

Application of Nanocatalysts in C-Te Cross-Coupling Reactions: An Overview

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ABSTRACT

Synthesis of organo-telluride compounds through nanoparticles catalyzed C-Te cross-coupling reactions has drawn considerable attention in the past several years. This mini review is an attempt to highlight the most important advances and contributions in this fast-growing research field with the emphasis on the mechanistic aspects of the reactions. The reactions are classified based on the type (e.g. coupling of organic halides with elemental Te(0), coupling of terminal acetylenes with ditellurides). Literature has been surveyed from 2009 to end of 2018.

1. Introduction

Transition-metal-catalyzed cross-coupling reactions provide powerful and general methodologies for formation of various carbon-carbon [1-3] and carbon-heteroatom bonds [4-6] with high precision under mild reaction conditions. In this context, numerous studies have been focused on the carbon-tellurium bond formation reactions for the synthesis of organo-telluride compounds [7-9], which are attractive synthetic targets for biochemists and synthetic chemists [10, 11]. Considering the fact that nano-sized metal catalysts are much more reactive and less expensive than their corresponding bulk catalysts [12-14], nanometal catalyzed C-Te cross-coupling reactions have attracted a lot of attention in recent years. As a continuation of our previous works [15,16] on cross-coupling and nanoparticles catalyzed reactions, in this focus review, we will highlight the most notable developments and advances on the nanoparticles catalyzed C-Te coupling reactions. The review is divided into three major sections (Figure 1). The first section will cover coupling of organic halides with elemental Te(0). The second focuses exclusively on cross-coupling of terminal

acetylenes with ditellurides. The third will discuss coupling of boronic acids with ditellurides.

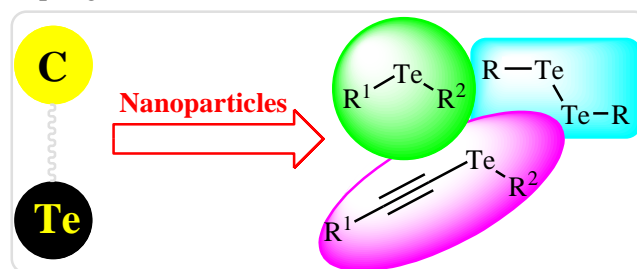


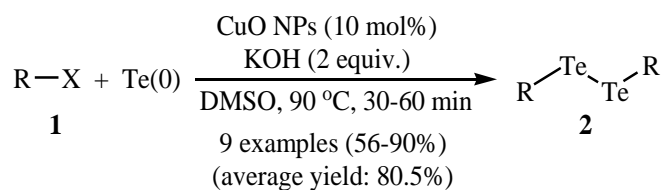
Fig. 1 Nanoparticles catalyzed C-Te cross-coupling reactions.

2. Coupling of organic halides with elemental Te(0)

In 2010, Rodrigues and Braga along with their co-workers studied the possibility of synthesizing diorganyl ditellurides **2** through a Cu NPs-catalyzed coupling reaction between organohalides **1** and elemental Te(0) [17]. Thus, the careful analysis of the optimized reactions revealed that the optimum condition for this C-Te cross-coupling reaction was the addition of CuO NPs (10 mol%), KOH (2 equiv.) at 90 °C, to a solution of halides **1** and Te(0) in DMSO under a nitrogen atmosphere. Under optimized conditions, the reaction

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tolerated various functional groups, including chloro, methoxy, hydroxy and amino functionalities and gave corresponding ditellurides **2** in moderate to high yields (Scheme 1). Some important information of the above-mentioned reactions are listed below: (i) organic iodides compare to organic bromides gave higher yields of the desired coupling products; (ii) aryl halides gave a higher yield of products than alkyl halides; and (iii) the electronic character of the substituents in aryl halides had little effect on the rate of the reaction. This interesting procedure was also successfully extended to the synthesis of diselenides *via* the reaction of organohalides with elemental selenium. Unfortunately, the reusability of the catalyst was not investigated in this report. According to the authors proposed mechanism, the reaction starts with the formation of copper complex **A** *via* the oxidative addition of organic halide **1** to CuO NPs, and meanwhile, generation of active ditelluroate anion from elemental tellurium in the presence of superbasic DMSO–KOH system, and then the generation of the complex **B** *via* ligand exchange of complex **A** with the ditelluroate anion. Subsequently, reductive elimination of intermediate **B** gives the initial coupling product **C** and regenerate the CuO NPs. Next, complex **C** by reaction with another complex **A** yields the complex **D**. Finally, the reductive elimination of **D** affords the expected ditellurides **2** with regeneration of the CuO NPs (Scheme 2). Shortly afterwards, the same authors have reported the cross-coupling of various organic halides and elemental tellurium, selenium or sulfur using commercially available CuO NPs (mean size 33 nm)/KOH combination as a catalytic system in DMSO under microwave irradiation [18]. The expected ditellurides, diselenides, and disulfides were obtained in fair to excellent yields (45-94%) in a very short reaction time (7-15 min). The comparison of the catalytic activity of CuO NPs with a series of copper catalysts such as CuFe₂O₄-nano, CuI, and CuO established its superior comparability with them in the term of product yield.

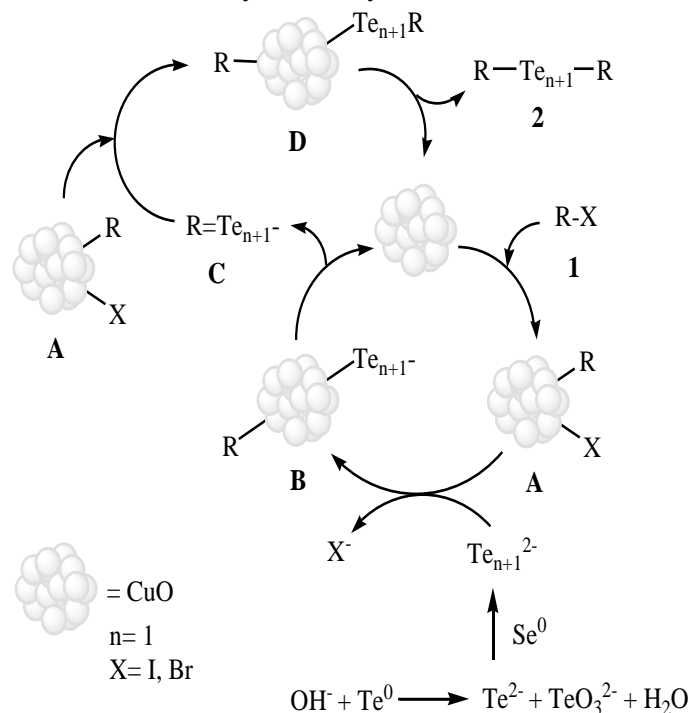


X = I, Br
R = Ph, 4-Me-C₆H₄, 4-Cl-C₆H₄, 2-Me-C₆H₄, 2-OH-C₆H₄,
2-NH₂-C₆H₄, 3-Py, C₇H₁₅

Scheme 1. CuO NPs-catalyzed coupling of organohalides **1** and elemental Te(0).

Following these works, the group of Kassae developed graphene oxide based nano-Fe₃O₄ composite (nano-Fe₃O₄@GO) by heating (85 °C) of graphene oxide with FeCl₃·6H₂O and FeCl₂·4H₂O (2:1 molar ratio) in water (30 min) and then treatment with 30% NH₃ (Figure 2) [19]. The black precipitated product was

easily separated by centrifugation (4000 rpm for 15 min) and washed with deionized water and dried at 60 °C. TEM analysis revealed the formation of spherical shaped nanoparticles that are homogeneously distributed over the graphene oxide sheets (Figure 3). The catalytic utility of the hybrid system was investigated for the synthesis of ditellurides **4** *via* the reaction of corresponding (hetero)aryl iodides **3** with Te(0) in the presence of over-stoichiometric amounts of KOH as a base in DMSO (Scheme 3). The results established excellent catalytic activity of the nanocomposite (yield up to 98%) which was as reusable and could be separated by an external magnet and reused for four runs with no loss of catalytic activity.



Scheme 2. Mechanism that accounts for the formation of diorganyl ditellurides **2**.

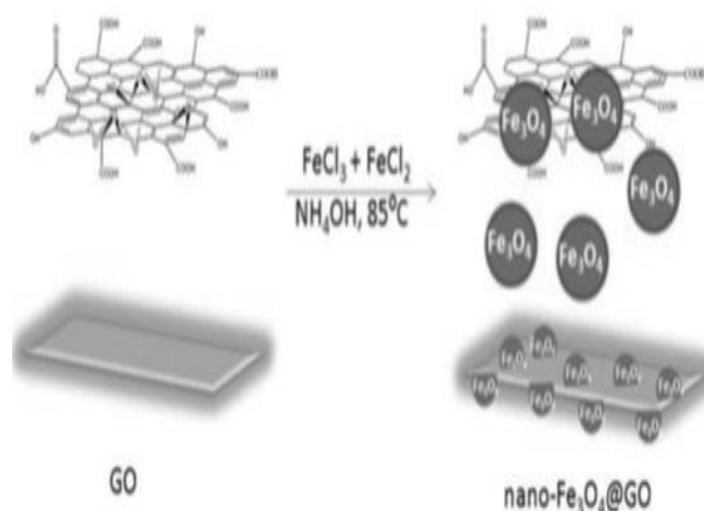


Fig. 2 Schematic diagram showing the formation of nano-Fe₃O₄@GO.

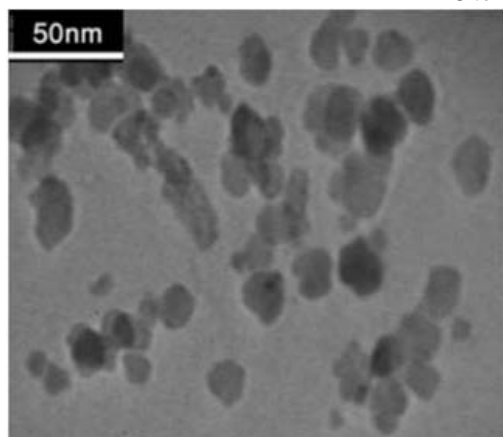
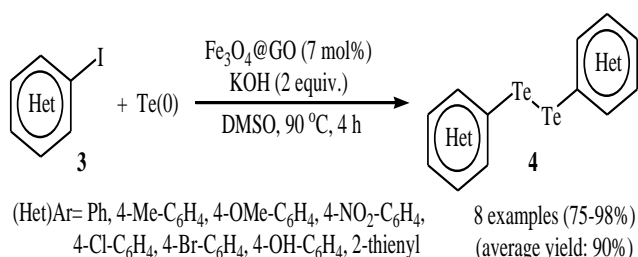


Fig. 3 TEM image of nano-Fe₃O₄@GO hybrid nanoparticles.

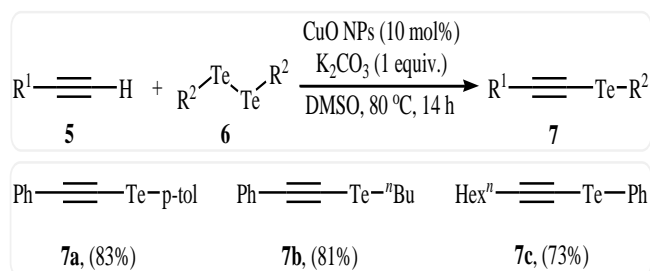


Scheme 3. Kassaei's synthesis of diorganyl ditellurides **4**.

3. Coupling of terminal acetylenes with ditellurides

In 2012, Braga and co-workers demonstrated that the cross-coupling reaction of terminal alkynes **5** with diorganyl ditellurides **6** was efficiently performed when using CuO nanopowder (10 mol%) and K₂CO₃ (1 equiv.) under ligand-free conditions at 80 °C [20]. Among the various solvents like DMF, DMSO, MeCN, DMSO/H₂O, THF; DMSO was the most efficient for the transformation. Various terminal acetylenes and ditellurides were used to establish the general applicability of the procedure. As shown in Scheme 4, both aromatic and aliphatic alkynes and ditellurides were applicable to this reaction. The protocol was also successfully utilized to the C-Se cross-coupling reaction of various terminal acetylenes with diorganyl diselenides, and the corresponding alkynyl selenides were obtained in moderate to excellent yields. After the reaction, catalyst was easily separated and reused for several runs without loss of activity. Transmission electron microscopy (TEM) analysis of the CuO NPs was performed before and after four reaction runs (Figure 4). The results proved that the reaction involves a heterogenous process. The authors proposed that the mechanism of this transformation involves the initial formation of the [alkenylCu] cluster **A** through the reaction of terminal alkyne **5** with the catalyst. The reaction of this intermediate with diorganyl ditelluride **6** gives the intermediate **B**, which undergoes reductive elimination to produce the observed products **7** and the species **C**. Finally, in the reaction medium, the copper catalyst is regenerated through the reaction of **C** with the

base, completing the cycle (Scheme 5).



Scheme 4. CuO NPs-catalyzed coupling of terminal alkynes **5** with diorganyl ditellurides **6** developed by Braga.

Subsequently, magnetite ferroferric oxide nanoparticles (Fe₃O₄ NPs) were also found by the same group to effectively catalyze the C-Te bond formation of terminal acetylenes and ditellurides [21]. Thus, in the presence of 10 mol% of commercially available Fe₃O₄ nanopowder (particle size: 50-100 nm; surface area: 60 m²/g) as catalyst and 1.0 equiv. of K₂CO₃ as a base in DMF at 80 °C, C-Te coupling of terminal alkynes **8** with diorganyl ditellurides **9** furnished corresponding alkynyl tellurides **10** in moderate to excellent yields (Scheme 6). They found that other nano and bulk iron based catalysts also promoted the reaction (*e.g.*, nano-CuFe₂O₃, Fe₃O₄, Fe⁰, Fe(acac)₃, FeCl₃); however, in lower yields. No reaction occurred in the absence of the catalyst. To understand the reusability of the nanopowder in these C-Te cross-coupling reactions, the coupling of phenylacetylene with diphenyl ditelluride was considered as a model reaction. After each cycle, the nano-Fe₃O₄ was recovered from the reaction medium using an external magnet and reused for the same reaction, and this was successive performed for four runs. Like pervious work, TEM images of the nanopowder was performed before use and after an additional four reaction runs and it was found that the magnetite nanoparticles were present in the reaction even in the fourth run. It should be noted that this catalytic system has also been successfully used in the C-Se cross-coupling of terminal acetylenes with diorganyl diselenides under the same conditions.

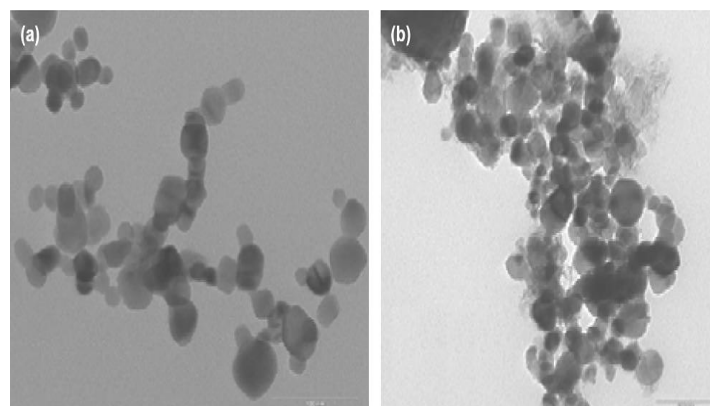
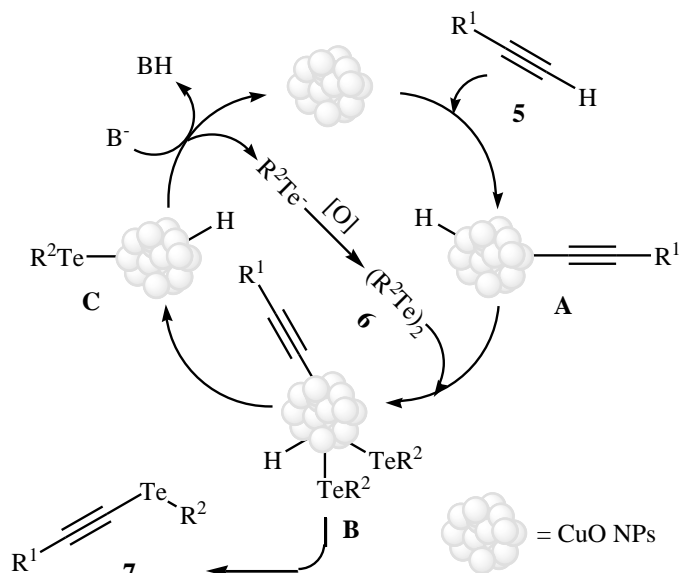
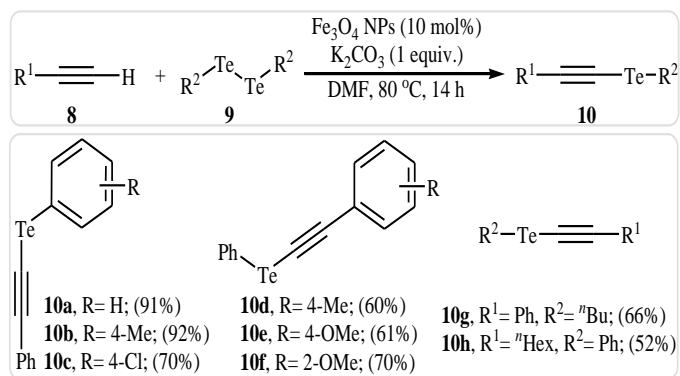


Fig. 4 TEM images of CuO nanopowder (a) Fresh CuO nanopowder; (b) CuO nanopowder after fourth run



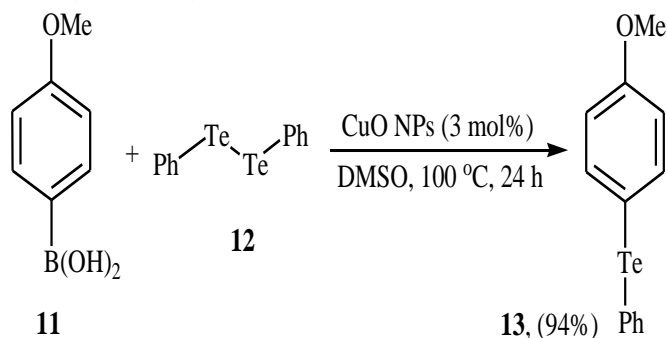
Scheme 5. Proposed mechanistic pathway for the formation of alkynyl tellurides **7**.



Scheme 6. Fe₃O₄ NPs-catalyzed C-Te coupling of terminal alkynes **8** with diorganyl ditellurides **9**.

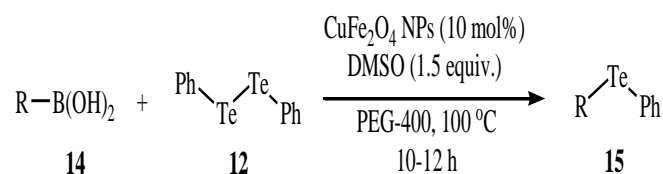
4. Coupling of boronic acids with ditellurides

One of the earliest reports of the applicability of nano-sized metal catalysts in telluration of boronic acids with ditellurides, has been reported by Alves and co-workers in 2009, when 4-methoxyphenyl boronic acid **11** underwent coupling with diphenyl ditelluride **12** in the presence of CuO NPs (3 mol%) in DMSO at 100 °C to form corresponding diaryl telluride **13** in a yield of 94% (Scheme 7) [22].



Scheme 7. CuO NPs-catalyzed coupling of 4-methoxyphenyl boronic acid **11** with diphenyl ditelluride **12**.

With the objective of designing of a comprehensive and green procedure to diorganyl tellurides through C-Te cross-coupling of boronic acids with diorganyl ditellurides, in 2013, Kundu, Mukherjee, and Ranu were able to demonstrate that a range of diaryl, aryl-heteroaryl, aryl-alkenyl, arylalkynyl unsymmetrical tellurides **15** could be obtained in good to almost quantitative yields from the treatment of corresponding boronic acids **14** with diphenyl ditelluride **12** employing nano-CuFe₂O₄ as an efficient catalyst and DMSO as an additive in eco-friendly polyethylene glycol (PEG-400) at 100 °C (Scheme 8) [23]. The optimized protocol tolerated various functional groups, including OMe, COMe, CO₂Et, CHO, NHCOMe, NO₂, OH, SMe, and OCF₃. This made possible the further derivatization of the products. As in telluride synthesis, a variety of unsymmetrical diorganyl selenides were also obtained efficiently by this procedure. Interestingly, the authors also successfully extended the scope of coupling partners to boronic esters and aryl trifluoroborates. It should be mentioned that the catalyst is recyclable up to eight runs without significant loss of its efficiency. However, after each cycle the CuFe₂O₄ nanoparticles which were initially of 20 nm size grew slightly bigger and after the 8th cycle, the particles attained 78 nm sizes and their catalytic activity was reduced substantially (Figure 5). The mechanism proposed to explain the coupling of boronic acids with ditellurides starts with the formation of intermediate **A** via oxidative addition of CuFe₂O₄ NPs to ditelluride **12**, and then transmetalation of **A** with boronic acid **1** to give intermediate **B**, which undergoes reductive elimination to produce the product **15** (Scheme 9, cycle I). On the other hand in cycle II the boronic acid **14** interacts with the catalyst to form the intermediate **C** which then reacts with another half of ditelluride leading to intermediate **B** which finally affords the observed product through a reductive elimination step. Thus both half of ditellurides are consumed in the reaction, making it atom-efficient.



R = aryl, heteroaryl, alkenyl, alkynyl

20 examples (73-96%)
(average yield: 87%)

Scheme 8. Ranu's synthesis of tellurides **15**.

Subsequently, the group of Park was able to demonstrate that copper nanoparticles [Cu₃(BTC)₂] (BTC=benzene-1,3,5-tricarboxylate) immobilized onto activated charcoal (AC) could also effectively catalyzed the C-Te coupling of various boronic acids/esters and diphenyl ditelluride under base and additive-free conditions at room temperature [24].

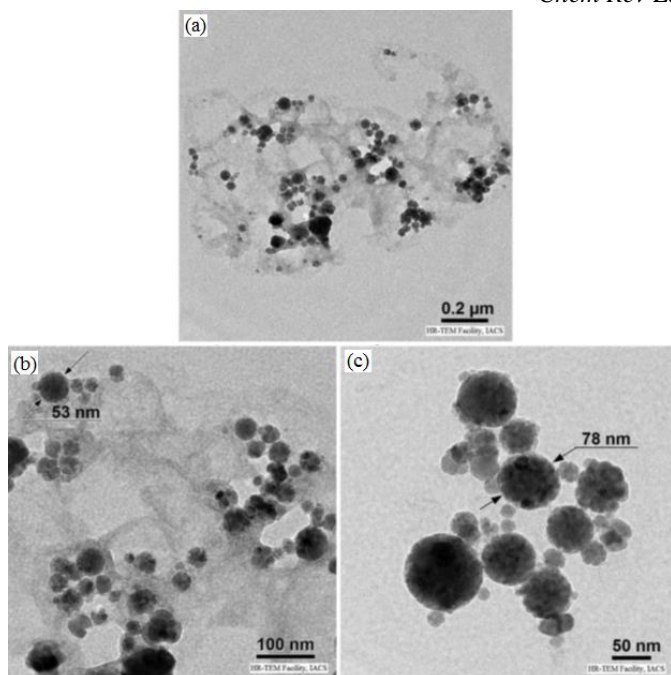
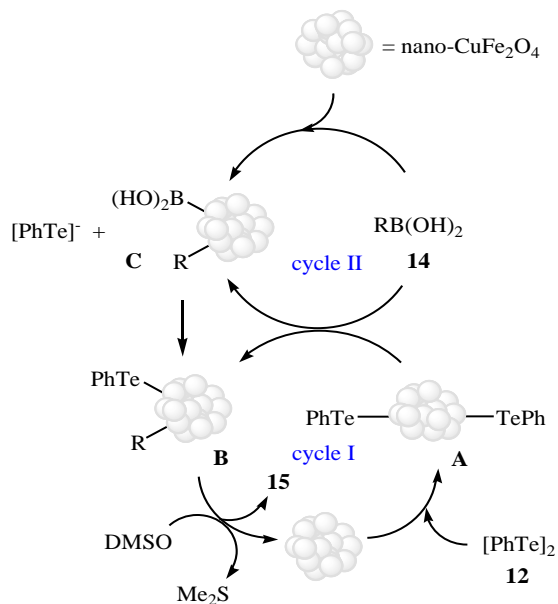


Figure 5. TEM images of (a) fresh CuFe_2O_4 NPs; (b) after 4th cycle; (c) after 8th cycle.

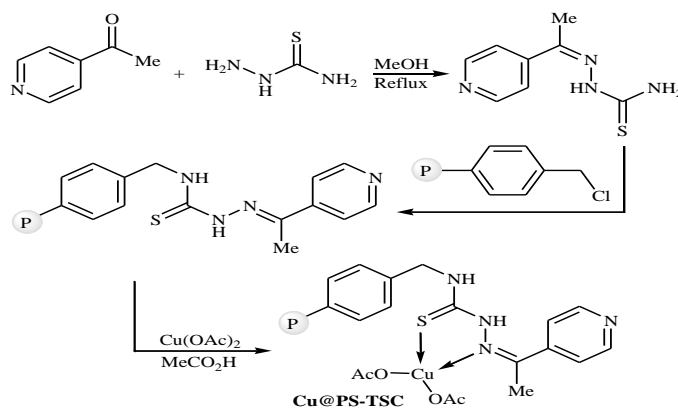
The attempt of catalyst reuse showed noticeable loss of catalytic activity from 100% in the first run to 73% in the fifth run. The authors explained this fact by oxidation of copper NPs in air, which was indispensable in the reaction because it acted as an oxidant.



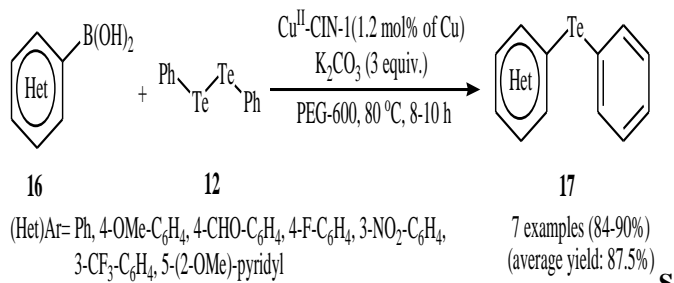
Scheme 9. Plausible mechanism for the formation of unsymmetrical tellurides **15**.

Inspired by these works, Roy, Bhaumik and Islam designed and synthesized a novel polymer supported Cu(II) nanocatalyst (Cu^{II} -CIN-1) *via* a three-step procedure through condensation of 4-acetylpyridine with thiosemicarbazone and subsequent treatment with chloromethylated polystyrene and then incorporation of Cu(II) NPs through reaction with copper acetate in

acetic acid (Scheme 10) [25]. The obtained organo-copper catalyst was characterized by using various analyses such as SEM, EDAX, FT-IR, TGA, and EPR experiments. This novel Cu(II) grafted functionalized polystyrene material was successfully used as an efficient heterogeneous catalyst for the synthesis of wide range of phenyl-(hetero)aryl tellurides **17** through phenyl tellurylation of (hetero)aryl boronic acids **16** with diphenyl ditelluride **12** in PEG-600 (Scheme 11). It is noted that this (Cu^{II} -CIN-1)-catalyzed C-Te coupling reaction is equally effective for both electron-rich and electron-poor aryl boronic acids. Recycling studies have demonstrated that the catalyst can be easily recovered and reused for six runs without noticeable loss of activity.



Scheme 10. Synthetic route for preparation of Cu@PS-TSC .



Scheme 11. Cu^{II} -CIN-1 catalyzed synthesis of phenyl-(hetero)aryl tellurides **17**.

5. Conclusions

In the past few decades, immense attention has been paid to the synthesis of organo-telluride compounds for the sake of their wide importance in medicinal and organic chemistry. Among various methods for construction of C-Te bonds, metal catalyzed cross-coupling reaction is considered one of the most interesting approaches due to the production of various type of organo-tellurides with high atom and step economy under mild conditions.

Recently, the use of nano-sized metal catalysts in this research arena has attracted considerable attention

because of these catalysts with their high surface area allow for rapid transformations under milder conditions. As illustrated, various type of organo-tellurides (e.g. diorganyl ditellurides, diorganyl telluride and alkynyl tellurides) could be synthesized in high yields and selectivities through the nanoparticles catalyzed C-Te cross-coupling reactions. We conclude this review by hoping that it will stimulate researchers to further research in this interesting field.

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