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Transition Metal Catalyzed Carbodiimide Synthesis

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Abstract: Carbodiimides are an significant class of cumulene-type nitrogenous compounds that serve as crucial intermediates and precursors in the synthesis organic molecules. They have drawn considerable attention among the organic chemists because of their easy availability, exceptional chemical activities and wide-ranging applications, particularly in the synthesis of nitrogen containing heterocycles. Due to their versatile applications, considerable research has focused to the development of new methodologies for the synthesis of functionalized carbodiimides; particularly by employing hypervalent iodine reagents and transition metal-catalysts. Recently, we reported a review article for the synthesis of carbodiimides using hepervalent iodine as reagents. In recent years, the transition metal catalyzed oxidative coupling of azides and isocyanides has emerged as an attractive method for the construction carbodiimides via nitrene transfer mechanism. In this review, we discuss recent advances in the transition metal-catalyzed synthesis of carbodiimides, highlighting innovative strategies and mechanistic insights.

Keywords: *Carbodiimides, Azides, Isocyanides, Transition Metal Catalysts, Palladium, Chromium and Iron.*

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I. INTRODUCTION

Carbodiimides are a unique heterocumulene nitrogenous compounds having general structural formula as R–N=C=N–R. They are highly reactive organic moieties which have wide applications in the pharmaceutical, agricultural, and polymer industry [1-2]. They are extensively used as excellent coupling reagents for the formation of peptide bond in protein synthesis [3-7]. Among these, *N*,*N*′-dicyclohexylcarbodiimide (DCC) and *N*,*N*′-diisopropylcarbodiimide (DIC) are two important chemical reagents used in many organic transformations [8-10]. The functionalized carbodiimides have proven to be an important class of nitrogenous building blocks in the synthesis of *N*-heterocycles of biological significance [11-15].

Due to their diverse applications in organic chemistry, extensive research efforts have been paid by the researches across the globe to developing new methods for the synthesis of functionalized carbodiimides. The most typical methods used for the preparation of carbodiimides include dehydration of ureas [16-18] or dehydrosulfurization of *N*,*N*′-disubstituted thioureas [19-21], and thermal decarboxylation of isocyanates [22-25] (Scheme 1). Many of these protocols suffer from several shortcomings, including requirement of high temperature, longer reaction time, use of toxic reagents and solvents, and poor atom economy, limiting their applications in organic synthesis. To elevate the limitations associated with traditional methods, scientist over the globe paid attention to develop new protocols that provide access to functionalized carbodiimides under sustainable conditions. Recently, many new synthetic methods, including hypervalent iodine reagents hypervalent iodine-mediated desulfurization of thioureas and transition metal-catalyzed cross-coupling reaction between isocyanides and azides, have been developed for the synthesis of carbodiimides. Usually, the preformed N-C-N skeleton or addition of N to C-N and a (N + C + N) strategies have been applied for the construction of carbodiimides. In the direct N + C-N type approach, the condensation of an amine with isonitrile such as direct cross-coupling of amines and isocyanides or transition metal-catalyzed nitrenes insertion into isonitriles, has also been explored for the preparation of carbodiimides [26]. In 2020, Wang and co-workers published a review article covering the application of carbodiimides for the synthesis of *N*-heterocycles [27]. Recently, we also published a review article for the synthesis of carbodiimides using hepervalent iodine as reagents [28]. In this article, the recent development towards the synthesis of functionalized carbodiimides using transition metal catalyst has been discussed.

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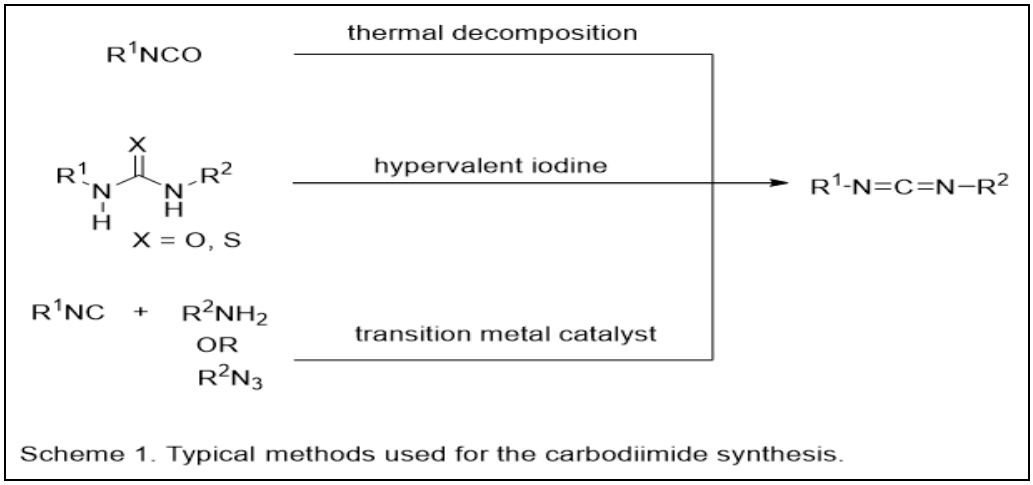


Scheme 1. Typical methods used for the carbodiimide synthesis.

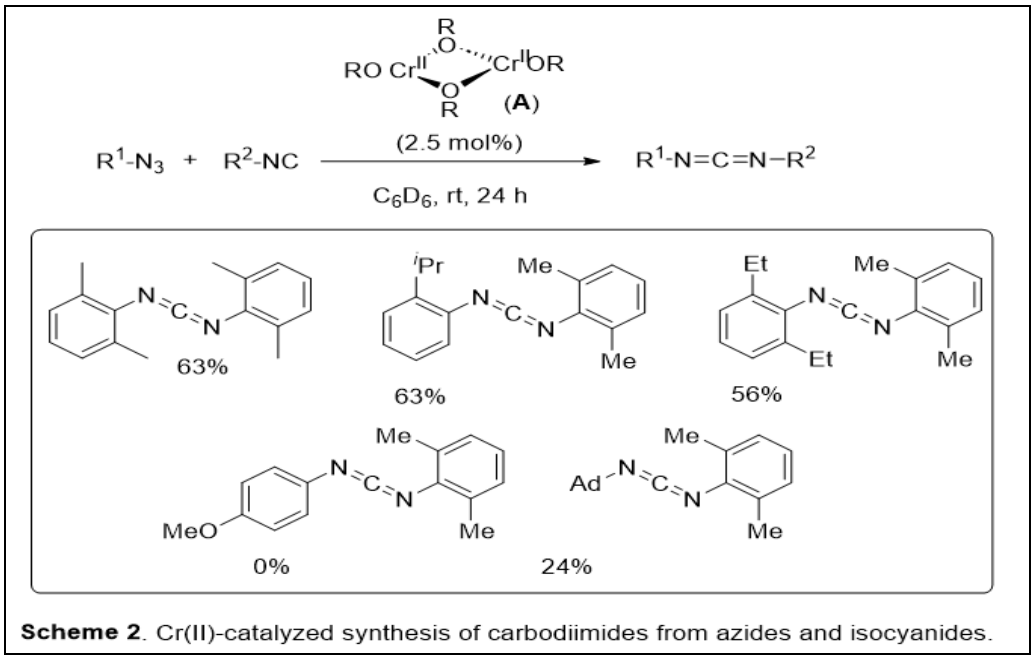
II. SYNTHESIS OF CARBODIIMIDES USING TRANSITION METAL CATALYSTS

Transition-metal catalyzed reactions, such as Chromium (Cr), Palladium (Pd), Nickel (Ni) or Iron (Fe)-catalyzed cross-coupling reaction between azides and isocyanides, titanium-catalyzed nitrene transfer from diazenes or azides to isocyanides and palladium-catalyzed insertion reactions of isocyanide with amines offer attractive strategies for the preparation of *N,N'*-dialkylcarbodiimides with high atom economy [29-31].

The transition metal-catalyzed formation of metal-nitrenes (M=NR) from azides, followed by the coordination of isocyanides and subsequent nitrene transfer to isocyanides, has emerged as an efficient method for synthesis of carbodiimides from azides and isocyanides [32-37]. In this context, $Fe(CO)_5$ has been utilized for the synthesis of carbodiimides from azides and isocyanides through a nitrene transfer mechanism. However, this reaction required a higher temperature to achieve the desired carbodiimides [38].

Hillhouse et al. developed a *N*-heterocycliccarbenes (NHC)-supported Ni(I) monochloride catalyst that enabled the nitrene transfer reaction from $MesN_3$ to isocyanide, leading to the formation of carbodiimides in high yields [39]. Notably, this protocol was failed to work with tosyl azide. Subsequently, Warren et al. developed a β-diketiminato Ni(I)-catalyst for the oxidative coupling between ^{t}BuNC and azides. The Ni-catalyst is proven very effective for the nitrene transfer, yielding a variety of carbodiimides in excellent yields [40].

Chromium-based metal catalysts have also been employed in catalytic nitrene transfer to isocyanides, leading to the formation of carbodiimides [41-42]. Groysman and co-workers demonstrated the synthesis of a low-valent Cr(II) complex (A) featuring a bis(alkoxide) ligand (Scheme2). They examined the efficiency of this Cr(II) complex facilitating the nitrene transfer to isocyanide [41]. Their studies revealed that the protocol was compatible with relatively bulky aryl azides or aryl isocyanides only. However, attempt to carbodiimides formation with less bulky aryl azides was unsuccessful.



Scheme 2. Cr(II)-catalyzed synthesis of carbodiimides from azides and isocyanides.

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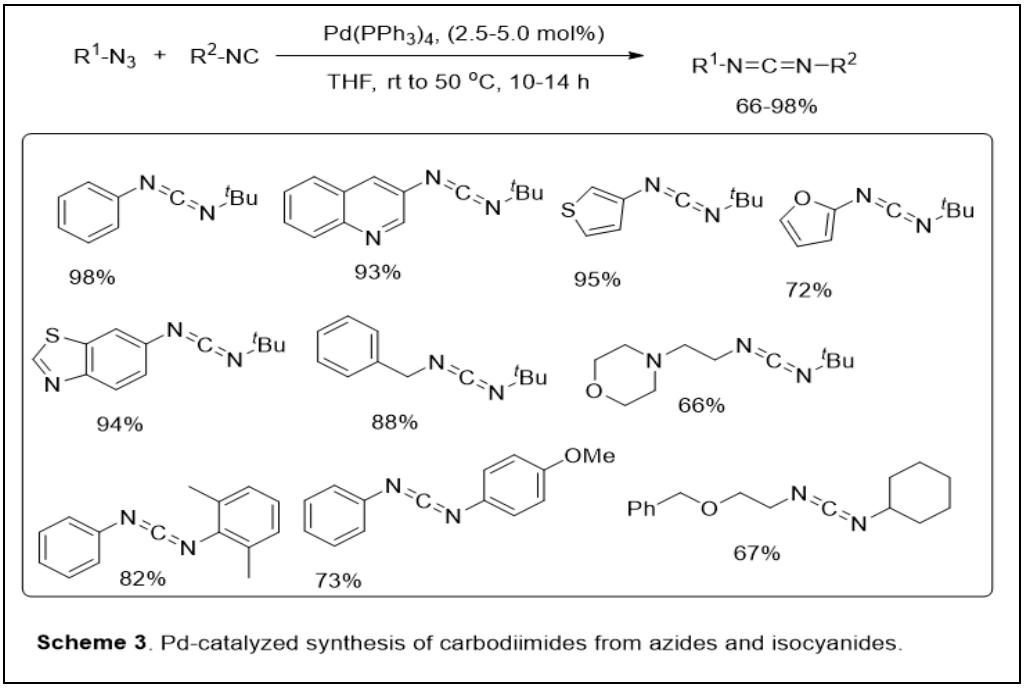
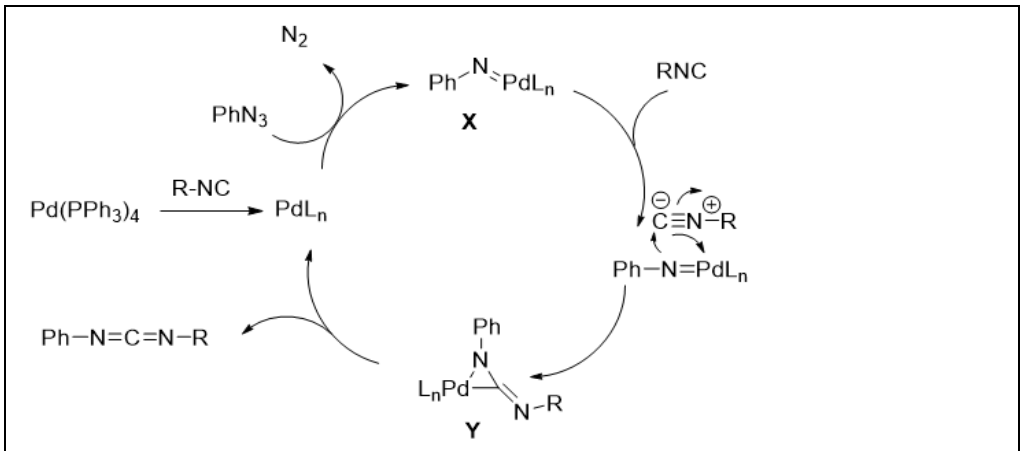
Zhang et al. reported the first synthesis of carbodiimides from aryl azide and isocyamides using a palladium catalyst. The cross-coupling reaction of azides with isocyanides was carried out with 2.5 mol% of $Pd(PPh_3)_4$ in THF solvent at rt to 50 °C for 10h, affording a wide variety of unsymmetrical carbodiimides in excellent yields (Scheme 3) [43]. This protocol tolerated a broad range of azides, including alkyl, aryl, and benzyl azides as well as a diverse alkyl and aryl isocyanides.



Scheme 3. Pd-catalyzed synthesis of carbodiimides from azides and isocyanides.

The reaction proceeds via a nitrene transfer mechanism, wherein a palladium-nitrene species (X) is formed through ligand exchange with isocyanide and concomitant release of nitrogen gas (Scheme 4). Subsequently, isocyanide insertion into the Pd-nitrene species leads to formation of a three-membered Pd-metalized intermediate Y. The free carbodiimide is obtanied from Y via a reductive elimination, regenerating the Pd(0) catalyst.



Scheme 4. Probable catalytic cycle of Pd-catalyzed cross-coupling of azides and isocyanides.

Recently, Tonks and co-workers [44] developed Titanium-imido halide complexes, such as $[Br_2Ti(N^tBu)py_2]_2$ which acted as a catalyst for the synthesis of unsymmetrical carbodiimides via nitrene transfer from diazenes to isocyanides. In a typical procedure, the reaction of diazobenezene (PhNNPh) with *tert*-butylisocyanide and 5 mol% of $[Br_2Ti(N^tBu)py_2]_2$ proceeded smoothly in $PhCF_3$ solvent at 115 ^{0}C for 24h, resulting to the formation of 1-*tert*-butyl-3 phenylcarbodiimide in 85% yield (Scheme 5). It was found that sterically hindered isocyanide substrates limit the yield of carbodiimides when diazobenezene is employed as the oxidant. A variety of electronically donating and withdrawing diazenes reacted with *tert*-butylisocyanide in C_6D_5Br solvent to give the corresponding 1-*tert*-butyl-3-arylcarbodiimides in good yields (Scheme 5). Notably, the yield of the products decrease with bulkier group such as ^{i}Pr group substituted at the *ortho*-position of diazene.

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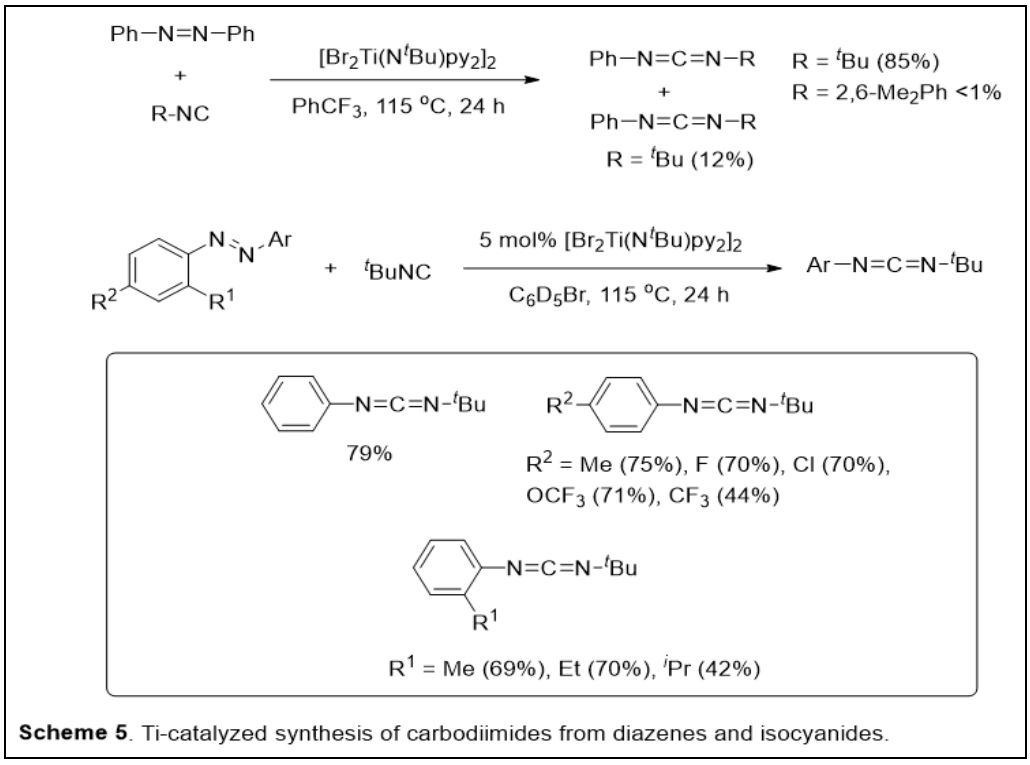
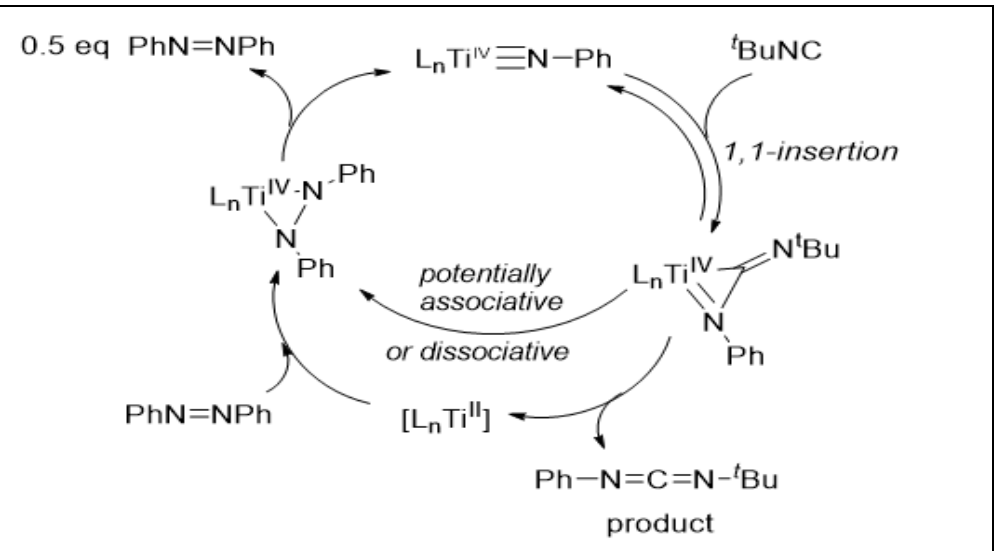


Scheme 5. Ti-catalyzed synthesis of carbodiimides from diazenes and isocyanides.

The authors investigated the reaction mechanism using both experimental and computational approaches, wherein it was revealed that the formation of the product is driven by electron transfer from an η^2-carbodiimide to a Ti-bound azobenzene. In the first step, an isocyanide coordinates to the Ti imido, followed by a 1,1 migratory insertion into the Ti-imido bond to generate an η^2-carbodiimide intermediate. Subsequently, the carbodiimide ligand is substitution by azobenzene (PhNNPh) either via an associative or dissociative mechanism, resulting in the formation of carbodiimide product with the release of an η^2-hydrazido. The η^2-hydrazido undergoes disproportionation to regenerate the Ti imido and PhNNPh (Scheme 6).



Scheme 6. Catalytic cycle of Ti-catalyzed synthesis of carbodiimides.

Rahman and Nicholas reported [45] the synthesis of carbodiimides from isocyanates using low valance transition metal catalyst. In this approach, two molecules of isocyanates are reacted in the presence metal catalyst such as $[(C_5H_5)Fe(CO)_2]_2$ or $MeCpMn(CO)_3$ in refluxing xylene, leading to the formation of carbodiimides (Scheme 7). The reaction proceeds through the formation of a four-membered heterocyclic complex, generated by the reaction of two molecules isocyanates with metal catalyst. This heterocyclic complex undergoes fragmentation with the loss of CO_2, producing a transition metal coordinated carbodiimides, which on dissociation gave carbodiimide product and completing the catalytic cycle.

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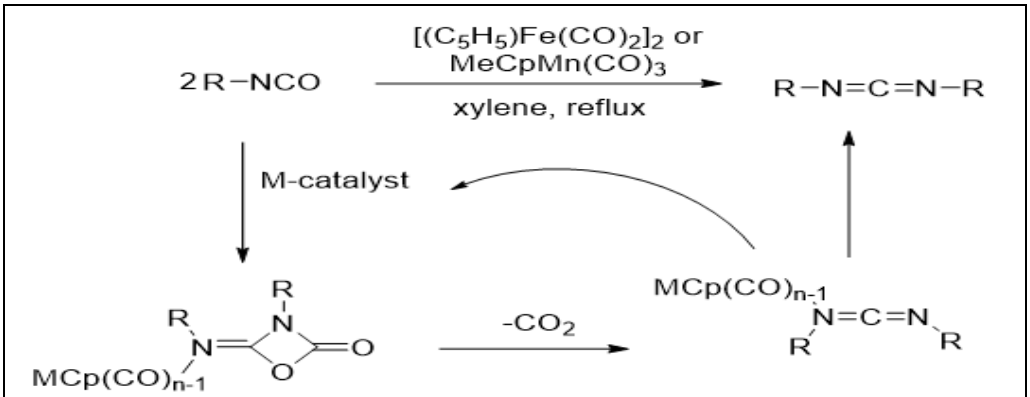
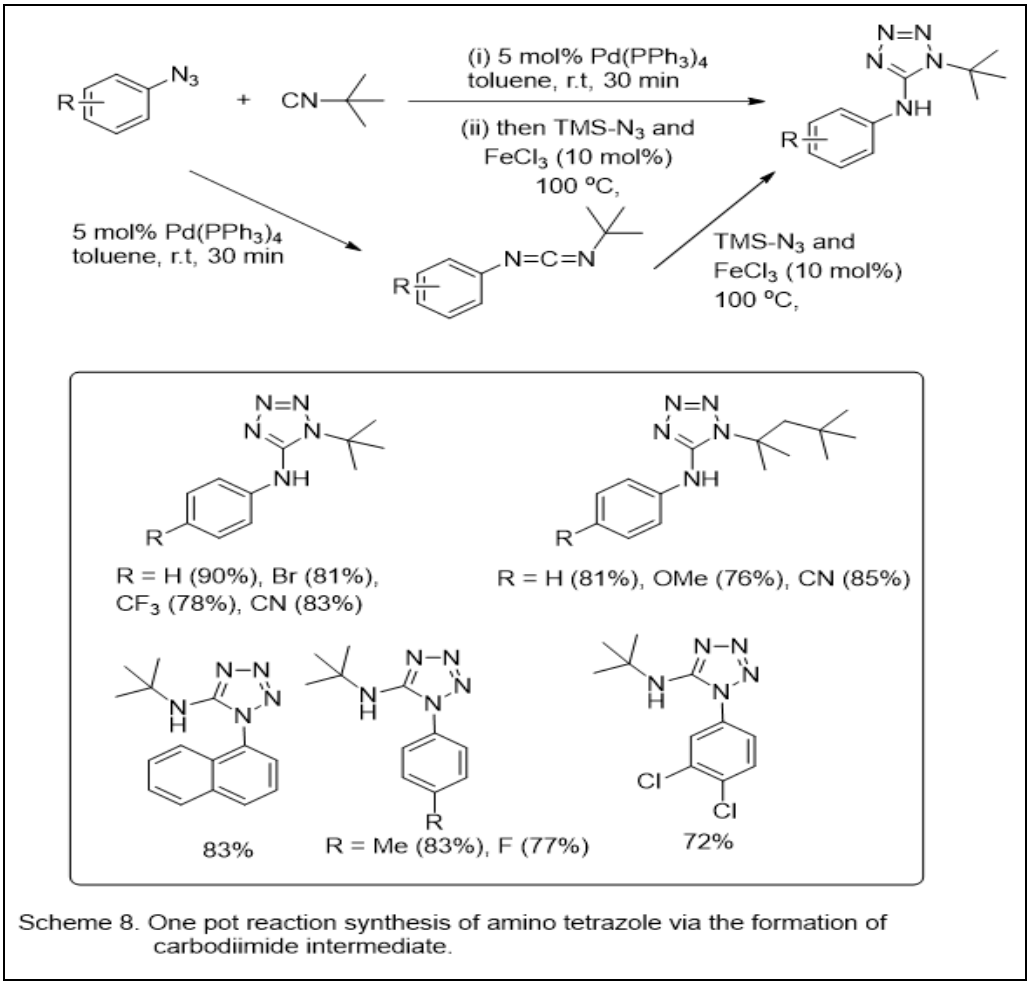


Scheme 7. Fe-catalyzed conversion of isocyanate to carbodiimide.

Sawant and co-workers [46] developed a three-component, two steps, one pot protocol for the synthesis of amino tetrazole from the reaction between aryl azides, isocyanides, and $TMSN_3$ via the formation carbodiimide intermediate. The process involves a sequential reactions catalyzed by Pd(0) and Fe(III) catalysts. Initially, the denitrogenative coupling reaction of azide and isocyanide is catalyzed by $Pd(PPh_3)_4$ to generate an unsymmetrical carbodiimide in situ. Subsequently, this intermediate reacts with $TMSN_3$ in the presence of $FeCl_3$ within a single pot to give amino tetrazole (Scheme 8). The reaction between aryl azide and isocyanides was proceeded with 5 mol% $Pd(PPh_3)_4$ as catalyst in toluene solvent at room temperature, yielding to carbodiimide intermediate in 30 minutes. After completion of carbodiimine formation, TMS-N_3 and $FeCl_3$ (10 mol%) were introduced and the reaction mixture was refluxed to100°C to furnish the 5-amino-1*H*-tetrazole in high yields. This protocol tolerated a broad range of aryl azides bearing both electron-donating and –withdrawing substituents, as well as various isocyanides, efficiently reacted with $TMSN_3$ to delivered the corresponding 5-amino-1*H*-tetrazoles.



Scheme 8. One pot reaction synthesis of amino tetrazole via the formation of carbodiimide intermediate.

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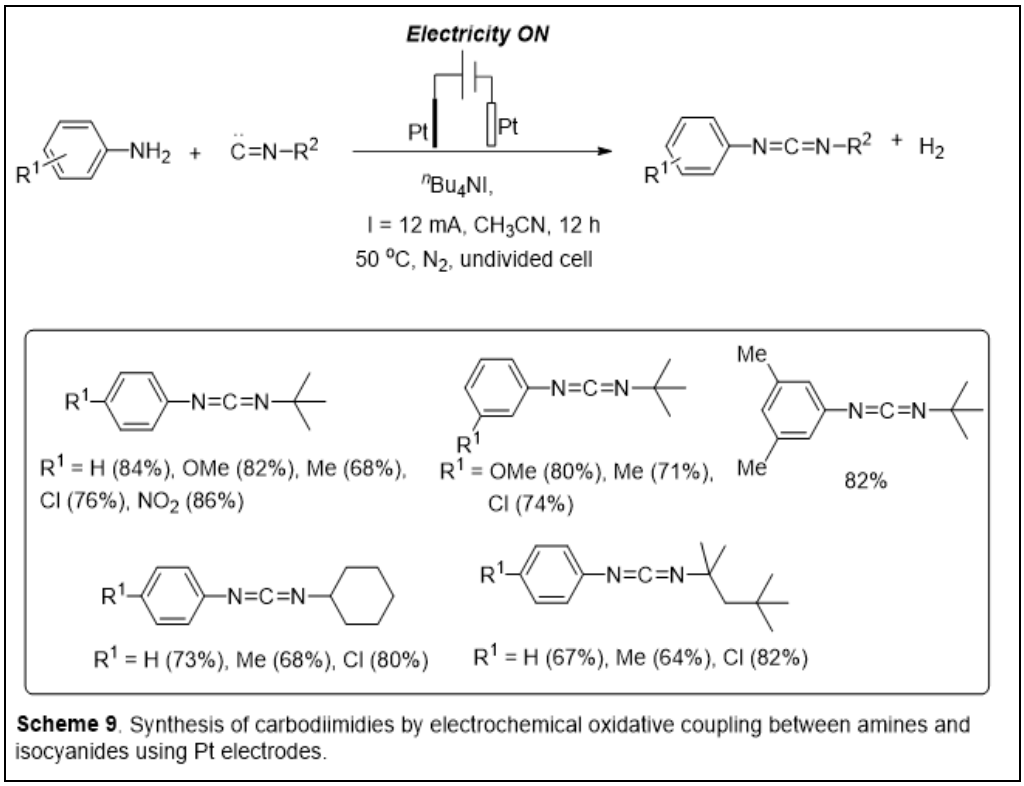
Very recently, Sharma and co-workers [47] developed an electrochemical oxidative cross-coupling method for the synthesis of carbodiimidies from amines and aliphatic or aryl isocyanides (Scheme 9). The reaction proceeds without involving any transition-metal catalysts, external oxidants, or toxic reagents, delivering carbodiimides in good yields. The reaction involves the treatment of amines and isocyanides under electrochemical oxidative conditions using platinum electrode, serve as both cathode and anode, at constant current of 12 mA for 12h in the presence of $^{n}Bu_4NI$ in CH_3CN solvent. This method protocol has been applied for the synthesis of a broad range of 1,3 disubstituted carbodiimides bearing aryl-aryl, aryl-alkyl and alkyl-alkyl groups. Hydrogen gas is generated as the only byproduct. Moreover, carbodiimides products were directly converted in situ into ureas in moderate to good yields by employing an electricity ON−OFF strategy.



Scheme 9. Synthesis of carbodiimidies by electrochemical oxidative coupling between amines and isocyanides using Pt electrodes.

III. CONCLUSION

In summary, the synthesis of carbodiimides catalyzed by transition metal-catalyst has been discussed. Many transition metal-catalysts, including palladium, chromium, titanium and iron have been employed for the preparation of functionalized carbodiimides. This includes the titanium-catalyzed nitrene transfer from diazenes or azides to isocyanides, Pd-catalyzed cross-coupling of azides with isocyanides or Pd-catalyzed amine insertion to isocyanide. These strategies offer efficient routes for the preparation of *N*,*N*′-disubstituted carbodiimides with high atom economy. More recently, an electrochemical oxidative cross-coupling reaction of amines with isocyanides using platinum electrodes has also been developed for the preparation of carbodiimidies. This protocol proceeds without involving any transition-metal catalyst, oxidant, or toxic reagents, where H_2 is generated only as by product. This electrochemical method is considered the most atom-economic and environmentally friendly protocol for the synthesis of carbodiimides. Due to the diverse applications of carbodiimides in the preparation of valuable nitrogen heterocycles of biological interest in industry, we hope that many more exciting protocols will be developed for the the synthesis of carbodiimides in near future.

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- *Conflict of Interest:*

The author declares no conflict of interest.

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