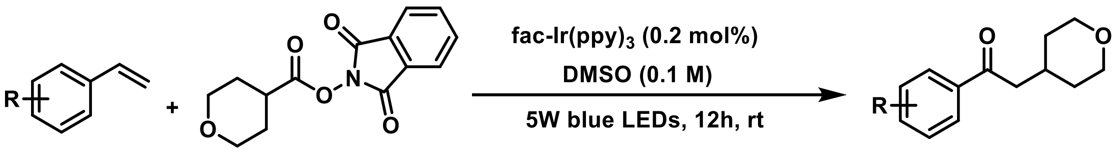


Scheme 1.11 Regioselective C-C coupling of phenols and terminal alkynes

A metal-free and environmentally friendly method has been devised by the Qiu-Yun Chen group for converting alkenes to ketones. In this methodology, alkenes are added oxidatively to aryl radicals through the aerobic oxidation of arylhydrazines induced by visible light [92].

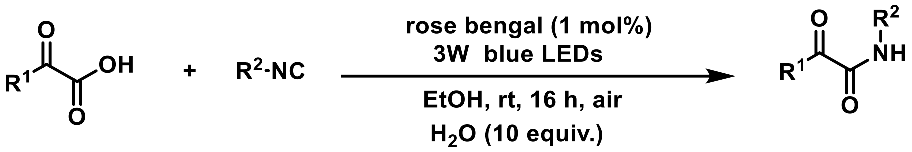
Scheme 1.12 Metal-free photoredox catalyzed conversion of alkenes to ketones

Frank Glorius and group in 2018 reported a photoredox catalyzed method for the oxidative coupling of easily available styrene with an alkyl radical to access α-alkyl-acetophenones under mild conditions using DMSO as a nontoxic oxidant [93].



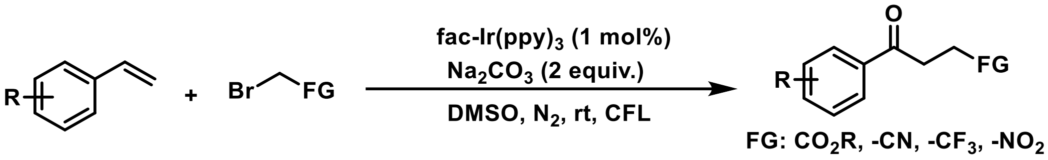
Scheme 1.13 Oxidative coupling of styrene with an alkyl radical

Wei and his group reported a photoinduced approach for the synthesis of α-ketoamides by decarboxylative acylation of α-oxocarboxylic acids with isocyanides using rose bengal as a photocatalyst in water. The current conversion was accomplished in a gentle and metal-free condition via a series of decarboxylation, radical addition, and hydration procedures, offering a new and easy method to create α-ketoamides from basic and easily accessible chemical feedstocks [94].



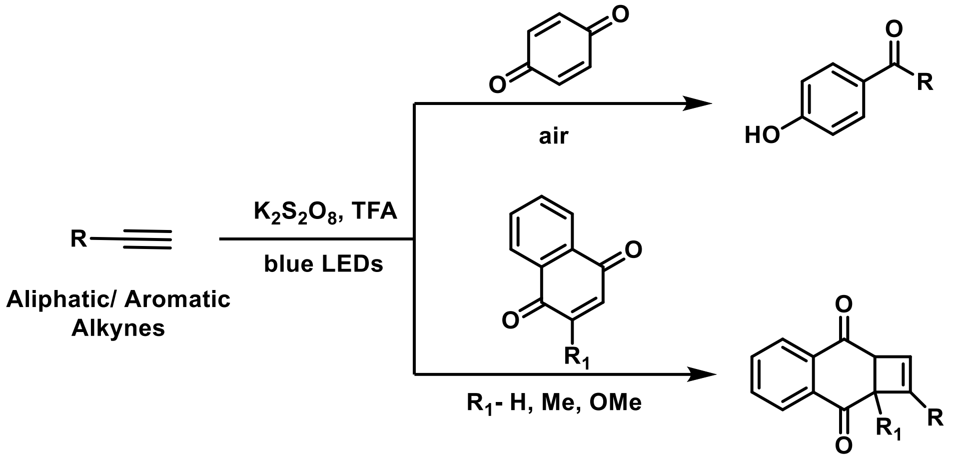
Scheme 1.14 Synthesis of α-ketoamides using rose bengal as a photocatalyst

Gong-Qing Liu and group developed a photocatalyzed method for coupling vinylarenes with bromocarboxylates for the synthesis of γ-ketoesters utilizing DMSO as solvent as well as oxidant [95].



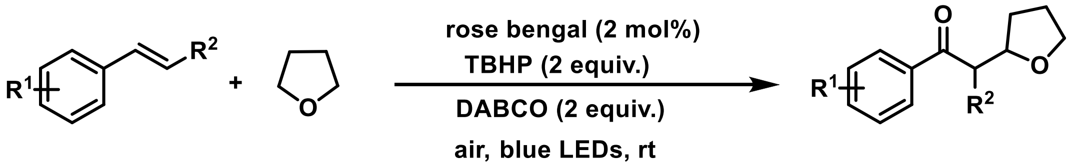
Scheme 1.15 Coupling vinylarenes with bromocarboxylates

Bhahwal Ali Shah and group developed a photoredox catalyzed, single-step [2+2] cycloaddition reaction for the synthesis of aryl/alkyl and bi-aryl ketones via aerobic oxidative coupling of terminal alkynes with benzoquinones. Furthermore, the alkene-alkyne cycloaddition is more advantageous with naphthoquinones because of the crossresonance between the aromatic ring and the carbonyl group, which leads to the creation of four-membered carbocyclic adducts [96].



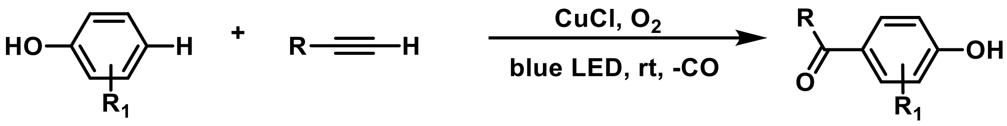
Scheme 1.16 Single-step [2 + 2] cycloaddition reaction

Alakananda Hajra and group reported a visible-light-induced oxidative coupling of vinylarenes with cyclic ethers for the synthesis of α-oxyalkylated ketones using TBHP as an oxidant and rose bengal as a photocatalyst [97].



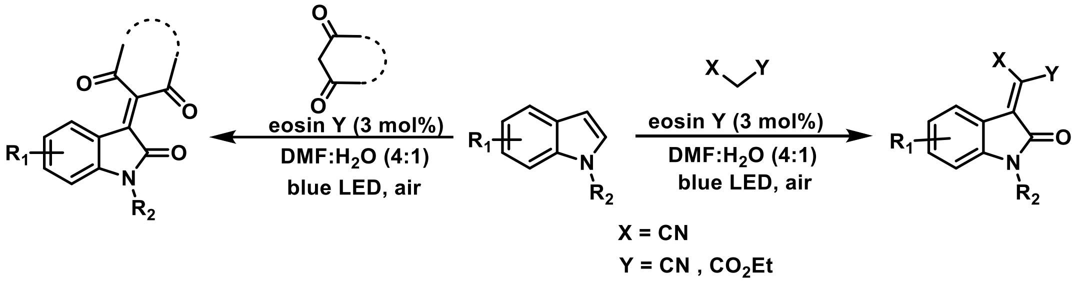
Scheme 1.17 Visible-light-induced oxidative coupling of vinylarenes

Ganglong Cui and Walter Thiel reported a photoredox catalyzed synthesis of hydroxyl-functionalized aryl ketones via aerobic oxidative C-C coupling of alkynes and phenols. Under mild conditions, this single-step regioselective reaction occurs using the straightforward and cost-effective CuCl catalyst, without the need for external oxidants and bases [98].



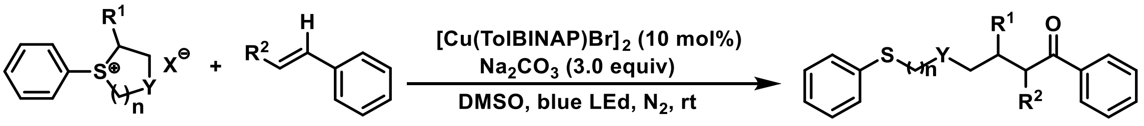
Scheme 1.18 Aerobic oxidative C-C coupling of alkynes and phenols

In 2022 Singh and co-worker, developed a metal-free photoredox-catalyzed method for the synthesis of 3-methyleneindolin-2-ones via oxidative coupling of indoles and active methylene compounds using eosin Y as an organophotocatalyst. Their methodology uses ambient air as an oxidant and also dearomatization of indoles is involved[99].



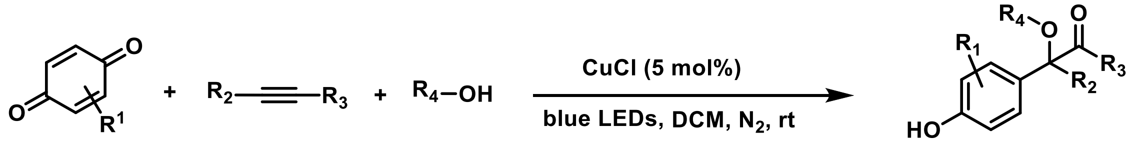
Scheme 1.19 Oxidative coupling of indoles and active methylene compounds

Daoshan Yang et.al. accomplished a photoredox process for the direct coupling of sulfonium salts with alkenes, employing copper (I) complex as a photosensitizer for the formation of α-alkyl-acetophenones. Dimethyl sulfoxide (DMSO), a moderate oxidant, is used in this procedure to selectively cleave the C–S bonds in sulfonium salts [100].



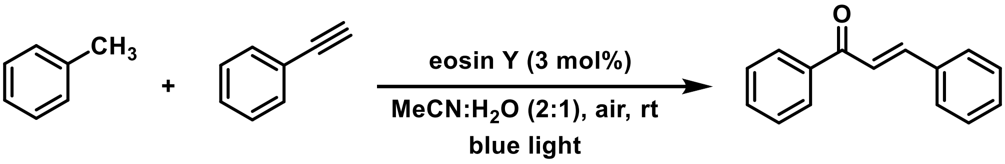
Scheme 1.20 Direct coupling of sulfonium salts with alkenes

Professors Mu-Jeng Cheng and Kuo Chu Hwang employed CuCl as a photocatalyst to synthesize highly functionalized ketones at room temperature. This synthesis involved the use of benzoquinone, alkyl/aryl alcohol, and alkyl/aryl terminal/internal alkynes [101].



Scheme 1.21 Photocatalyzed synthesis of highly functionalized ketones

Singh et.al. reported an atom-efficient photochemical synthetic pathway for oxidative C-C coupling to form α,β-unsaturated ketones via hydroacylation of aromatic alkynes. With the utilization of eosin Y as a metal-free photocatalyst and air as an oxidant, this methodology is compatible for the synthesis of biological scaffolds [102].



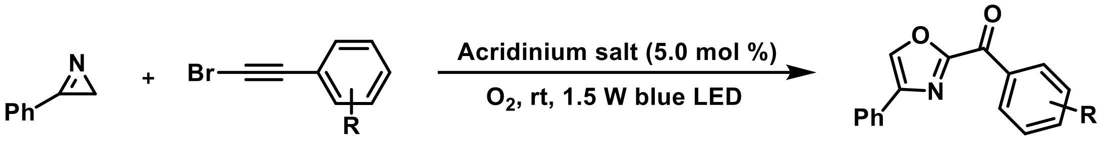
Scheme 1.22 Oxidative C-C coupling to form α,β-unsaturated ketones

1.4.2 Visible-light-mediated C-N coupling reactions

In synthetic chemistry, there is a continued effort to develop effectual and selective methods for easily forming carbon–nitrogen bonds due to their prevalence in natural products, functional materials, and pharmaceutical agents. A promising approach in achieving direct C–H amination involves employing photoredox catalysis. When subjected to photoirradiation, the photocatalyst aids in generating organoradical species through a single electron transfer (SET) pathway. These radicals can subsequently interact with various coupling partners to produce aminated products. Photocatalytic C–N bond formation includes oxidant-free amination and oxidative amination, typically initiated by the formation of a nitrogen radical cation, an aryl radical cation, or a nitrogen radical [103-108].

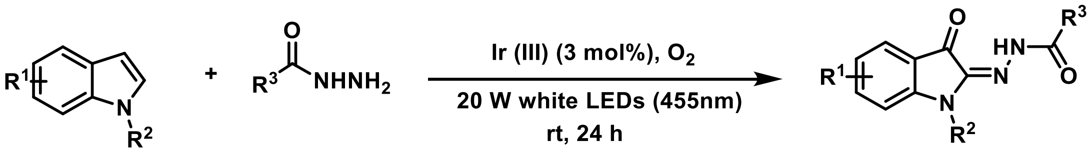
Considering the importance of C-N bonds, herein we have provided some recent advances in oxidative C-N coupling reactions mediated by photocatalysis.

Wang et.al. developed a synthetic path for the visible-light-induced synthesis of substituted oxazoles via coupling of 2H-azirines, alkynyl bromides, and molecular oxygen at room temperature [109].



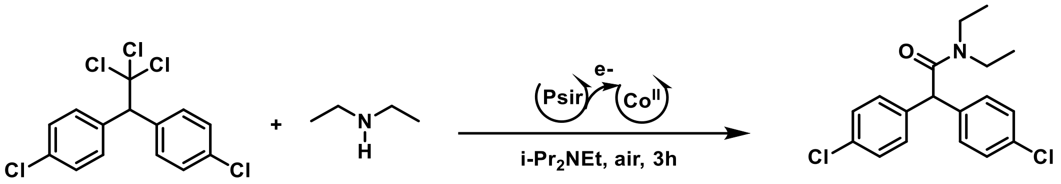
Scheme 1.23 Visible-light-induced synthesis of substituted oxazoles

Bhisma K. Patel and group described a visible-light-mediated technique that selectively difunctionalizes N-substituted indoles utilizing benzohydrazides in an oxygen environment, causing indoles to simultaneously undergo C-3 oxidation and C-2 amination [110].



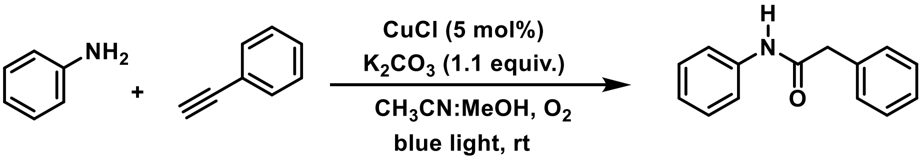
Scheme 1.24 Oxidative coupling of N-substituted indoles with benzohydrazides

Yoshio et.al. presented an easy, straightforward, and very effective method for the synthesis of visible-light-driven amides from trichlorinated organic compounds using the B12 complex, hepta-methyl cobyrinate perchlorate, as the catalyst and $[Ir(dtbbpy)(ppy)_2]PF_6$ as the photosensitizer that absorbs visible light [111].



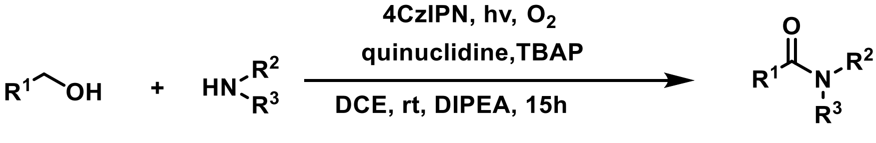
Scheme 1.25 Visible-light-driven synthesis of amides

Kuo Chu Hwang's group presented a regioselective methodology for the synthesis of acetamide catalyzed by photoredox copper catalyst, achieved through the C–N cross-coupling of amines with terminal alkynes. This reaction utilizes oxygen (O_2) as an oxidant under visible-light-irradiation at room temperature [112].



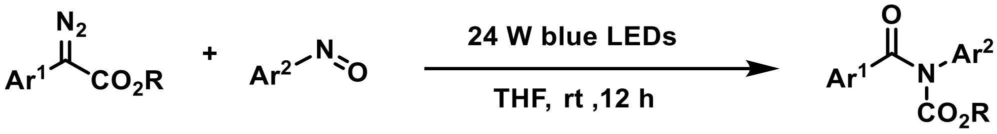
Scheme 1.26 C–N cross-coupling of amines with terminal alkynes

Professor N. D. Pradeep Singh and his research group reported an aerobic oxidative cross-coupling reaction for synthesizing amides. They utilized a combination of organic photocatalyst quinuclidine (HAT catalyst) and tetra-n-butylammonium phosphate (TBAP, hydrogen-bonding catalyst) for the direct coupling of alcohols and amines [113].



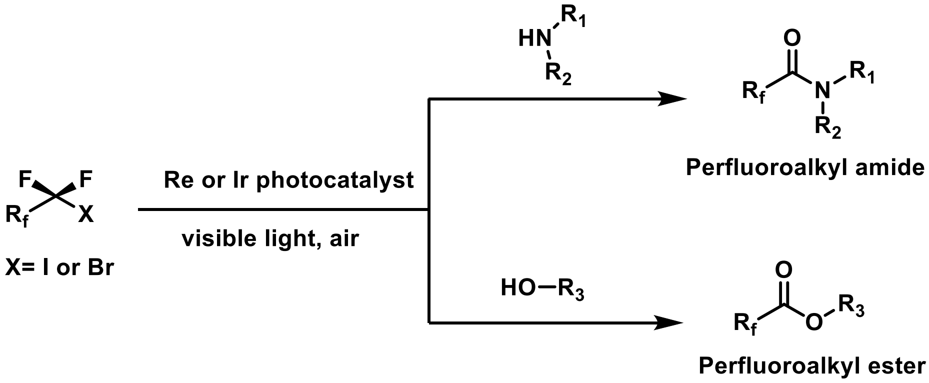
Scheme 1.27 Aerobic oxidative cross-coupling reaction for synthesizing amides

Wen-Jing Xiao and the group developed a catalyst-free and visible-light-mediated sustainable strategy for the synthesis of amide bonds via in-situ nitrone formation [114].



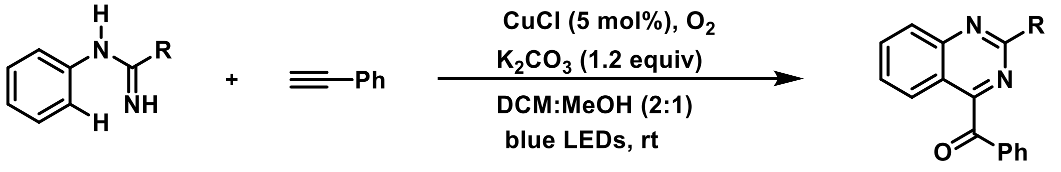
Scheme 1.28 Visible-light-mediated nitrone formation

Chi-Chiu Ko's group explored visible-light-induced photocatalytic reactions to synthesize perfluoroalkyl amides and esters from their respective amines and alcohols, employing various triplet emitters as photocatalysts via generation of perfluoroalkyl radicals [115].



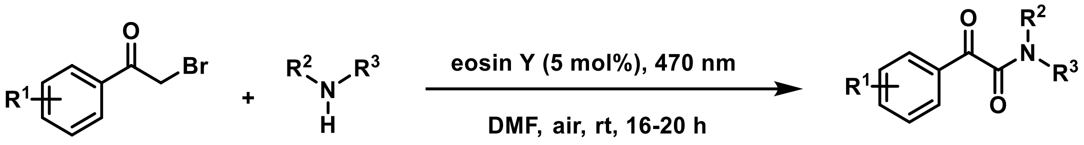
Scheme 1.29 Photoredox catalyzed C-N/C-O coupling

Kuo Chu Hwang and group have presented a protocol employing visible light-induced photo-redox copper catalysis to facilitate the oxidative C(sp^2)–H annulation (Friedel–Crafts-type cyclization) of amidines with terminal alkynes at room temperature, resulting in the formation of functionalized quinazolines. This investigation emphasizes the photo-oxidative C(sp^2)–H annulation of amidines at room temperature catalyzed by copper(I)-phenylacetylide [116].



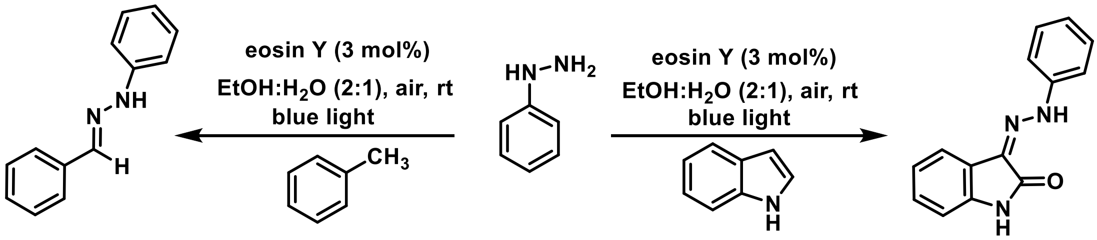
Scheme 1.30 Visible-light-induced oxidative Csp^2–H annulation

Prof. Dr. Krishna Nand Singh and the group developed a methodology for the visible-light-initiated oxidative C-N cross-coupling reaction for the amination of 2-bromoacetophenones to afford α-ketoamides using eosin Y as organophotocatalyst under ambient air [117].



Scheme 1.31 Oxidative C-N cross-coupling reaction to afford α-ketoamides

Singh and group reported a protocol for the synthesis of hydrazones via oxidative cross-coupling of phenylhydrazines with activated indoles and methylarenes under visible light irradiation using ethanol: water as the solvent and eosin Y as photocatalyst [118].

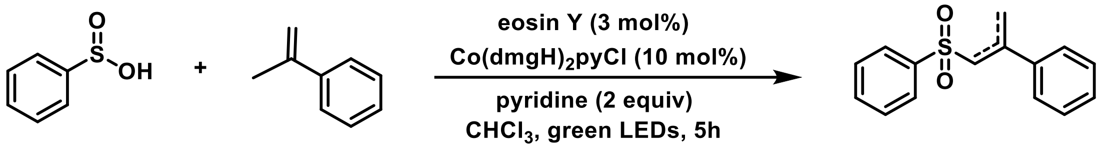


Scheme 1.32 Synthesis of hydrazones via oxidative cross-coupling

1.4.3 Visible-light-initiated C-S/ C-O coupling reactions

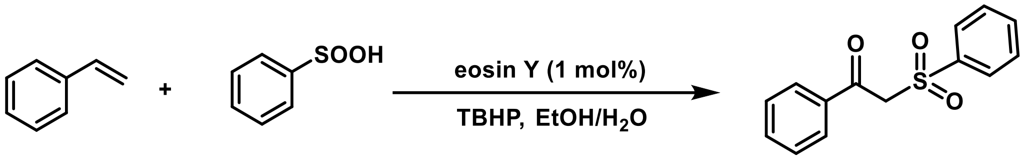
The synthesis of C–S bonds holds significant synthetic importance due to the abundance of sulfur-containing natural products, drugs, and agrochemicals, as well as the growing relevance of sulfur-containing compounds in material and polymer chemistry. Organic compounds containing sulfur are frequently utilized in many different areas of chemistry. Due to the growing number of authorized organosulfur-based medications, numerous investigations have focused on incorporating sulfur functional groups into organic compounds. Photoredox catalysis has garnered considerable interest in building structurally diverse and intricate organic frameworks due to its inherent attributes such as high efficiency, environmental friendliness, sustainability, renewability, cost-effectiveness, and ease of use. In pharmaceuticals, functionalized materials, and natural compounds, C–O linkages are common. Additionally, ethers, esters, and phenols are just a few of the numerous molecules with carbon–oxygen linkages that are produced on a large scale and serve as essential components in organic synthesis [105,119-124]. Considering these foundations, we have opted to outline the progress made in developing protocols for visible-light-induced oxidative coupling to synthesize C−S bonds.

Aiwen Lei and group reported a method for the visible-light-induced C-S coupling for dehydrogenative sulfonylation of α-methyl-styrene derivatives to produce allylic sulfones, employing eosin Y as a photosensitizer along with a cobaloxime as a catalyst without the use of oxidant. This process utilizes a cost-effective metal catalyst and ensures atom economy, offering an attractive strategy for upcoming synthetic chemistry endeavors [125].



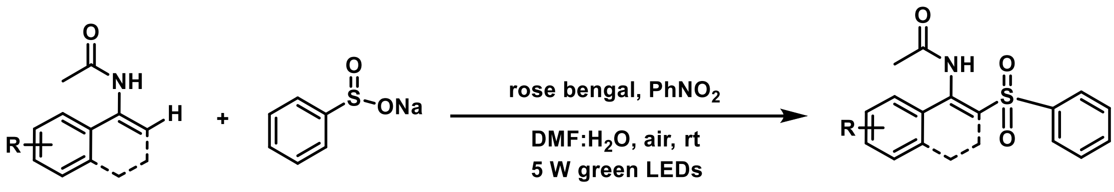
Scheme 1.33 Visible-light-induced C-S coupling via dehydrogenative sulfonylation

Hua Wang's group reported a transition metal-free approach for the synthesis of β-ketosulfones via direct oxysulfonylation of styrenes with sulfinic acids using eosin Y as an organic photoredox catalyst [126].



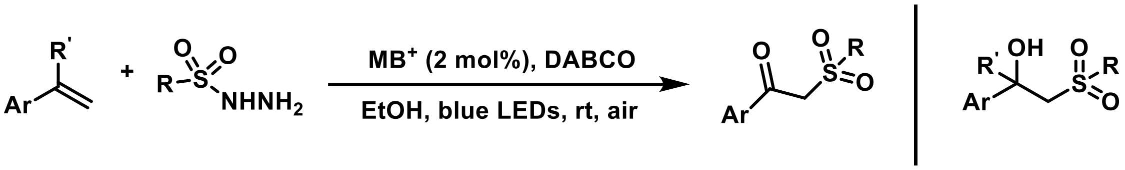
Scheme 1.34 Metal-free approach for the synthesis of β-ketosulfones

Ronghua Zhang reported a transition metal-free protocol for the synthesis of β-acetylamino acrylosulfones from sodium sulfinates and enamides under visible light irradiation using rose bengal as an organic photoredox catalyst [127].



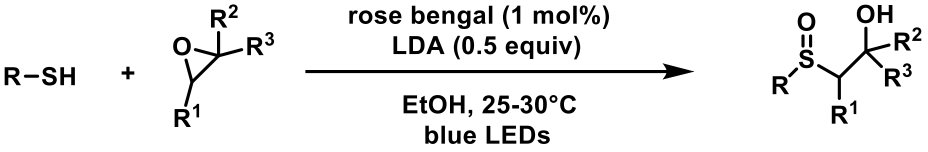
Scheme 1.35 Photocatalyzed C-S coupling

Chunyin Zhu and group proposed a method for metal-free photoinduced C-S oxidative coupling of alkenes with sulfonyl hydrazides to generate β-keto/hydroxyl sulfones using O_2 as oxidant [128].



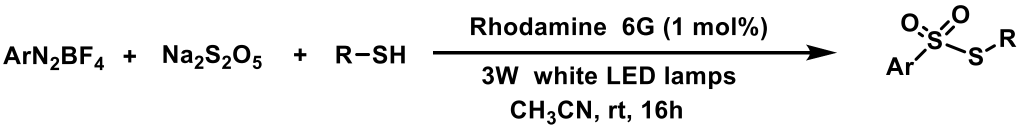
Scheme 1.36 Metal-free photoinduced C-S oxidative coupling of alkenes

In 2021, Ablimit Abdukader et al. described a visible-light-mediated coupling reaction between epoxides and thiols to produce β-hydroxylsulfoxides, employing rose bengal as a metal-free photocatalyst and LDA as a base [129].



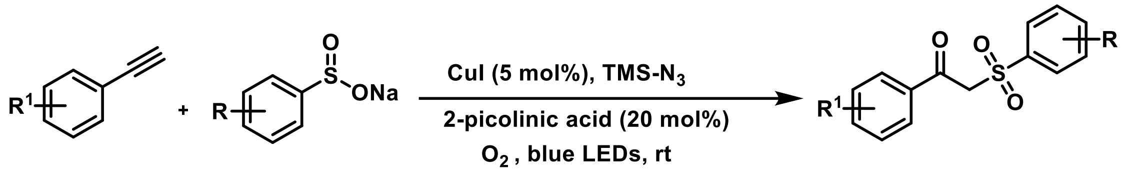
Scheme 1.37 Visible-light-mediated coupling reaction between epoxides and thiols

Lin He and colleagues documented a photoredox-catalyzed approach for synthesizing thiosulfonates, employing rhodamine 6G as a metal-free photocatalyst. Their methodology involves the sulfonylation of thiols with aryldiazonium, utilizing sodium metabisulfite as a surrogate for SO_2 [130].



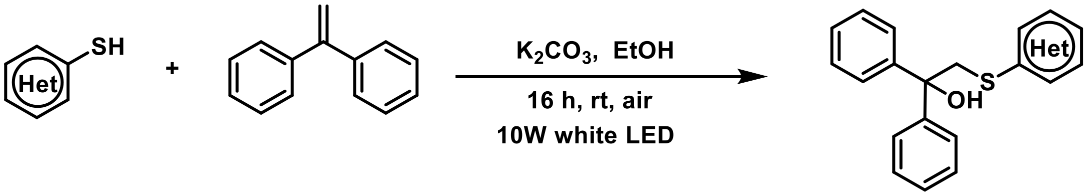
Scheme 1.38 Photoredox-catalyzed approach for synthesizing thiosulfonates

Kuo Chu Hwang and group for the first time, succeeded in forming a C-S bond utilizing photoredox trimethylsilyl azide (TMS-N_3)-assisted copper-catalyst via oxy-sulfonylation of terminal alkynes to form β-keto sulfones [131].



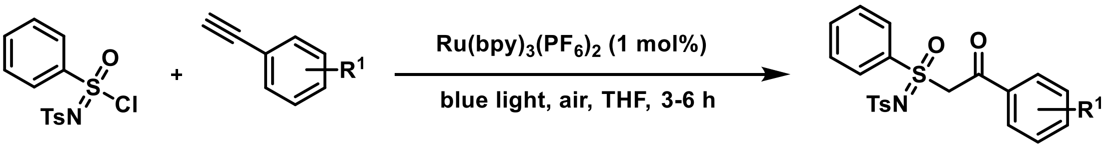
Scheme 1.39 C-S coupling via oxy-sulfonylation of terminal alkynes

Chenjiang Liu and group introduced a novel approach for the visible-light-induced hydroxysulfenylation of styrenes to synthesize β-hydroxysulfides. This method, facilitated by an electron-donor-acceptor (EDA) complex, utilizes ambient air as an oxidant and does not necessitate the use of external photocatalysts or transition metals [132].



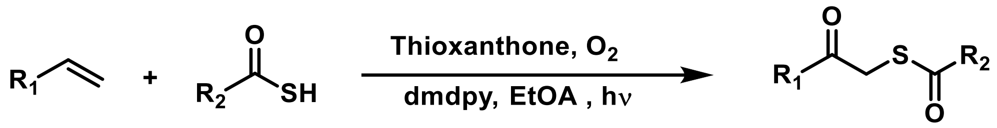
Scheme 1.40 Synthesis of β-hydroxysulfides *via* EDA complex

Carsten Bolm and group proposed a visible-light-induced, ruthenium-catalyzed oxidative coupling of N-tosyl-protected sulfoximidoyl chlorides with aryl alkynes to afford β-keto sulfoximines employing air as oxidant [133].



Scheme 1.41 Oxidative coupling of sulfoximidoyl chlorides with aryl alkynes

Zhankui Sun and colleagues devised a photoinduced approach to generate α-ketothiol esters through radical addition of thionic acids to alkenes. This method employs thioxanthones as a metal-free photocatalyst and O_2 as an oxidant [134].



Scheme 1.42 Photo induced radical addition of thionic acids to alkenes

Qiang Liu and group developed an atom economical and visible-light-induced method for the synthesis of benzoin bis-ethers by coupling alkynes and alcohols using organophotocatalyst, Mes-Acr$^+$ ClO$_4^-$, and air as oxidant [135].

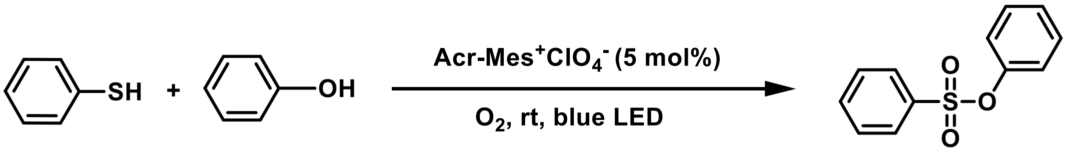
Scheme 1.43 Coupling of alkynes and alcohols in blue light

1.4.4 Visible-light-initiated S-N/S-O Coupling reactions

Chemists have shown considerable interest in organosulfur compounds due to their significance in natural products, pesticides, organic synthesis, and medicinal chemistry. Compounds containing S-N/S-O bonds are particularly noteworthy as they serve as essential components in various biologically active compounds. The remarkable biological activity displayed by their reaction products, such as sulfonamides, isothiazoles, and thiadiazoles, including antibacterial, anti-inflammatory, and insecticidal properties, has led to a renewed focus on S–N/S-O coupling mechanisms in recent times. Oxidative approaches have emerged as an effective tool in organic reactions for generating heteroatom–heteroatom bonds. Among these strategies, photoredox-catalyzed oxidative S–N/S-O bond formation reactions have garnered significant attention as they offer provide practical, effective, and eco-friendly substitutes for the existing procedures for synthesizing a variety of organic compounds commonly employed in organic chemistry. These changes occur via the combination of S- and N-/O- reagents with an oxidant, employing radical or ionic mechanisms [136-141].

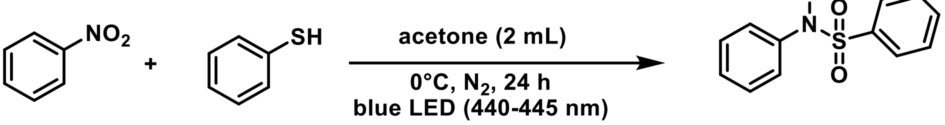
In view of these findings, we have chosen to summarize the developments in creating protocols for the oxidative coupling triggered by visible light to synthesize S–N/S-O bonds.

Aiwen Lei et.al. proposed a visible-light-mediated oxidative S-O cross-coupling method for synthesizing alkyl benzenesulfonates. This technique utilizes Mes-Acr$^+$ ClO_4- as an organophotocatalyst and O_2 as an oxidant, without the need for any ligand co-additives [142].



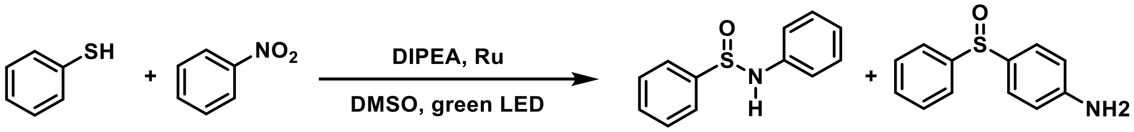
Scheme 1.44 Visible-light-mediated oxidative S-O cross-coupling

Yonggui Robin Chi and his team developed a photoinduced protocol for sulfonamide synthesis by direct coupling of nitro compounds and thiols without the need for any photocatalyst. This protocol facilitates direct oxygen atom transfer from the nitro group to the thiol [143].



Scheme 1.45 Visible-light-induced oxidant free synthesis of sulfonamides

Jianyu Cao et.al. reported a photoinduced method for synthesizing sulfinamides and sulfoxides via the coupling of nitroarenes and thiophenols. The formation of sulfinamides and sulfoxides depends on the type of photocatalyst and the amount of thiophenol [144].



Scheme 1.46 Photoinduced method for synthesizing sulfinamides and sulfoxides

In view of the importance of photo-induced C-H/S-H functionalization for the oxidative coupling reactions and visible-light as a renewable, sustainable, and benign source of energy, our interest is to explore the chemistry (synthesis and structural characterization) of hydrazones, C-C unsaturated bond formation, chalcones, and sulfonamides under visible-light irradiation. The studies have been described in subsequent **chapters 2-5**.

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