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REVIEW

A Useful Methods of Direct Sulfenylation at the 3-Position of Indoles Employing various Sulfenylating Agents

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3-Sulfenylated indoles as a significant heterocyclic scaffold have garnered enormous interest and utility in the past several decades due to significant biological and pharmaceutical activities, including anti-HIV, anti-obesity and anti-cancer activity. To date, several methods have been developed for the synthesis of 3-sulfenylated indoles using distinct sulfenylating agents. In this review, the recent developments in the metal free synthesis of 3-sulfenylated indoles moieties are covered, which show insight into both the large distinct reactions condition as well as mechanism.

Keywords: Indole, Sulfenylation, Sulfenylating agents, 3-Sulfenylated indoles.

INTRODUCTION

The formation of carbon-heteroatom bond plays a significant role in organic synthesis that has extensive applications in pharmaceutical chemistry for drug discovery, resulting to enhance the demand for generating new methodologies for such transformation [1,2]. Amid all, the formation of the carbon-sulfur (C-S) bond has a remarkable place for the production of efficient chemical entities, as the organosulfur attached compounds belong to a superior class of organic compounds, found in distinct biologically active natural products, pharmaceuticals and agrochemicals [3-6]. The indole thioethers containing organosulfur derivatives (indole-S-R) are one of the peculiar moieties in the medicinal chemistry as well as for pharmaceutical fields. Specifically, 3-sulfenylindole substrates hold prominent pharmacological potential including anti-viral (HIV-1) [7], anti-allergic [8] and anti-cancer [9], *etc*. as well as potential drug candidates for drug discovery programs [10-12] (Fig. 1).

To explore the more comprehensive biological roles and synthetic applications of 3-sulfenylindoles derivatives, the evaluation of novel, effective and practical methods to furnish such compounds, from simple, easily and readily available starting materials is still undiscovered and desirable.

During the past decades, the formation of modified S-containing aryl donors (*e.g*. aryl halides, aryl boronic acids, etc.) using transition metal catalyzed Ullmann condensation have been explored excessively and are enlightened as to the most potent and reliable strategies for the generation of di-(hetero)aryl thioethers [13]. Also, different synthetic methods have been established to construct 3-sulfenylindole scaffolds. In this context, the derivatives of 3-sulfenylindole could be synthesized by cyclization reactions of phenylhydrazine hydrochloride [14], 2-alkynylanilines [15], *N*,*N*-dialkyl-2-iodoanilines *via* one-pot reaction [16,17]. Howbeit, the most eminent protocol to synthesize the 3-sulfenylindole is the electrophilic substitution of preexisting indole rings promoted by different electrophilic sulfenylating agents like thiols, disulfides, sulfonium salts, arylsulfonyl hydrazides, sulfinates, *N*-thioamides, quinone mono-O, S-acetals, sulfenyl halides, arylsulfonyl chloride, *etc*. (**Scheme-I**).

In virtue of the previous attainment and the advantage of thioethers, herein, we review the direct sulfenylation of indoles with different electrophilic sulfenylating agents, leading to 3-sulfenylindoles scaffolds.

Thiols sulfenylating agent: Accordingly, the sulfenylation reaction of indoles using common sulfenylating agent

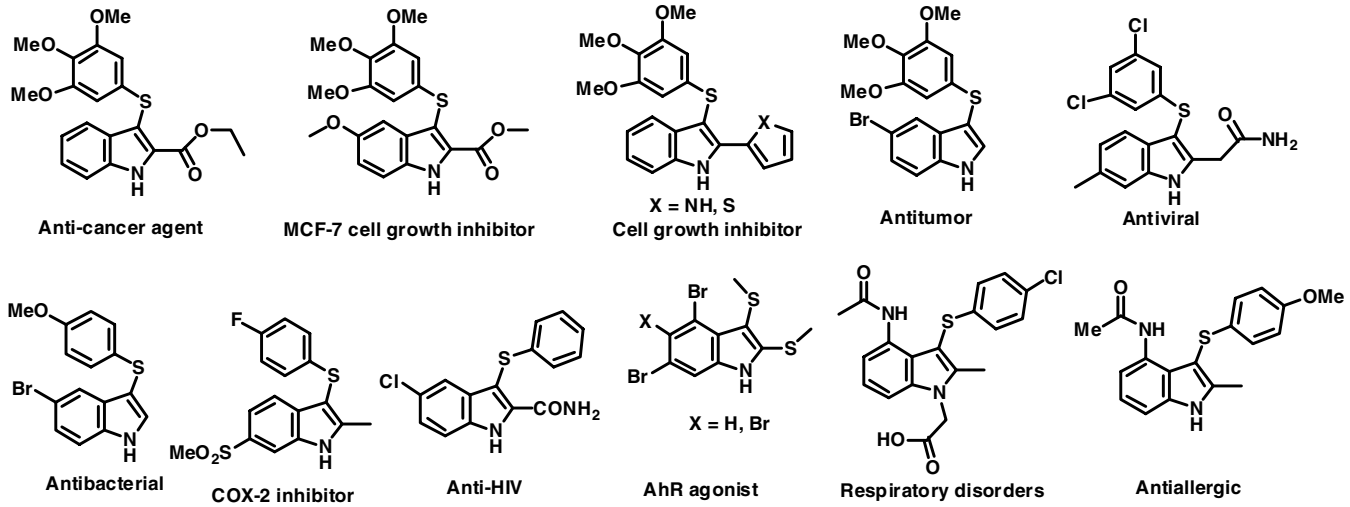
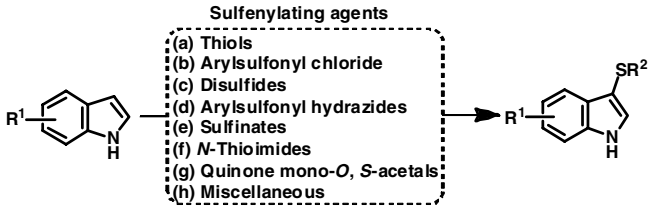
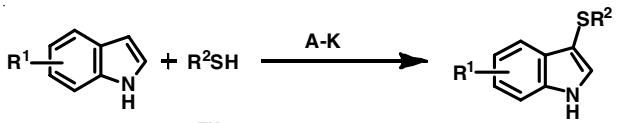


Fig. 1. Some biologically active 3-sulfenylindole derived molecules



Scheme-I: Electrophilic substitution of indole rings promoted by different sulfenylating agents

e.g. thiophenols has been most successful. In this context, various oxidative coupling conditions have been developed for sulfeny-lation of indoles in **Scheme-II**.



A: Selectfluor™, CH_3CN, room temp.
B: NaOH, DMSO, 70 °C
C: I_2 (5 mol%), CH_3CN, 80 °C, Air
D: I_2 (10 mol%), TBHP (1.05 equiv.), CH_3CN, 60 °C
E: BSA (0.3 mol%), I_2 (30 mol%), TBAB (10 mol%), H_2O, 50 °C, O_2 baloon
F: Cs_2CO_3 (20 mol%), DMSO,100 °C, Air,
G: KIO_3 (40 mol%), DMSO/H_2O (1:3), 100 °C
H: Flavin (2 mol%), I_2 (2 mol%), O_2 (1 atm), CH_3CN, 25 °C
I: NO_2BF_4 (10 mol%), air, HFIP, room temp.
J: Graphene oxide (GO), H_2O, 40 °C
K: Electrolytic conditions
In case of heterocyclic thiols
TFA/DMSO, or oxone, or KIO_3

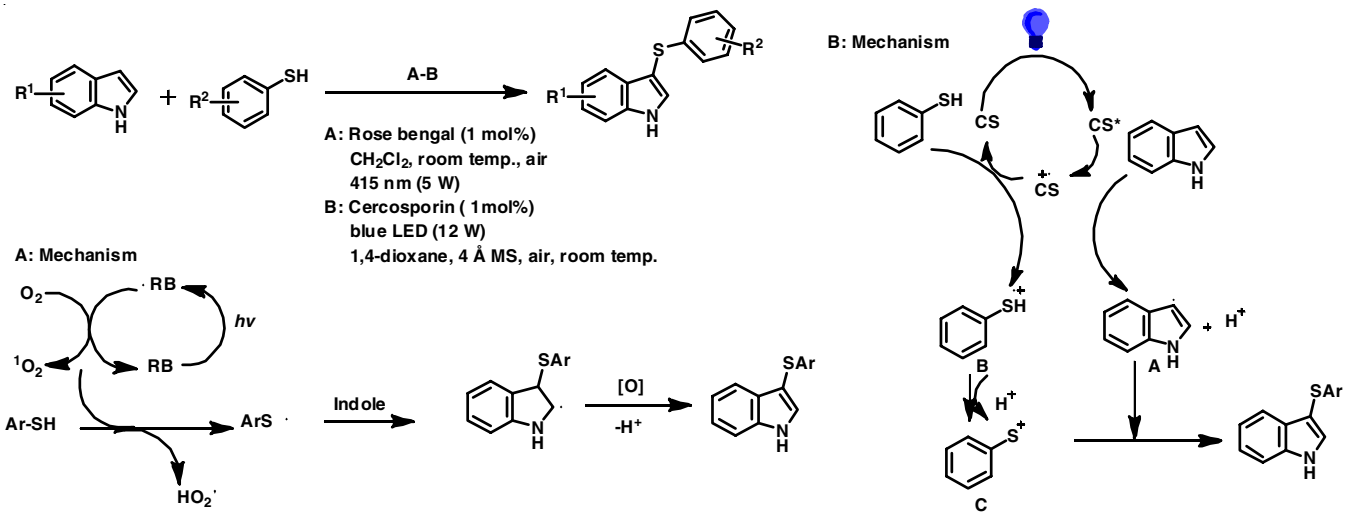
Scheme-II: Sulfenylation of indoles with thiol

Yadav *et al*. [18] reported the direct 3-thiolation of indoles with aromatic thiols in the presence of Selectfluor™ in higher yields as well as selectivity. This reaction is successful even with indoles having no substitution in the 2nd position. In 2014, Wen *et al*. [19] synthesized 3-sulfenylated indoles using NaOH as a promoter from indoles and thiols. Qu and co-workers [20] developed a method to furnish mono- and *bis*-arylthioindoles through direct I_2 catalyzed oxidative sulfenylation of indoles using thiophenols. To further expand this work, Wang *et al*. [21] further demonstrate the construction of 3-thioindoles regioselectively similar I_2 catalyst to perform direct thiolation of indole with thiols.

Sinha *et al*. [22] thereafter reported the sulfenylation of C(sp2)-H of indole from thiophenol using bovine serum albumin (BSA)-iodine together as a catalyst through *in situ* cleavage of disulfide (S-S bond) under aqueous medium, as individually BSA or I_2 isn't capable to form C-S bond in the same pot. Jiao *et al*. [23] reported the Cs_2CO_3-catalyzed aerobic oxidative cross-dehydrogenative coupling of thiols with both phosphonates as well as arenes to generate thiophosphates and sulfenylarenes respectively in presence of air and DMSO.

Further, Liu *et al*. [24] in 2018 performed the C-H sulfenylation of indole catalyzed by simple inorganic iodine salt KIO_3 as the sole catalyst to give 3-sulfenylated indoles in DMSO/H_2O at 100 ºC. To further evaluate an aerobic indole sulfenylation, Iida *et al*. [25] in 2018 reported a redox organocatalysis system using together flavin-iodine as a catalyst and thiol as the thiolating agents to furnish the metal-free aerobic oxidative direct sulfenylation of indoles at ambient temperature. Where, flavin was supposed to perform a key role by combining with oxygen atmosphere to convert the iodine anion into catalytically active elemental iodine and also transforming thiols into disulfides that may not possible under such conditions in absence of it. Also, the environmentally friendly the sulfenylindole was developed by oxidative coupling conditions such as graphene oxide/O_2 [26] or electrolytic conditions [27]. However, the use of heterocyclic thiols for direct sulfenylation reaction of indoles was reported few methods using various strong oxidants such as TFA/DMSO [28], oxone [29], KIO_3 [30].

In 2017, Chen & Fan [31] discovered an adequate method to synthesize 3-sulfenylated indoles using sulfenylated agent thiophenols *via* visible light-induced photocatalysis and Rose Bengal as the photosensitizer (**Scheme-III**). Based on the experimental outcome, this processing pathway contains singlet oxygen produced by energy transfer from excited Rose Bengal

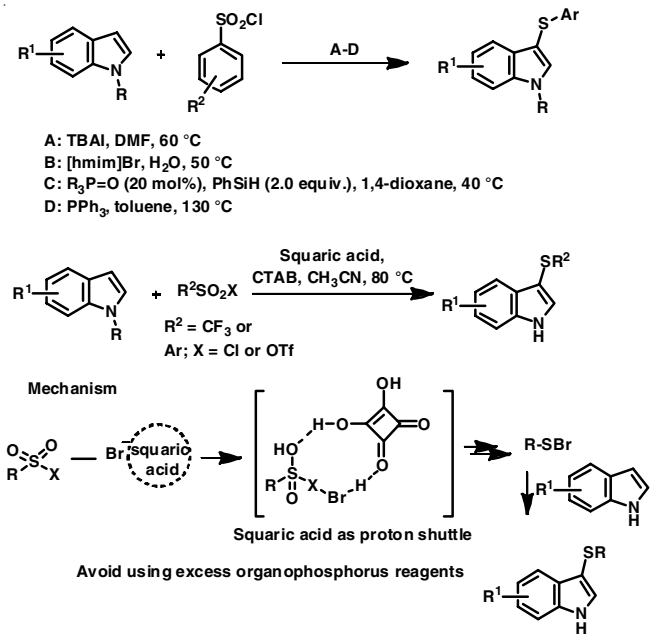


Scheme-III: Visible light-induced 3-sulfenylation of indoles

was suggested. Recently, Hazarika & Barman *et al*. [32] also reported the direct sulfenylation of indoles with thiols through visible-light-induced photoredox catalysis using cercosporin. Firstly, CS* that formed under the visible-light irradiation of the blue LED lamp, acts as a strong reductant to convert indole into radical intermediate A and cercosporin radical cation $CS^{\bullet+}$ (a powerful oxidant), which further oxidizes thiophenol to give the thiol radical cation B and form cercosporin CS simultaneously. This cation B then undergoes deprotection to form the stabilized radical C, which finally reacts with the radical intermediate A to furnish 3-(phenylthio)-1*H*-indole (**Scheme-III**).

Sulfonyl chlorides as sulfonylating agent: Xiang *et al*. [33] used sulfonyl chlorides as sulfonylating agents to perform sulfenylation of indoles promoted *via* TBAI to generate 3-sulfenylated indoles in one pot with good yield and high regioselectivity. Later in 2019, Sinha *et al*. [34] evaluated a step-economical and temperature tunable protocol for the sulfenylation of indole by using [Hmim]Br-$ArSO_2Cl$ whose synergistic effect promotes the synthesis of 3-arylthio indole at room temperature exclusively. Howbeit, they found that a similar reaction on enhancing temperature up to 50 ºC afforded an unpredictable 2-halo-3-arylthio indole with the formation of C-S and C-S/C-X (X = Cl and Br) bonds without any additional external halogenating agent *via* cascade sulfenylation halogenation reactions in the presence of metal-oxidant-base-free conditions (**Scheme-IV**).

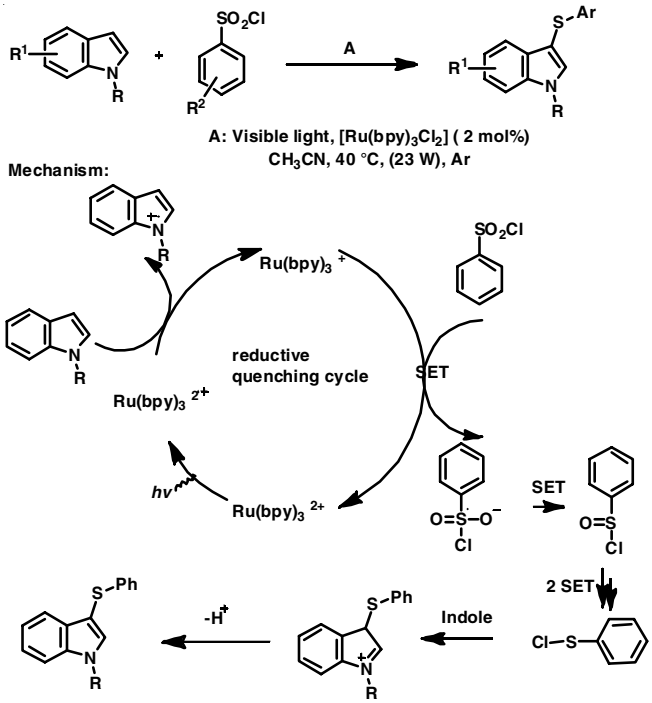
Radosevich *et al*. [35] demonstrated an organophosphorus-catalyzed strategy for electrophilic sulfenylation from sulfonyl chlorides *via* deoxygenating O-atom transfer. This process involves C-S bond formation promoted by an easily accessible phosphetane in conjunction with a hydrosilane terminal reductant to afford a general entry to sulfenyl electrophiles. The mechanistic study showed that the two-fold sulfonyl substrate deoxygenation progress by the arbitration of an off-cycle drowsing state thiophosphonium ion. In continuation of this, You *et al*. [36] reported an effective methodology for the electrophilic sulfenylation of indole catalyzed by triphenylphosphine from arylsulfonyl chlorides. In this process arylsulfonyl chlorides, which act as a sulfur source in the presence



Scheme-IV: Sulfenylation of indoles with sulfonyl chlorides

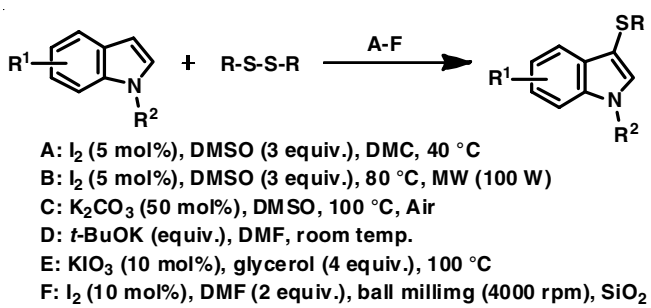
of triphenylphosphine underwent reductive coupling with electron rich indoles to furnish 3-sulfenyl indoles in good yields. Mechanistically, they suggested that electrophilic sulfenyl iodides (RSCl) as a key intermediate that may behave as a selective source of RS^+ equivalents (**Scheme-IV**). Currently, Jiang & Yi [37] disclosed for the first time the sulfenylation of indoles *via* collaborative CTAB- and squaric acid-promoted deoxygenation of sulfonyl substrates. The proposed mechanism of this process suggests that squaric acid dramatically diminished the energy barrier in the initial stage of deoxygenation. This stage promotes the reduction of a large number of functionalized sulfonyl chlorides and sulfonic anhydrides. After that a bromine catalyzed redox cycle was developed to stimulate the dual deoxygenation of sulfonyl chlorides, which conquer the imminent use of the organophosphorus reductant. Additionally, squaric acid also serves as an suitable reductant to promote this bromine catalytic cycle.

Also, 3-sulfenyl indoles have been synthesized *via* conducting the photo-redox reaction between *N*-methylindoles and arylsulfonyl chlorides in average yields (**Scheme-V**) [38]. The authors proposed the hypothetical process may include (i) *N*-methyl indole reductively quenching photocatalyst to generate highly reductive $Ru(bpy)^{3+}$; (ii) aryl sulfonyl chloride reductively quenching and other steps of SET generating key intermediate electrophilic sulfenyl iodides (ArSCl), which attack with *N*-methylindole to produce the desired product.



Scheme-V: Synthesis of 3-sulfenyl indoles by photoredox reaction

Disulfides as sulfenylating agent: In 2012, Ge & Wei [39] reported an adequate strategy for the formation of 3-sulfenylated indoles *via* iodine catalyzed oxidative system from disulfides (**Scheme-VI**). This process required 5 mol% inexpensive iodine as a catalyst in an environmentally benign solvent dimethyl carbonate (DMC) using more than one equivalent oxidant, such as DMSO and without demanding oxygen or moisture-free reaction conditions (**Scheme-I**) and suitable for *N*-protected or unprotected indoles. This procedure mechanistically may involve electrophilic sulfenyl iodides (RSI) as the key intermediate, which can provide the electrophile RS^+ and react with indole moiety.

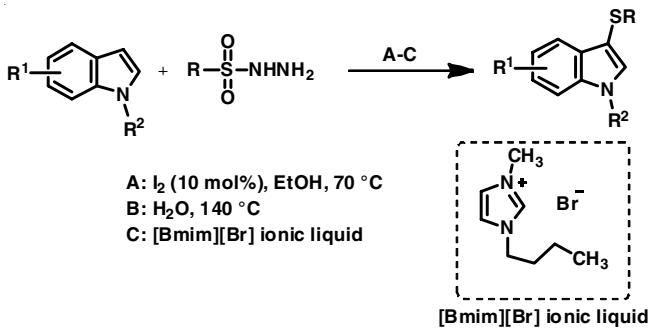


Scheme-VI: Sulfenylation of indoles with disulfides

Braga *et al*. [40] reported a method for the formation of 3-sulfenyl indoles from disulfides as a sulfenylating agent, DMSO as an oxidant and molecular iodine as a catalyst without using any solvent. However, Zhang *et al*. [41] described an easy and adequate method to synthesize C(3)-H sulfenylation of free (N-H) indoles catalyzed *via* K_2CO_3 from disulfides. This process progress is satisfactory in presence of moderate reaction conditions, easy operation to afford the designed 3-sulfenylindoles in very good yields. Further, to explore 3-sulfenylation of indoles at room temperature, Liang *et al*. [42] reported an efficient and convenient route for the C3 sulfenylation of indoles with high regioselectivity by employing *t*-BuOK as a promoter at room temperature. This transformation applied to both *N*-protected and *N*-unprotected indoles.

Braga *et al*. [43] in 2018 developed a greener protocol for the synthesis of 3-S-indoles through direct C(sp^2)-H bond sulfenylation using KIO_3 as a non-toxic, easy-to-handle catalyst and a stoichiometric amount of glycerol. The reaction features are high yields, based on atom economy, easy performance on Gram-scale, as well as applicability to different types of *N*-heteroarenes. Zhong *et al*. [44] realized an efficient C3 sulfenylation of indoles with disulfides under Ball Milling using in combination with iodine catalysis (**Scheme-VI**). This protocol is accomplished by using the thiolating agent's disulfides as well as catalyst molecular iodine to provide broad structural diversity under aerobic oxidation conditions. Moreover, mechanistic studies revealed that the solid and liquid grinding auxiliaries synergistically facilitate this process.

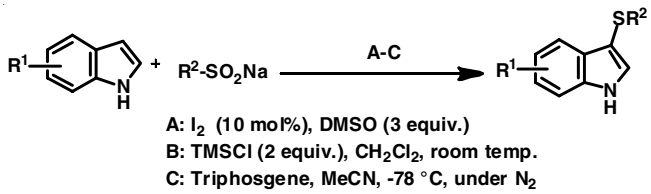
Sulfonyl hydrazides as sulfonylating agent: Yang & Tian [45] in 2013 reported an iodine-catalyzed sulfenylation of indoles regioselectively with sulfonyl hydrazides in good yields (**Scheme-VII**). In 2016, Wang *et al*. [46] discovered a simple green chemical protocol for the thiolation of indoles from sulfonyl hydrazides catalyzed by water in absence of any ligand or additive to afford a wide range of 3-sulfenylindoles and involving only nitrogen and water as a byproduct that made this process a more environmentally benign and economical reaction pathway. To further improvise this protocol, Raghuvanshi & Verma [47] explored a novel and efficient green protocol for regioselective thiolation of indole without using any catalyst. This procedure tolerated a wide range of substituents such as alkyl, nitro, trifluoromethyl and methoxy groups and underwent high conversion with excellent regioselectivity in a short reaction time and also involve the use of a recyclable ionic liquid, all this privilege assist this process to be a practical and greener alternative.



Scheme-VII: Sulfenylation of indoles with sulfonyl hydrazides

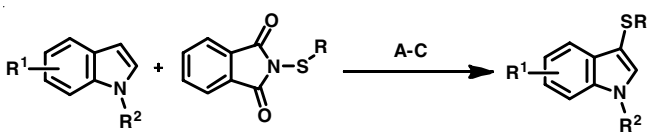
Sodium sulfinate as sulfonylating agent: Sodium sulfinate is found to be stable, easily operable and widely applicable as sulfonylating agent. Deng *et al*. [48] demonstrated the process of sulfenylation of free indoles *via* iodine catalyst from sodium sulfinates to furnish regioselective 3-arylthioindoles with high yields. This process pathway involves the formation of 1,2-diphenyldisulfane using I_2 from sodium sulfinates that further undergo reaction with I to produce an electrophilic species PhSI, which finally react with indole to afford the designed product.

In continuation of this work, Wu *et al*. [49] described firstly the regioselective protocol for the C3-H thiolation of indoles using TMSCl as a promoter from sodium arylsulfinates. Divergence to that the catalyzation of similar reaction conditions under the same set of a substrate with TMSOTf in place of TMSCl underwent C2-H thiolation of indoles regiospecifically. Recently, Guo *et al*. [50] also developed electrophilic thiolation by using commercially available triphosgene as a reductant and the appropriate alkyl- or arylsulfinates, which were transformed in situ into electrophilic RSCl intermediates in the presence of triphosgene (**Scheme-VIII**).



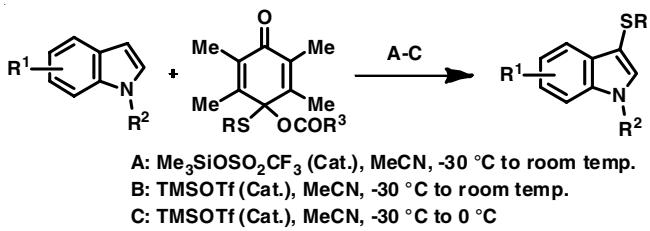
Scheme-VIII: Sulfenylation of indoles with sodium sulfinate

Thiophthalimide as a sulfenylating agent: Tudge *et al*. [51] developed the most illustrious protocol for immediate sulfenylation of the indole moiety by using readily available thiophthalimide reagents as a sulfenylating agents and halide-containing salts as a catalyst at 90 ºC. Particularly, this method can be operated to sulfenylate deactivated indole with sterically preferring sulfur moieties in an adequate manner. Further Marcantoni *et al*. [52] improvised this methodology by conducting the regioselective sulfenylation of indole at room temperature in the presence of sulfenylating agents S-arylthiophthalimides and catalyst $CeCl_3{\cdot}7H_2O$/NaI. Finally, Silveria *et al*. [53] reported a simple and general strategy for the formation of 3-sulfenyl indole by treating S-arylthiophthalimides with indoles using $CeCl_3$ as an efficient catalyst at 70 ºC (**Scheme-IX**).



Scheme-IX: Sulfenylation of indoles with thiophthalimides

Quinone mono-O,S-acetals as a sulfenylating agent: In the last few years, several compelling synthetic protocols for the formation of 3-sulfenylindoles have been discovered. Amidst, the direct sulfenylation of the indole ring by quinone mono-O,S-acetals as a sulfenylating agent is another significant one. Kita *et al*. [54] were the first who reported a facile and efficient sulfenylation method using quinone mono-O,S-acetals which gives high yields under almost neutral conditions. Furthermore, the same group [55,56] also reported a novel sulfenylation method induced by aromatization of quinone mono-O,S-acetals which possesses a pentafluorophenylthio group, is the most effective reagent from the standpoint of the adaptability for various substrates (**Scheme-X**).



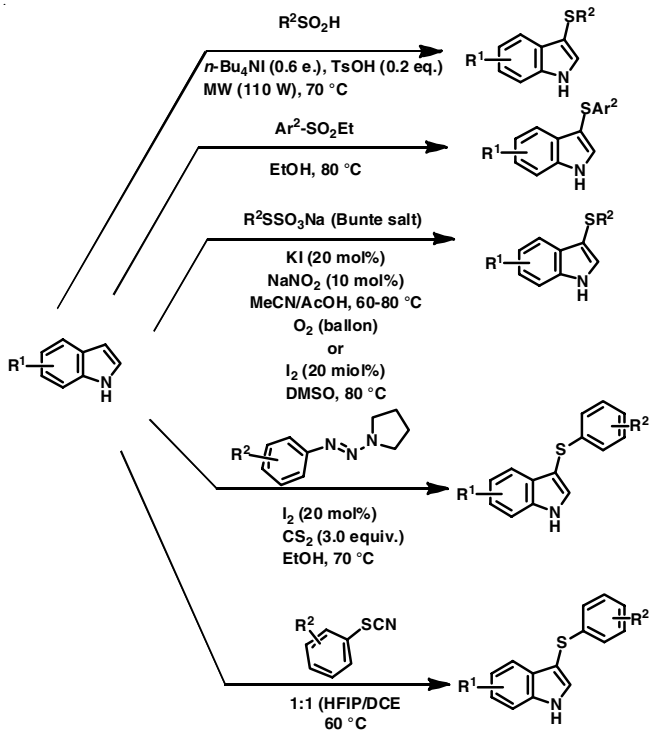
Scheme-X: Sulfenylation of indoles with quinone mono-O, *S*-acetals

Miscellaneous reactions for direct sulfenylation at the 3-position of indoles: Rahaman *et al*. [57] disclosed the direct sulfenylation of indoles with sulfinic acids in the absence of catalyst under microwave irradiation in 10 min (**Scheme-XI**). This method tolerates the several function groups. Sulfinic esters because of their impressive features such as easy-to-make, air as well as moisture stable and anomalous electrophilic and nucleophilic behavior, act as important building blocks for the production of organic sulfur compounds. In this context, Yang *et al*. [58] reported the catalyst-free method for the synthesis of structurally diverse indole thioethers in presence sulfinic esters as new sulfur electrophiles. In 2018, Shen *et al*. [29] reported the 3-sulfenylation of indoles with Bunte salts with inexpensive potassium iodide as catalyst and sodium nitrite as co-catalyst. Also, Luo *et al*. [60] successfully developed Bunte salts as the sulfenylating agents for 3-sulfenylation of indoles (**Scheme-XI**). The reactions bear the regioselective generation of 3-sulfenylindoles in good yields. Mechanistically, electrophilic sulfenyl iodides (RSI) are supposed to be the main intermediate.

A novel and effective sulfenylation strategy has been discovered by Singh *et al*. [61] for the thiolation of indoles regioselectively by using together a cheaper and easily available 1-aryltriazene and CS_2 as a sefenylating agent and molecular iodine as a catalyst. In 2021, Kalaramna & Goswani *et al*. [62] also reported the employment of thiocyanates as a sulfur source for the formation of indole thioethers *via* HFIP-mediated sulfenylation of indole by treating thiocyanates with indoles.

Conclusion

A rapid growth in the development of various significant carbon-heteroatom bond formation emerge as a power tool in organic chemistry. Amid all, the C-S bond formations covered in this review are at the forefront to form momentous 3-sulfenylated indoles compounds. A wide variety of sulfenylating agents,



Scheme-XI: Sulfenylation of indoles with various sulfenylating agents

such as thiols, arylsulfonyl hydrazides, sulfinates, disulfides, *N*-thioamides, sulfonium salts, quinone mono-O, S-acetals, sulfenyl halides, arylsulfonyl chloride, *etc*. are found to catalyze the simple C-S bond formations with greater yield and stereoselectivity. At the current stage, it is believed that the design and development of new methods showing new modes of action will make the C-S bond formation even more practical and handy for the synthesis of biologically significant sulfenylated indole molecules.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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