

Review

Recent Progress of Metal Nanoparticle Catalysts for C–C Bond Forming Reactions

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Abstract: Over the past few decades, the use of transition metal nanoparticles (NPs) in catalysis has attracted much attention and their use in C–C bond forming reactions constitutes one of their most important applications. A huge variety of metal NPs, which have showed high catalytic activity for C–C bond forming reactions, have been developed up to now. Many kinds of stabilizers, such as inorganic materials, magnetically recoverable materials, porous materials, organic–inorganic composites, carbon materials, polymers, and surfactants have been utilized to develop metal NPs catalysts. This review classified and outlined the categories of metal NPs by the type of support.

Keywords: metal nanoparticles; support; C–C bond forming reactions



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1. Introduction

The synthesis of new functional molecules and the development of new reactions are important fields at the core of synthetic organic chemistry. Among them, C–C bond formation is one of the most important reactions. Until now, numerous kinds of C–C bond forming reactions, including regioselective reactions, stereoselective reactions, and cross-coupling reactions such as Suzuki–Miyaura, Mizoroki–Heck, Stille, Hiyama, Ullmann, and Sonogashira coupling reactions, have been developed and applied to synthesize many kinds of functional molecules such as drugs, natural products, optical devices, and industrially important starting materials. Homogeneous transition metal catalysts act in a pivotal role to achieve the above reactions, a huge kind of catalysts and ligands have been developed. However, homogeneous catalysts have a number of drawbacks, in particular, the lack of reuse of the catalyst. This leads to a loss of expensive metals and ligands and to impurities in the products. Although numerous kinds of heterogeneous catalysts have been developed in order to address these problems, heterogeneous catalysts are inferior to homogeneous catalysts at some points, such as in reactivity.

On the other hand, transition metal nanoparticles (NPs), which can be readily obtained by several methods, such as chemical reductions and thermal decompositions, are of great interest due to their high reactivity derived from their extremely small size and their large surface to volume ratio. Metal NPs have received much attention for their use in many promising catalytic and biomedical applications because they exhibit unique magnetic and catalytic properties that are not shown in bulk materials. The pioneering catalytic application of metal NPs was reported by Rampino and Nord in 1941 [1]. Since the pioneering works on C–C coupling reaction by metal NPs were reported by Reetz in 1996 [2,3], over the last few decades, the use of transition metal NPs in catalysis has expanded considerably and a huge variety of metal NP catalysts have been developed. Due to the enormous number of publications outlining metal NPs in recent years [4–30], this review will only focus on the metal NPs used as a catalyst for C–C bond forming reactions over the last five years. This review classified and outlined the categories of metal NPs by the type of support, such as inorganic materials, magnetically recoverable materials, porous materials, organic–inorganic composites, carbon materials, polymers and surfactants (Scheme 1).

Inorganic materials	Metals, metal oxides, clays, etc.
Magnetically recoverable materials	Fe ₃ O ₄ , MFe ₂ O ₄ , materials functionalized on Fe ₃ O ₄ , etc.
Porous materials	Materials with porous structure such as zeolite and covalent organic frameworks, etc.
Organic-Inorganic composites	MOFs, inorganic materials functionalized by organic molecules, etc.
Carbon materials	Carbon, carbon nanotubes, graphene, graphene oxides, carbon nitrides, etc.
Organic polymers and surfactants	Polymers, dendrimers, plants extract, polysaccharides, surfactants, etc.
Others	Organic molecules with low molecular weight such as solvents and phosphines, etc.

Scheme 1. Category of support.

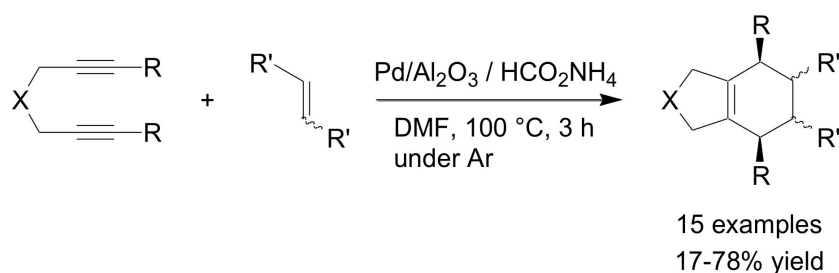
2. Several Supports for Metal Nanoparticles

2.1. Inorganic Materials

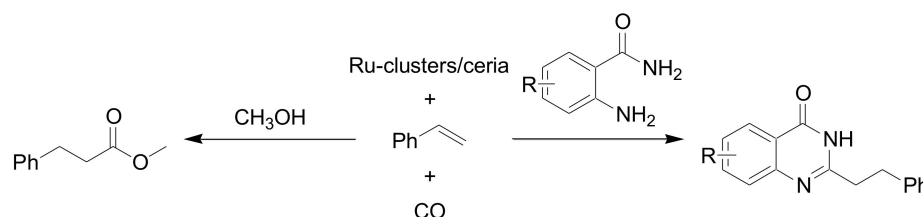
One of the most fundamental supports for metal NPs is inorganic materials such as metal oxides, clay, and so on [31–33]. There have been developed many highly active inorganic materials and supported metal NP catalysts, such as those which proceed the C-C bond forming reaction at room temperature [34–51]. Typical recent examples include the following:

Trudell et al. reported a reliable method for the encapsulation of Pd NPs in halloysite, and the resultant Pd@halloysite catalyzed the Suzuki-Miyaura coupling reaction of aryl bromides at room temperature [52]. Verberckmoes et al. found that uncalcined, co-precipitated hydrotalcite-supported Pd NP catalysts are preferred above the calcined, impregnated ones when catalyzing basic promoted organic reactions. The reason for this is that the lack of calcination resulting in an ordered and porous hexagonal hydrotalcite structure causes a high accessibility of the active centers in combination with the high support basicity [53]. Pd NPs stabilized on magnesium oxide–carbon quantum dots catalyzed the Suzuki-Miyaura coupling reaction of aryl bromides and chlorides at room temperature. In addition, Pd loading and leaching in catalysts can be estimated by using fluorescence emission because a good relationship was observed between fluorescence intensity and the loading of Pd [54].

Miura and Shishido et al. developed one-pot synthesis of cyclohexene derivatives from the reductive cycloisomerization of diynes and subsequent [4+2] cycloaddition with dienophiles, based on the hypothesis that Pd species functioned as a redox site to form a palladacycle via the oxidative addition of two alkyne moieties (Scheme 2) [55]. They also found that the [2+2+2] cycloaddition of alkynes proceeded smoothly by PdAu alloy NPs while monometallic Pd and Au NPs were ineffective [56,57]. In the hydroalkoxycarbonylation of olefins using Ru NPs on CeO₂, controlling the regioselectivities of linear esters and branched esters has been found to be related to the Ru size [58,59]. The Ru/Ceria catalyzed quinazolinones synthesis was also achieved by the same group (Scheme 3) [60].



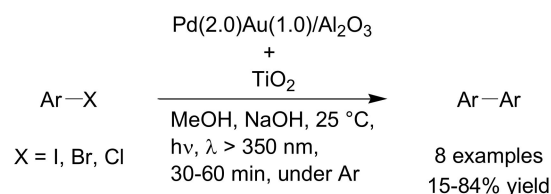
Scheme 2. One-pot synthesis of cyclohexene derivatives by Miura and Shishido et al. Reproduced from [55], Wiley-VCH: 2020.



Scheme 3. Three-component coupling reactions catalyzed by Ru-clusters/ceria. Reproduced from [58], Elsevier: 2020 and [60], Wiley-VCH: 2018.

Metal NPs have been also applied to polymerization reactions. Cu NPs supported on SiO_2 catalyzed the controlled living radical polymerization to afford poly(*n*-butyl acrylate) and poly(*n*-butyl acrylate-block-styrene) [61]. Pd NPs coated on the surface of the fabricated titanate nanotubes was utilized for electrochemical polymerization of *o*-phenylenediamine in acid medium [62].

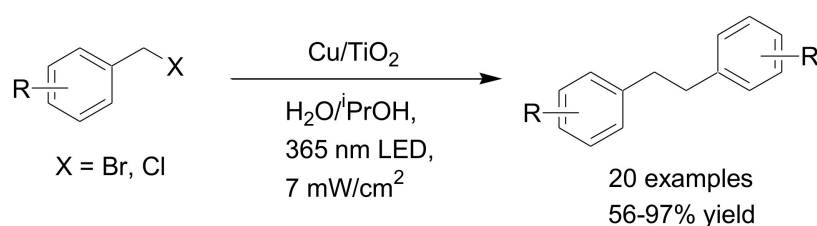
Huge efforts have been devoted to develop the metal NPs as a photocatalyst for C–C bond forming reactions. Hosseini-Sarvari et al. reported the synthesis of nano Pd/ TiO_2 with band gap >3 eV through a photodeposition method under sunlight, and their activity for Suzuki-Miyaura coupling reaction under visible light irradiation [63]. They also reported photocatalytic activity of nano Pd/ ZnO for Suzuki-Miyaura, Hiyama, and Buchwald-Hartwig reactions under visible light irradiation at ambient temperature [64]. The study of Lanterna and Scaiano et al. indicated that the direct excitation of Pd NPs can catalyze Sonogashira coupling reaction [65]. The same research group also found that the Ullmann coupling reaction of aryl iodides proceeded smoothly under both UV and visible light irradiation in the presence of Pd@ TiO_2 whereas a similar dose of visible light did not initiate the reaction [66]. The photocatalytic activity of Pd NPs supported on SiC in Sonogashira coupling reaction under visible light irradiation has been also reported. The rate of the photocatalytic reaction can be controlled by the light intensity and irradiation wavelength respectively [67]. Yoshida et al. found a blended catalyst of TiO_2 and Pd-Au/ Al_2O_3 showed high catalytic activity for photocatalytic Ullmann coupling reaction (Scheme 4). The photogenerated electrons on the TiO_2 photocatalyst reduced the aryl halide to generate the corresponding radical species, while the Pd-Au bimetallic nanoparticles activated another aryl halide and facilitated its reaction with the photogenerated aryl radical to yield biaryls. To improve of both the photocatalyst and the metal NPs independently is an attractive and promising approach to improve the entire catalytic performance [68]. The coupling reaction of pyridine with benzene and THF with alkane, alkene, and arene using Pt NPs or Pd-Au NPs loaded TiO_2 photocatalyst have been also reported by the same research group [69–72].



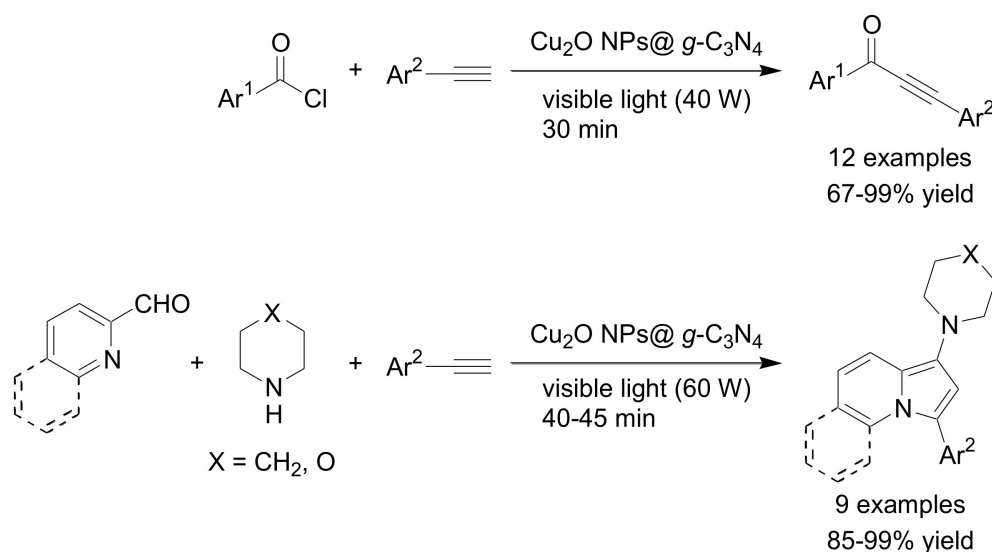
Scheme 4. Photocatalytic Ullmann coupling with the blended catalyst consisting of TiO_2 and Pd-Au/ Al_2O_3 . Reproduced from [68], RSC: 2018.

Surface structure engineering has afforded many breakthroughs in enhancing the photocatalytic activity of titania. Among them, readily accessible urchin-like structures with properties of an interconnected porous framework and a high specific surface area and can increase the efficiency of light harvesting as well as facilitate the accessibility of reactants to the active sites [73]. InP/ ZnS quantum dots were used as a sole photocatalyst to catalyze the C–C coupling reaction between 1-phenyl pyrrolidine and phenyl-*trans*-styryl sulfone without the aid of any cocatalyst or sacrificial oxidant or reductant [74]. The density

functional theoretical (DFT) calculations of the adsorption energies of reaction intermediates leads to a design of the photocatalyst. Su et al. found the Cu NP-modified TiO₂ presented a high selectivity towards photocatalytic homocoupling of benzyl bromide into bibenzyl with a remarkable apparent quantum efficiency (AQE) of 15% by evaluation of the adsorption energies of benzyl radicals and bromine atoms on a series of selected metal surfaces (Scheme 5) [75]. Cu₂O NPs supported on graphitic carbon nitride make a photoactive catalyst that has been developed for the preparation of ynone, aminoindolizines, and pyrrolo [1,2-a] quinoline. The electrons present in the conduction band under irradiation play an important role in enhancing the charge density of the Cu₂O NPs, which strengthens the π -complex between Cu₂O NPs and alkyne molecules, and acting as scavengers for the terminal hydrogen of alkyne to form the copper acetylide complex (Scheme 6) [76]. The size effect of Pt on the photocatalytic nonoxidative methane conversion efficiency was systematically investigated over x-Pt/Ga₂O₃ with the particle size (x) ranging from 1.5 to 2.7 nm, where a volcano-shaped relation was observed [77].

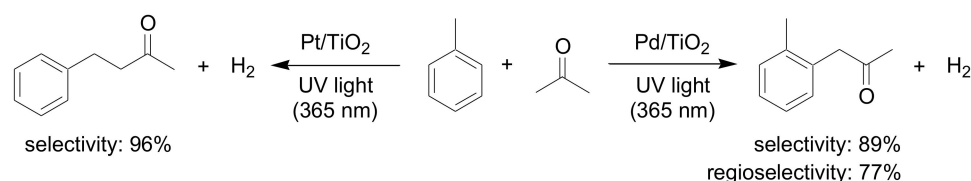


Scheme 5. Photocatalytic dehalogenative coupling of benzyl bromides using Cu/TiO₂ photocatalyst. Reproduced from [75], ACS: 2021.



Scheme 6. Cu₂O NPs/g-C₃N₄ catalyzed synthesis of ynones, aminoindolizines, and pyrrolo[1,2-a]quinolones. Reproduced from [76], ACS: 2020.

The nature of the metal nanoparticle cocatalyst deposited on a TiO₂ photocatalyst dictated the product selectivity for the cross-coupling. The reaction of toluene with acetone gave 1-(*o*-tolyl)propan-2-one in the presence of Pd NPs, while Pt NPs promoted the cross-coupling reaction between the methyl group of toluene and acetone to afford 4-phenylbutan-2-one (Scheme 7) [78].

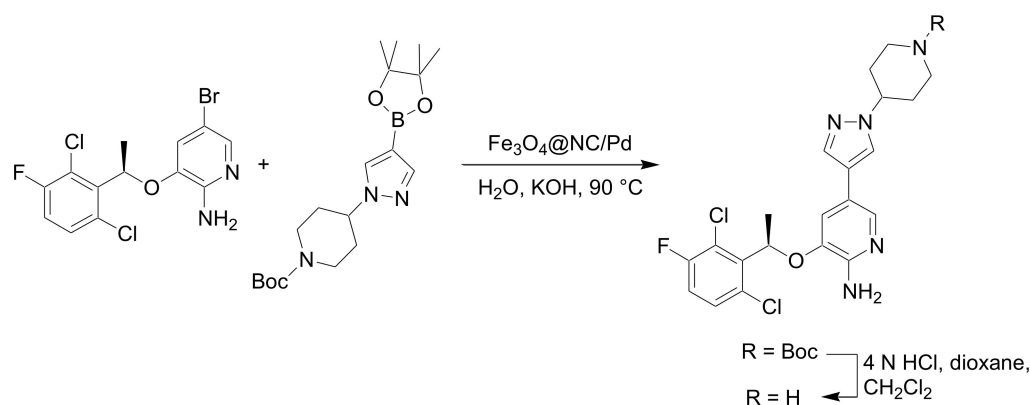


Scheme 7. Selective photoreaction using metal immobilized on TiO₂. Reproduced from [78], Springer: 2020.

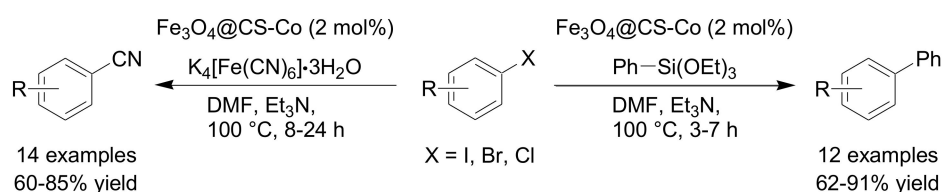
2.2. Magnetically Recoverable Materials

Metal NPs with a magnetic core can be easily separated from the reaction mixture by using an external magnet. Magnetic separation is an alternative to filtration or centrifugation as it prevents loss of catalyst and increases the reusability. This makes materials like Fe₃O₄ a promising support for nanocatalysts. Magnetic nanoparticles have received considerable attention in terms of biocompatibility, thermal stability against degradation, large surface area, and low cost. Therefore, it is suitable for designing magnetically retrievable metal NPs [79–86]. Lately, many excellent studies about magnetic composite nanocatalysts have been reported [87–118].

Nasseri et al. improved their bifunctional catalytic system based on copper with an ionic tail reported in 2019, and developed a magnetically water dispersible Cu-Co bimetallic catalyst for the efficient base/Pd-free C-C and C-N cross-coupling reactions [119,120]. Magnetite coated by amino acid functionalized chitosan in consideration of improving physicochemical properties such as solubility and mucoadhesiveness, was used as a support for Pd NPs. On using this catalyst, the Suzuki-Miyaura and Sonogashira coupling reactions were carried out efficiently at room temperature [121]. Based on this, a new catalytic system with an extremely low loading of expensive metal (ppm or ppb) has been developed. Eshghi et al. developed arginine-modified Fe₃O₄@carbon magnetic nanoparticles with highly dispersed Cu NPs and ppm levels of Pd [122]. To improve the catalytic activity, nitrogen-doped materials are used as a support [123,124]. According to this idea, Shen and Qiao et al. synthesized novel magnetically Fe₃O₄@Pd NPs by fixing Pd on the surface of nitrogen-doped magnetic nanocomposites (Scheme 8) [125]. Hajipour et al. synthesized magnetically separable nano-nickel catalysts, which catalyzed efficiently for fluoride-free Hiyama coupling reaction, through a “click” reaction of azide-functionalized magnetic nanoparticles with 2-ethynylpyridine followed by immobilization of nickel nanoparticles [126]. Co NPs immobilized on magnetic chitosan has been used for the first time for the cyanation of aryl halides, and also promoted the Hiyama coupling reaction (Scheme 9) [127].

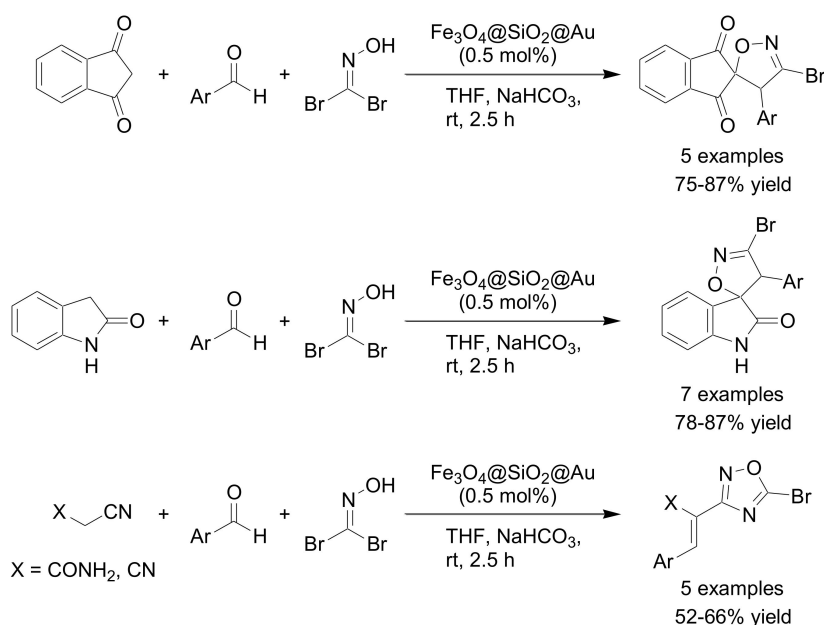


Scheme 8. Application of Fe₃O₄@NC/Pd in the synthesis of crizotinib. Reproduced from [125], MDPI: 2018.



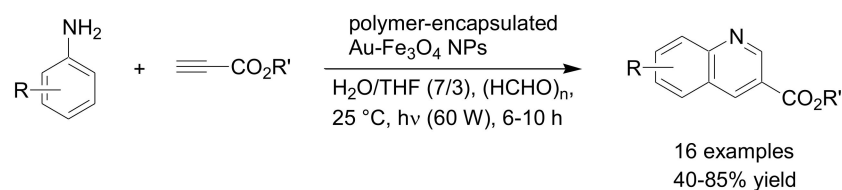
Scheme 9. Cyanations and Hiyama coupling reactions using Co NPs immobilized on magnetic chitosan. Reproduced from [127], ACS: 2020.

A Fe_3O_4 /oleic acid/Pd NPs catalyst was used for the Sonogashira polymerization. The polymerization occurred by a step-growth mechanism and resulted in a molar mass of 3.8 kg/mol [128]. The sequential Knoevenagel condensation/1,3-dipolar cycloaddition reactions proceeded using $\text{Fe}_3\text{O}_4@\text{SiO}_2@\text{Au}$ catalyst to give substituted spiroisoxazones and oxadiazoles with good regio- and stereoselectivity under mild reaction conditions (Scheme 10) [129].

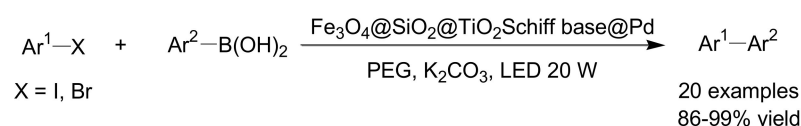


Scheme 10. Synthesis of spiroisoxazones and oxadiazoles using $\text{Fe}_3\text{O}_4@\text{SiO}_2@\text{Au}$ catalyst. Reproduced from [129], Elsevier: 2020.

Bhalla et al. developed a supramolecular porous ensemble consisting of oligophenylene derivatives and $\text{Au-Fe}_3\text{O}_4$. A series of catalysts efficiently catalyzed Kumada reaction, Heck reaction, and the synthesis of quinoline carboxylates (Scheme 11) [130–132]. Azadi and Kazemi et al. prepared the core-shell magnetic nano photocatalyst. The catalyst composed of a central magnetite core, an interlayer of silica, a shell of titania, and finally a Schiff base complex of Pd NPs. The Suzuki-Miyaura coupling reaction occurred under blue LED irradiation, while no product was obtained under a white and green LED irradiation (Scheme 12) [133].



Scheme 11. Photocatalytic synthesis of quinoline carboxylates through a $\text{C}(\text{sp}^2)\text{-H}$ activation reaction. Reproduced from [132], ACS: 2017.

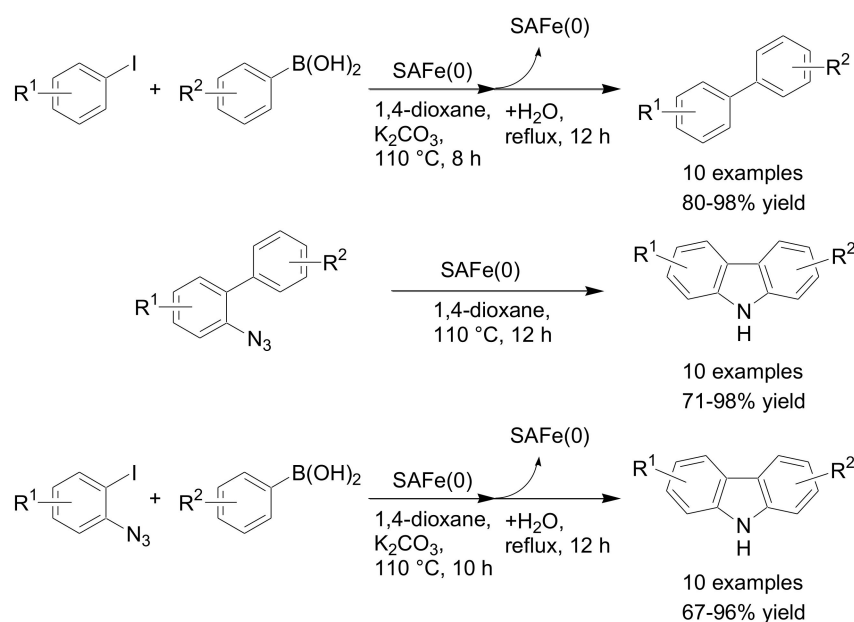


Scheme 12. Visible-light-driven photocatalytic Suzuki-Miyaura Coupling reaction. Reproduced from [133], Wiley-VCH: 2021.

2.3. Porous Materials

Several porous materials, such as zeolite, mesoporous silica, covalent organic frameworks (COFs), and microporous organic polymers (MOPs), have also been utilized as the support for metal NPs. These materials possess several highly desirable properties: pore topologies that possess long-range structural ordering, a uniform pore size, and high surface areas. Due to the typical advantages of the porous supports, the size control of the stabilized metal NPs and the selective reactions dependent on the pore size have been achieved [134–151].

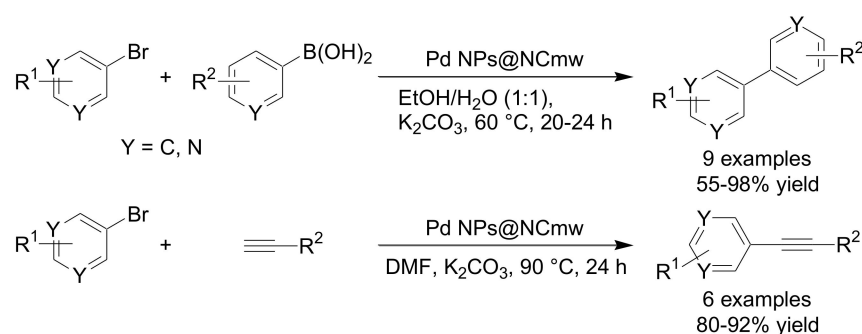
Li and Chen et al. confirmed that the number of acid sites within the zeolite frameworks were directly proportional to the catalytic activity of Pd NPs in Suzuki-Miyaura coupling reaction [152]. Gu and Zhang et al. have developed a novel covalent organic framework (COFs)-templated strategy for the size-controlled synthesis of stable and highly dispersed ultrafine metal NPs. With the aid of the evenly distributed thioether groups in the ordered framework structure, ultrafine metal NPs with a narrow size distribution were successfully obtained [153]. Arisawa et al. developed a well-established metal–nanoparticle catalyst preparative protocol by simultaneous in situ metal NPs and nanospace organization (PSSO). Several sulfur-modified Au-supported metal (Pd, Ni, Ru, and Fe) catalysts were constructed by self-assembled metal NPs, which were encapsulated in a sulfated *p*-xylene polymer matrix, and showed high catalytic activities for several C–C coupling reactions (Scheme 13) [154–158].



Scheme 13. One-pot synthesis of carbazole derivatives catalyzed by self-assembled multilayer Fe(0) NPs. Reproduced from [154], ACS: 2020.

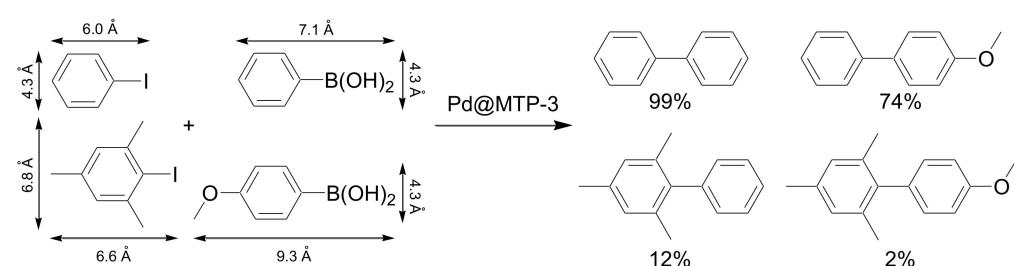
El-Shall et al. found that ultra-small CuPd bimetallic nanoparticles deposited on a mesoporous-fumed silica support could be participated efficiently to the Suzuki-Miyaura coupling reaction of aryl bromides to give the corresponding coupling product within 30 min [159]. Bae, Byun, and Kim et al. prepared small and large Au NPs stabilized in mesoporous TiO₂ and poly(*N*-isopropylacrylamide) particles, respectively. These Au NPs

exhibited a notably high catalytic activity in the homocoupling of phenylboronic acid, and interestingly, there was no obvious correlation between the apparent E_a values and the size of Au NPs [160]. Dewan et al. reported the first synthesis of a renewable, recyclable, environmental benign bio-nanocellulose-based honeycomb-like heterogeneous surface from waste pomegranate peel. Pd NPs loaded onto the bio-nanocellulose is the effective catalyst for C–C coupling reaction to synthesize the potential bioactive biaryl/heterobiaryl and alkynyl/heteroalkynyl derivatives (Scheme 14) [161].

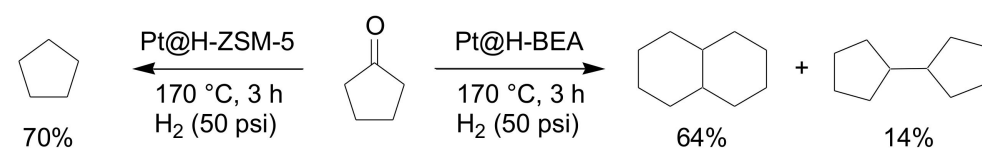


Scheme 14. Heteroaryl cross-coupling reactions catalyzed by Pd NPs@NCmw. Reproduced from [161], ACS: 2021.

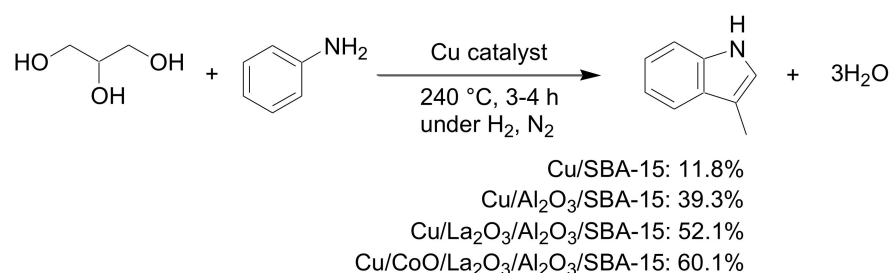
Xie et al. reported a one-step strategy for the design of size-selective heterogeneous catalysts, which was composed of microporous polymer carriers and ultrafine Pd NPs. This research will expand the application scope of microporous organic polymers in size-selective heterogeneous catalysis (Scheme 15) [162]. Product selectivity is attributable to the size selectivity of micropores. Pt NPs encapsulated in H-BEA zeolite (Pt@H-BEA) catalyzed a one-step conversion of biomass-derived cyclopentanone to C10 cyclic hydrocarbons, i.e., bicyclopentane and decalin. While cyclopentane was produced with a yield of >70% on Pt@H-ZSM-5, which have the narrower pores (Scheme 16) [163]., Shi et al. achieved the selective synthesis of 3-methylindole from the reaction of aniline with glycerin using Cu NPs/SBA-15 modified with Al_2O_3 , La_2O_3 , and CoO. The characterizations revealed that the effect of Al_2O_3 , La_2O_3 , and CoO to enhance the polarity of the carrier, weaken the acidity, and to increase the number of weak acid centers, respectively (Scheme 17) [164].



Scheme 15. Size-selective C–C coupling reactions catalyzed by microporous polymer stabilized Pd NPs. Reproduced from [162], ACS: 2021.



Scheme 16. Product selectivity controlled by pore size of zeolite-encapsulated Pt NPs. Reproduced from [163], ACS: 2020.

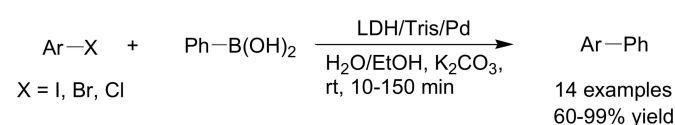


Scheme 17. Selective synthesis of 3-methylindole from the reaction of glycerin with aniline. Reproduced from [164], Springer: 2021.

2.4. Organic–Inorganic Composites

One of the most useful methods to obtain the inorganic materials with excellent properties as a support for metal NPs is functionalization of inorganic materials with organic molecules. On the other hand, metal–organic frameworks (MOFs), which includes a typical organic–inorganic composite, have attracted extensive attention as supports for metal NPs due to their huge surface area, large porosity, recyclability, and tunable functionality. Many kinds of metal NPs immobilized on the functionalized inorganic materials and MOFs have been reported until now [165–187].

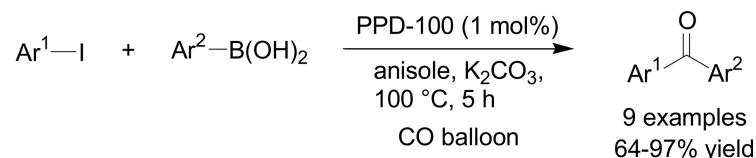
Malta et al. synthesized the hydroxypropylated α -, β -, or γ -cyclodextrins-stabilized Pd NPs supported on ceria, and compared the reactivity in Suzuki–Miyaura coupling reactions. The catalysts based on β - and γ -cyclodextrins-stabilized Pd NPs showed higher reactivities than α -cyclodextrins-stabilized Pd NPs, probably due to a higher degree of particles up to 5 nm [188]. A Suzuki–Miyaura coupling reaction took place smoothly at room temperature using thiourea-bridged periodic mesoporous organosilica and supported Pd NPs as a catalyst [189]. Ha et al. synthesized dual (temperature and pH)-responsive poly(*N*-isopropyl acrylamide-*co*-methacrylic acid) functionalized SBA-15. This material supported the fact that Pd NPs showed high catalytic activity for Suzuki–Miyaura coupling reactions at room temperature, while the activity decreased at higher temperature than LCST of PNIPAM [190]. It has been reported that Suzuki–Miyaura coupling reaction proceeded at room temperature using Pd NPs stabilized on CaAl-layered double hydroxide functionalized with tris(hydroxymethyl)aminomethane (Scheme 18) [191].



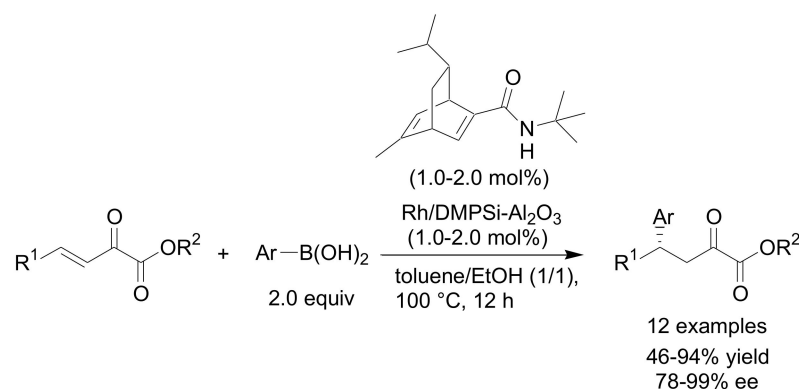
Scheme 18. The Suzuki–Miyaura cross-coupling reaction at room temperature catalyzed by LDH/Tris/Pd. Reproduced from [191], Elsevier: 2018.

Pd NPs stabilized on 12-tungstophosphoric acid modified zirconia catalyzed the Suzuki–Miyaura coupling reaction efficiently and TOF reached to $\text{ca. } 100000 \text{ h}^{-1}$ [192]. Pd NPs decorated into a biguanidine modified-KIT-5 showed high catalytic activity for Suzuki–Miyaura coupling reaction under sonication at room temperature. The coupling product was obtained efficiently within 15 min [193]. Pd NPs immobilized on zirconium phosphate glycine diphosphonate nanosheets was confirmed to be an effective catalyst for Suzuki–Miyaura and Mizoroki–Heck reaction, and was applicable to the flow system [194]. Pd NPs supported on the hybrid nanomaterials based on thiol functionalized halloysite nanotubes and highly cross-linked imidazolium salts showed high performance in Suzuki–Miyaura and Mizoroki–Heck coupling reactions, and TOF of $3.88 \times 10^6 \text{ h}^{-1}$ was achieved in Suzuki–Miyaura coupling reaction [195]. Control synthesis of polyacrylamide brushes grafted onto silica particles (SiO_2 -g-PAAm), which can be used for the support of Pd NPs was achieved using reversible addition–fragmentation chain transfer (RAFT) polymerization. Appropriate activity and recyclability of SiO_2 -g-PAAm-Pd indicated in the Mizoroki–Heck coupling reaction of iodobenzene with *n*-butyl acrylate [196]. In Ullmann-type aryl iodides

homocoupling, Au and Pd NPs loaded on ZIF-8 have been confirmed to be more efficient than the catalyst after calcination [197]. Kobayashi et al. developed poly(dimethyl)silane-immobilized metal NPs with alumina as a second support, and the resulting catalysts have been utilized in several reactions (Schemes 19 and 20) [198,199].

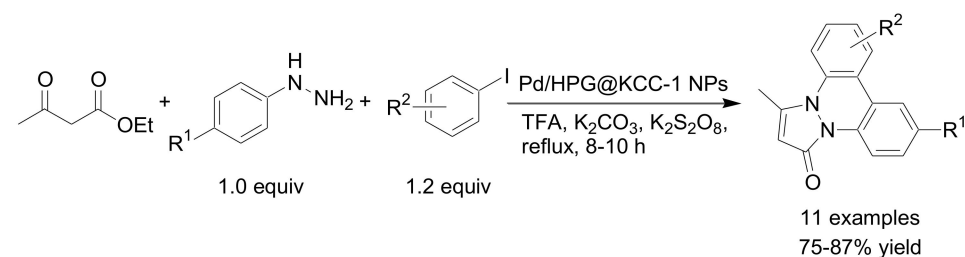


Scheme 19. The carbonylative Suzuki-Miyaura coupling reactions catalyzed by polysilane/ Al_2O_3 -immobilized Pd NPs. Reproduced from [198], Thieme: 2021.

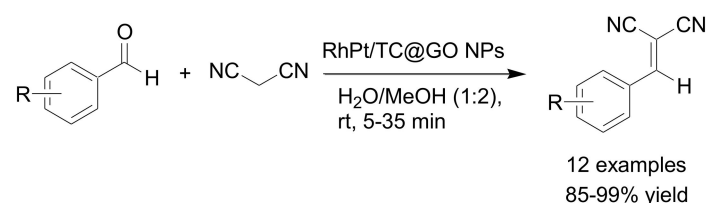


Scheme 20. Asymmetric 1,4-addition of arylboronic acids to β,γ -unsaturated α -ketoesters. Reproduced from [199], Wiley-VCH: 2020.

A one-pot synthesis of benzo[*c*]pyrazolo[1,2-*a*]cinnoline-1-ones was achieved with Pd NPs dispersed on octakis[3-(3-aminopropyltriethoxysilane)propyl]octasilsesquioxane functionalized fibrous nanosilica (KCC-1) (Scheme 21) [200]. Thiocarbamide-functionalized graphene oxide-supported RhPd NPs have been tried for the Knoevenagel condensation of malononitrile and aryl aldehydes and showed an excellent catalytic activity to give the product within 35 min at room temperature (Scheme 22) [201].



Scheme 21. Synthesis of benzo[*c*]pyrazolo[1,2-*a*]cinnoline-1-ones in the presence of Pd/HPG@KCC-1. Reproduced from [200], RSC: 2017.



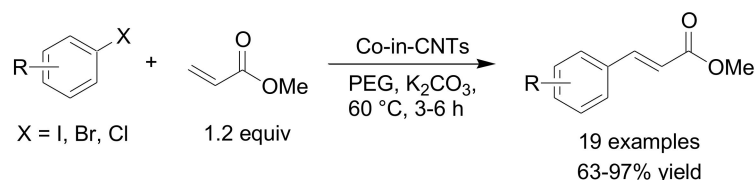
Scheme 22. Knoevenagel condensation at room temperature catalyzed by Rh-Pt NPs. Reproduced from [201], Elsevier: 2018.

Parida et al. reported that amino-functionalized Zr-based MOF (UiO-66-NH₂) was a suitable photocatalyst and support for metal NPs because it has a high surface area, tunable pores, and high thermal and chemical stabilities [202]. The same research group also investigated the utility of a graphene oxide/ZnCr-layered double hydroxide hybrid nanocomposite [203]. Chen and Wang et al. found that the porous coordination frameworks (PCFs) using the DIB-TETA as organic linkers and inorganic NPs as nodes exhibited superior photocatalytic performances in a noble metal-free Suzuki-Miyaura coupling reaction [204].

2.5. Carbon Materials

Charcoal is a classic commercial support for catalysts. Carbon materials have been proven to be suitable supports for heterogeneous catalysis, due to high thermal and chemical stability, their special electronic properties, and tunable textural properties such as surface area, porosity, and surface chemistry. To date, numerous kinds of metal NPs supported on carbon materials such as graphene and carbon nanotubes have been developed [205–231].

Ni@Pd core-shell NPs on carbon nanotubes (CNT) have been reported to show a high catalytic activity for carbonylative Suzuki-Miyaura cross-coupling reactions. The immobilization of the Ni@Pd NPs on CNT not only prevented their aggregation, but also significantly enhanced the accessibility of the catalytically active sites [232]. Hajipour and Farrokhpour et al. achieved the immobilization of Co NPs within a carbon nanotube channel, and found that Co NPs-in-CNTs, as compared to Co NPs-out-CNTs, exhibited excellent activity for Mizoroki-Heck reactions (Scheme 23) [233]. Chung et al. decorated Rh NPs on fullerene C60 to obtain a highly efficient nanocatalyst for Suzuki-Miyaura coupling reactions [234]. Chen and Li et al. designed an electron-deficient Au NPs-based catalyst via Schottky contact with boron-doped carbons for room temperature Stille coupling reaction. The electron-deficiency of Au NPs significantly increased the activation of C-Br bonds in alkylbromides and successive coupling reaction with allylstannanes [235].

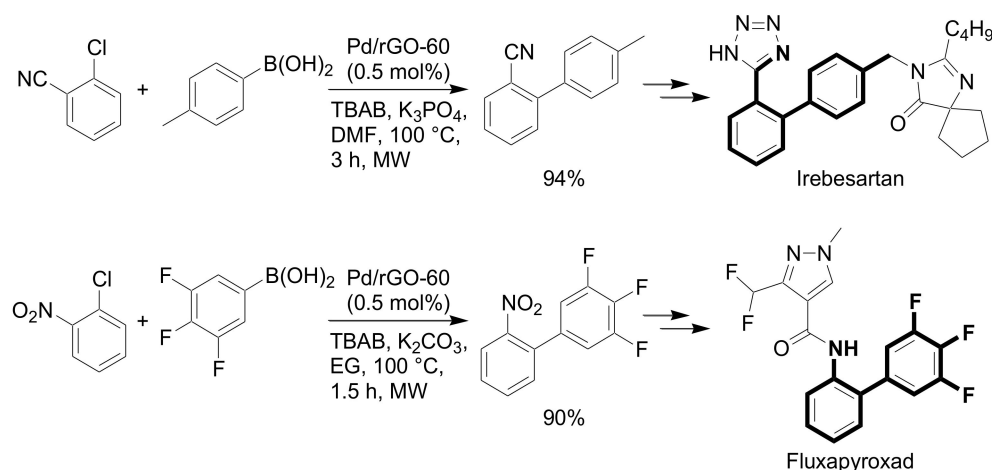


Scheme 23. Heck reaction with carbon-nanotube-encapsulated Co NPs. Reproduced from [233], RSC, 2019.

Astruc et al. successfully immobilized $\alpha\text{-Fe}_2\text{O}_3$ nanocluster on graphene oxide (GO) by utilizing the supramolecular interaction between amphiphilic tris(triazolyl)-polyethylene glycol and GO. $\alpha\text{-Fe}_2\text{O}_3/\text{GO}$ worked well as a catalyst for Suzuki-Miyaura coupling reaction with only parts-per-million loading [236]. Taniike et al. revealed that a graphene oxide framework prepared with benzene 1,4-diboronic acid as a two-side linker was a superior support of Pd NPs to that with phenylboronic acid as a one side linker [237].

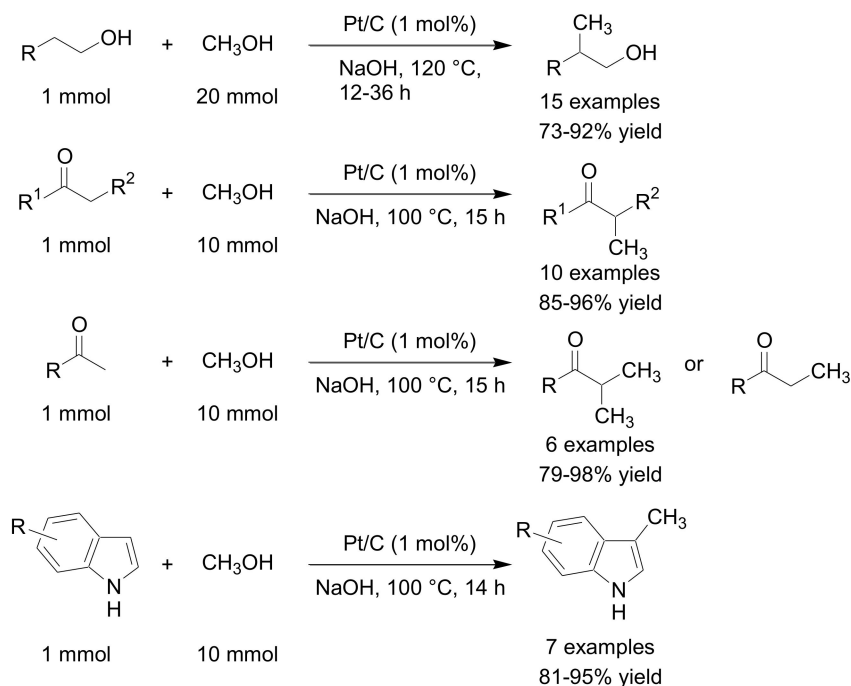
The coupling reaction of aryl chlorides can be achieved by using reduced graphene oxide-supported Pd NPs. The size of NPs and reactivity was dependent on the preparation temperature, and this catalyst was applied for the synthesis of key intermediates of important Sartans and Fluxapyroxad medicines (Scheme 24) [238]. Hoseini et al. utilized self-assembly at the toluene–water interface to produce a PdNiZn nanosheet and PdNiZn/reduced graphene oxide (rGO) ultrathin spherical NPs. The presence of rGO enhanced the catalytic activity, probably due to altering the electronic properties [239]. Ni NPs supported reduced graphene oxide, which has also been prepared by the hydrothermal process and investigated the catalytic activity for the homocoupling of arylboronic acids and alkynes [240]. Graphene acid is a convenient platform for the surface anchoring of Pd NPs with a narrow and sharp distribution. The size of NPs can be easily con-

trolled by the amount of the Pd precursor, and the catalyst showed a high activity in the Suzuki-Miyaura coupling reaction and oxidative homocoupling of arylboronic acids under environmentally friendly conditions [241]. Three-dimensional graphene, which has excellent properties such as ultrahigh surface-to-volume ratio, high porosity, low density, etc., was used for the effective support of Au NPs and PdCo-bimetallic NPs [242,243].

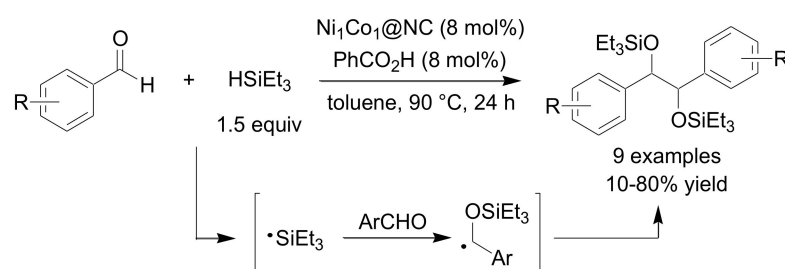


Scheme 24. The synthesis of the intermediate of Sartans and Fluxapyroxad. Reproduced from [238], RSC: 2020.

C-methylations of alcohol, ketones, and indoles have been achieved using methanol and Pt/C as a sustainable C1 source and a catalyst, respectively. The reaction is driven by a borrowing-hydrogen mechanism (Scheme 25) [244]. Nitrogen-doped carbon-encapsulated Ni/Co NPs catalyzed pinacol couplings have been reported. The reaction mechanism is different to the classical pinacol coupling pathway, and the initial formation of silyl radicals is proposed (Scheme 26) [245].

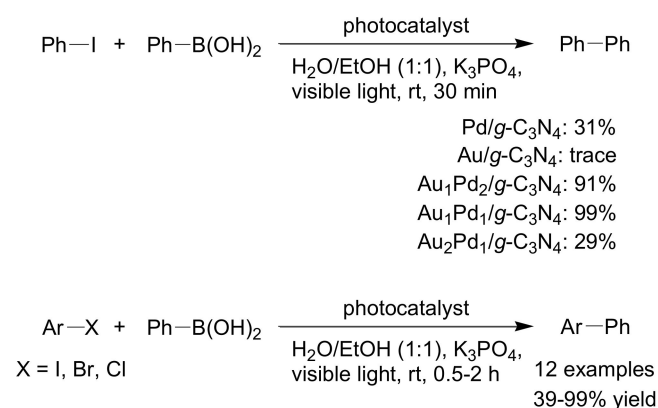


Scheme 25. C-methylation of alcohols, ketones, and indoles with methanol as a C1 source. Reproduced from [244], ACS: 2018.



Scheme 26. Silylative Pinacol coupling. Reproduced from [245], ACS: 2018.

Graphitic carbon nitride-supported Pd NPs ($g\text{-C}_3\text{N}_4/\text{Pd}$) is an efficient photocatalyst for Suzuki-Miyaura cross-coupling reactions. It has been confirmed that the reaction is driven by the light because the conversion of iodobenzene has the same trend as the absorption of light by the $g\text{-C}_3\text{N}_4/\text{Pd}$ [246]. Dabiri et al. prepared AuPd alloy NPs immobilized on graphitic carbon nitride sheets, which enhanced Suzuki-Miyaura cross-coupling reactions at room temperature under visible-light irradiation. The photocatalytic activities strongly depend on the Au:Pd ratio of the alloy NPs. The activity of $\text{Au}_1\text{Pd}_1/g\text{-C}_3\text{N}_4$ was much higher than that of the catalysts, compared with other Au:Pd ratios, probably because the electron transfer between the two metals occurs efficiently in the alloy NPs with an Au:Pd weight ratio near 1:1 (Scheme 27) [247]. Lim et al. investigated the role of the graphene interface in the photocatalyst, and found that the fast electron transfer was achieved in the presence of the reduced graphene oxide layer. Consequently, the highest catalytic activity for the visible-light induced C–C coupling reaction was obtained with Pd-nanodot-modified reduced GO-coated Au NPs [248].



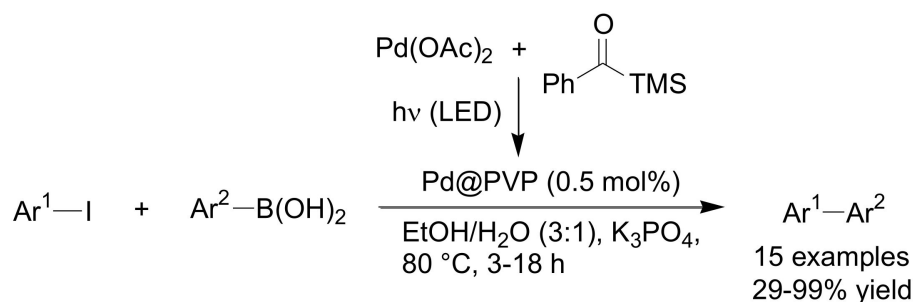
Scheme 27. Visible-light-enhanced Suzuki-Miyaura cross-coupling reactions. Reproduced from [247], Elsevier: 2020.

2.6. Organic Polymers and Surfactants

Polymers such as poly(vinylpyrrolidone) (PVP) and surfactants including quaternary ammonium salt with a long alkyl chain are commonly used as stabilizers in the synthesis of metal NPs. For example, Rampino et al. used poly(vinyl alcohol) (PVA) to protect Pd and Pt NPs in 1941, and El-Sayed et al. initially reported the use of Pd nanoparticles stabilized by PVP as catalysts in the Suzuki-Miyaura coupling reaction of aryl iodides with arylboronic acids in aqueous media [1,249]. Dendrimers are also often utilized as the stabilizer for metal NPs, and pioneering studies were reported by Crook, Tomalia, and Esumi [250–252]. On the other hand, as a greener process, phytosynthesis that utilizes parts of whole plants to synthesize metal NPs is also under exploitation and is an advantageous and profitable approach [253]. Numerous kinds of metal NPs stabilized by organic compounds with a high molecular weight have been reported [254–294].

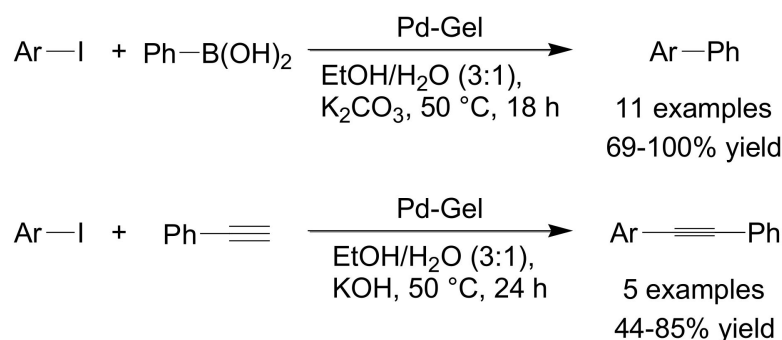
Peinemann et al. prepared Pd NPs with a subnanometer size (<1 nm) supported within the highly cross-linked network, which catalyzed Suzuki-Miyaura coupling reactions at a low temperature (<40 °C) [295]. Pd NPs stabilized by heteroatom donor-decorated

polymer immobilized ionic liquid catalyzes the Suzuki-Miyaura coupling reaction of aryl bromides with remarkable efficacy in aqueous media under exceptionally mild conditions. Improvements in catalyst performance arising from the introduction of PEG are attributed to an increase in dispersibility and/or solubility, facilitating access to more exposed active sites [296]. The room temperature Suzuki-Miyaura coupling reactions have been confirmed in the presence of Pd NPs supported by polydopamine and Pd NPs synthesized using *Sapindus mukorossi* seed extract [297,298]. Studer et al. prepared Pd NPs by visible light irradiation to the DMF solution of silyl ketones and Pd(OAc)₂. The diameter of Pd NPs could be adjusted to 1.9 to 5.2 nm depending on the photoinitiator used (Scheme 28) [299]. Pd NPs stabilized on poly(*o*-aminothiophenol) prepared by oxidation polymerization of *o*-aminothiophenol in the presence of Pd(NO₃)₂ showed a high catalytic activity for the Suzuki-Miyaura coupling reaction of aryl chloride in water [300]. The amphiphilic property of the eumelanin support helps Pd NPs to catalyze the Suzuki-Miyaura coupling reaction in water through a hydrophobic effect [301]. A series of Pd_xM_{147-x} (M = Cu, Pt, Au, Rh, Ru) stabilized on poly(amidoamine) dendrimers were synthesized and their catalytic activities were investigated in Suzuki-Miyaura coupling reactions. Pd₇₄Cu₇₃ DEN showed a similar activity to Pd₁₄₇ DEN and DFT calculations illustrated that the similar activity of the Pd₁₄₇ and Pd₇₄Cu₇₃ DENs originate from the comparable energy barriers of the rate-determining steps [302].



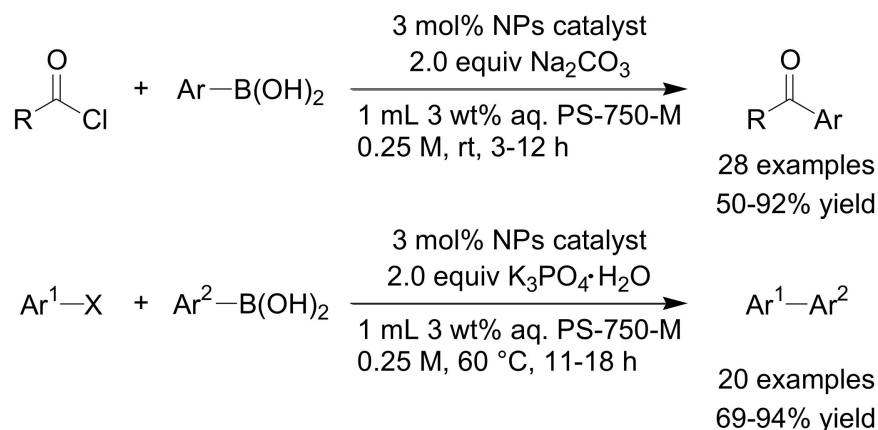
Scheme 28. The catalytic activity of Pd NPs prepared under visible light irradiation. Reproduced from [299], ACS: 2018.

Thang et al. developed the facile preparation method of polymer–metal nanocomposites for an improved catalytic performance by utilizing ultrasound as both the initiation and reducing source. Metal NPs were immobilized on the hydrophilic shell of the polymer matrix, and the size of the NPs were closely related to the ratio of tertiary amine groups in the polymer matrix to metal atoms [303]. Highly efficient Tsuji-Trost allylations in water have been achieved using Pd NPs stabilized by PVP. A very high TON of 537,000 was obtained in this system [304]. Yu et al. obtained the effective catalysts, which showed high activity for carbonylative Sonogashira coupling reactions by the introduction of salen moieties into highly cross-linked polyacrylamide [305]. Pd NPs were encapsulated within hybrid hydrogels made from an acylhydrazide-functionalized 1,3:2,4-dibenzylidene sorbitol (DBS-CONHNH₂) low-molecular-weight gelator combined with agarose polymer gelator via an in situ reduction of Pd(II). These heterogeneous gel-phase catalysts were successfully applied for several C–C coupling reactions (Scheme 29) [306–308]. Simple hydrophobic polymers without a coordination site such as polystyrene and poly(tetrafluoroethylene) have been confirmed to stabilize metal NPs and polymer-supported metal NPs were applicable to the recyclable catalyst for several reactions in water [309–313].



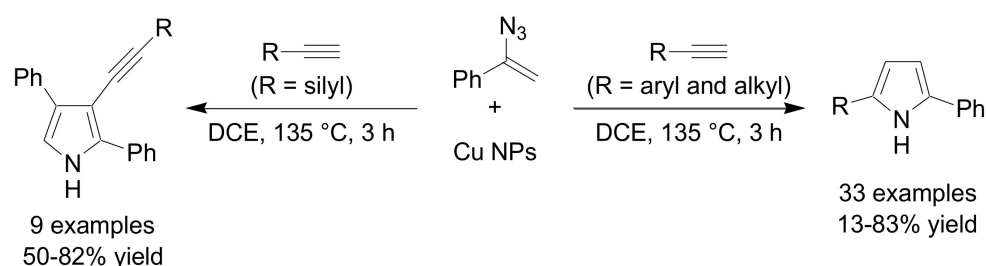
Scheme 29. Pd NPs@hybrid hydrogels catalyzed C–C coupling reactions. Reproduced from [307], Elsevier: 2020, and [308], RSC: 2018.

Oble and Rieger et al. synthesized a nanostructured well-defined core-shell nanogel with the ability to stabilize Pd NPs in its core by using reversible addition-fragmentation chain-transfer (RAFT)-mediated aqueous dispersion polymerization [314]. One of the most effective catalytic systems is micellar catalytic systems, which have been developed and expanded by Lipshutz and Handa groups. In their reaction systems, several reactions proceed at room temperature by designing an appropriate surfactant which form a micellar reaction field in water (Scheme 30) [315–328].

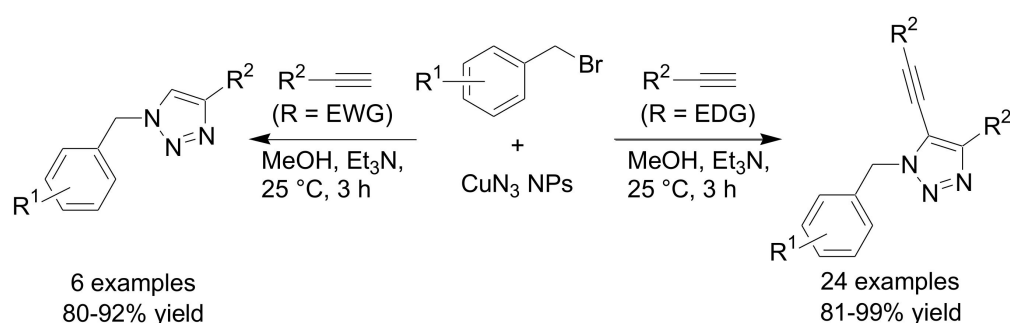


Scheme 30. One example of an efficient coupling reaction by micellar catalysts. Reproduced from [316], ACS: 2021.

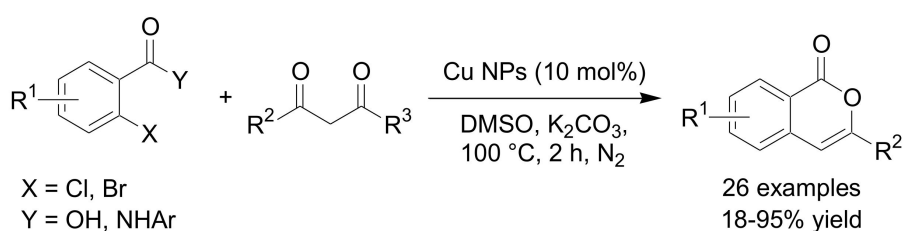
Yang and Jiang et al. reported a Cu NPs-catalyzed substrate-dependent chemodivergent transformation of vinyl azides with a terminal alkyne. 2,5-Disubstituted pyrroles were selectively obtained with aryl and aliphatic alkynes, whereas silylated alkynes afforded 2,3,4-trisubstituted pyrroles (Scheme 31) [329]. Substrate-dependent chemodivergent was also observed in the reaction of substituted benzyl bromides with terminal alkynes catalyzed by CuN₃ NPs. The electron donating group containing terminal alkyne produced 5-alkynyl 1,4-disubstituted triazoles whereas for alkynes with terminal electron withdrawing group facilitated the formation of 1,4-disubstituted triazoles (Scheme 32) [330]. The synthesis of 3-substituted isocoumarins from 2-chlorobenzoic acids and 1,3-diketones have been achieved utilizing Cu NPs. When 2-bromo-*N*-phenylbenzamide was used as a substrate, the reaction completed within 15 min (Scheme 33) [331]. Metal NPs can be applicable for the multi steps reactions. For example, Abdolmohammadi et al. reported the synthesis of [1]benzopyrano[*b*]pyridine-3-carbonitriles through Knoevenagel condensation, Michael addition, cyclization, tautomerization, and aromatization (Scheme 34) [332]. Trzeciak et al. have developed DNA-stabilized metal NPs and reported their catalytic applications in several C–C bond forming reactions such as Suzuki-Miyaura, carbonylative Sonogashira, and hydroformylation [333–337].



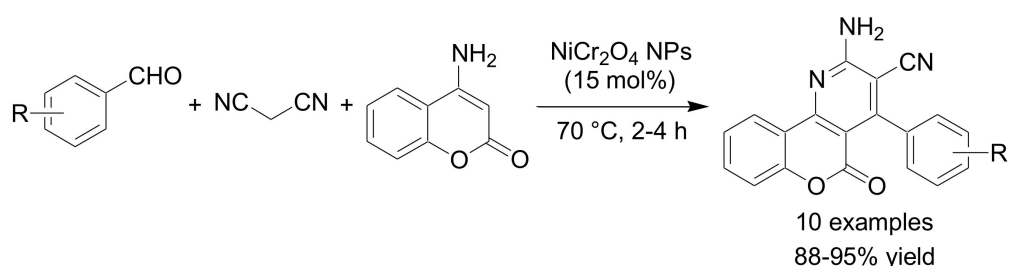
Scheme 31. Switchable reactivity between vinyl azides and terminal alkynes. Reproduced from [329], ACS: 2019.



Scheme 32. Alkyne-dependent synthesis of 1,2,3-triazole derivatives. Reproduced from [330], Nature Research: 2020.

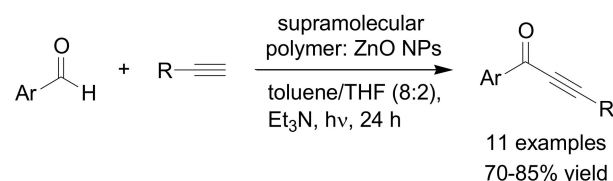


Scheme 33. One-pot synthesis of isocoumarin derivatives. Reproduced from [331], Elsevier: 2017.



Scheme 34. One-pot synthesis of [1]benzopyrano[b]pyridin-3-carbonitrile derivatives. Reproduced from [332], Taylo&Francis: 2020.

Bhalla et al. found that Supramolecular polymer of perylene bisimide derivative and ZnO NPs exhibited remarkable efficiency in direct dehydrogenative cross-coupling between terminal alkynes and aldehydes for the synthesis of ynones under visible light irradiation (Scheme 35) [338]. They also found that thiophene appended perylene bisimide derivative undergoes oxidative polymerization in the presence of gold ion to generate supramolecular polymeric ensemble, which showed photocatalytic activity in Mizoroki-Heck reaction [339].

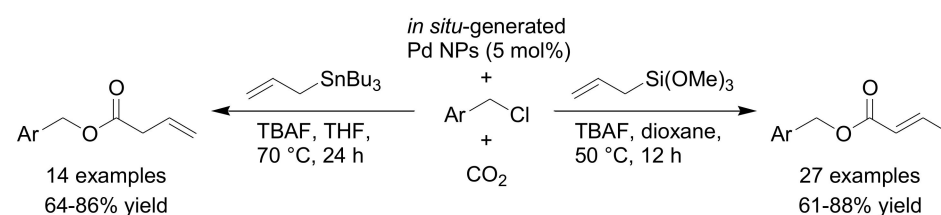


Scheme 35. One-pot synthesis of [1]benzopyrano[b]pyridin-3-carbonitrile derivatives. Reproduced from [338], RSC: 2018.

2.7. Others

As seen, the pioneering works of Reetz and Jeffery [2,340], organic molecules and simple tetraalkylammonium salts are also able to be used as stabilizers for metal NPs [341]. The use of ionic liquids alone as NP stabilizers and reaction media for the Suzuki-Miyaura coupling reaction was also shown to be efficient [342]. The introduction of nitrogen- and phosphorus-based molecules is also of special interest, because they not only act as a stabilizer of metal NPs, but also as ligands, which enhance the catalytic activity of metal NPs. The metal NPs stabilized by small molecules were used not only as prepared ones, but also as in situ generated ones [343–361].

TPPTS (triphenylphosphine-3,3',3''-trisulfonic acid trisodium salt), which is one of the most well-known water-soluble phosphine ligands acted as not only as a stabilizer, but also as an activator for Pd NPs. Pd NPs/TPPTS catalyzed the Suzuki-Miyaura coupling reactions of aryl bromides at room temperature to afford the coupling product within 1 h [362]. In situ-generated Pd NPs by gallic acid was an extremely simple, green, and active catalyst, which catalyzed C–C coupling reactions at room temperature [363,364]. Kumar et al. developed a selective synthesis of Pd₉Te₄ and PdTe, which are applicable for the catalyst in the Suzuki-Miyaura coupling reactions of aryl chloride [365]. By the same research group, a series of bidentate organochalcogen ligands (N, E; E = S/Se) were synthesized and they investigated the applicability for the support of Pd NPs [366,367]. Sewald et al. confirmed that solvent-stabilized Pd NPs were applicable for bio-orthogonal side-chain derivatizations of amino acids [368]. In situ-generated Pd NP-catalyzed three-component coupling of chloromethylarene with allyltrimethoxysilane and carbon dioxide (i.e., a carboxylative Hiyama coupling reaction) successfully produced α,β -unsaturated esters, whereas the coupling reaction with allyltributylstannane (i.e., a carboxylative Stille coupling reaction) gave β,γ -unsaturated esters (Scheme 36) [369,370].

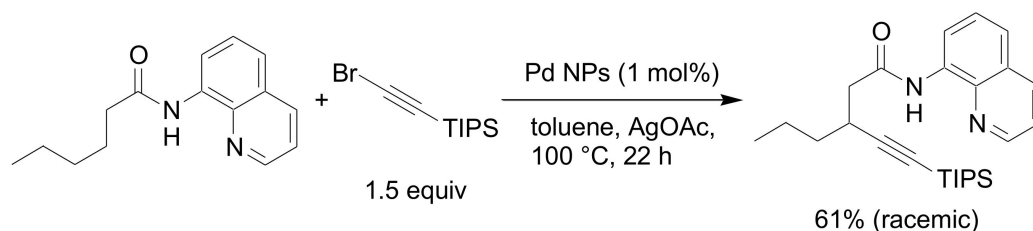


Scheme 36. The difference between carboxylative Stille coupling reaction and carboxylative Hiyama coupling reaction. Reproduced from [369], Wiley-VCH: 2017, and [370], ACS: 2013.

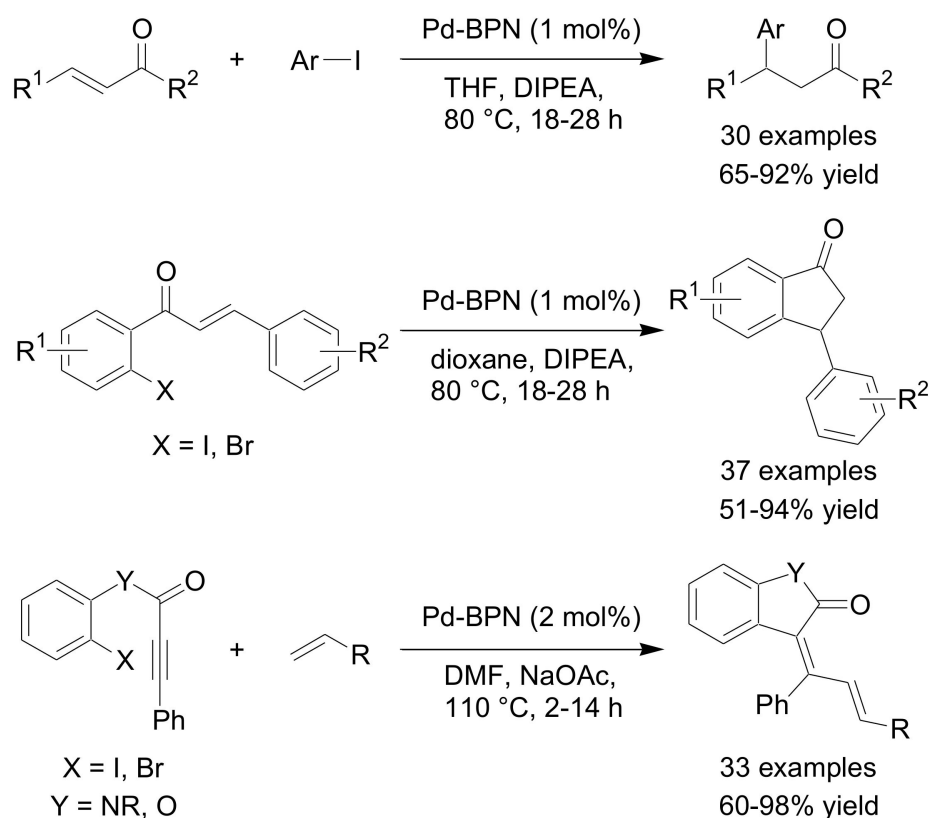
Sathish and Praveen et al. developed a supercritical processing method for the preparation of Au NPs, and found that the resultant Au NPs showed a high catalytic activity for Suzuki-Miyaura and Sonogashira coupling reactions [371]. The catalytic activity of Co₃O₄ in the Mizoroki-Heck reaction was confirmed by the Bagherzadeh group [372].

Tanaka et al. achieved the β -alkynylation of amides via C(sp³)-H activation using Pd NPs stabilized by bis[N,N'-(2-indalolyl)]-1,5-diazacyclooctane (Scheme 37) [373]. Wu et al. have established a cascade alkynylation and selective hydrogenation catalyzed by covalent binaphthyl-stabilized Pd NPs to provide a novel and highly efficient methodology for accessing Z and Z,Z-selective phosphinyl [3]dendralenes [374]. Binaphthyl-stabilized Pd NPs were also utilized to synthesize diphenylallylidene-methylindolin-2-ones, indanone derivative, 3-allylidene-2(3H)-oxindoles, and 3-allylidene-2(3H)-benzofuranones

(Scheme 38) [375–378]. Obora et al. found that Ir NPs showed excellent catalytic activity in β -(aryl)methylation of alcohol [379,380]. Feng, Wang, and Bao et al. developed an efficient method for the selective synthesis of δ -lactone from the telomerization of 1,3-butadiene with CO_2 . This reaction was catalyzed by ultrasmall Pd NPs generated in situ [381]. Telmisartan-stabilized Cu NPs were utilized to synthesize naphtho[2,3-*g*]phthalazine derivatives as potential inhibitors of tyrosinase enzymes [382].



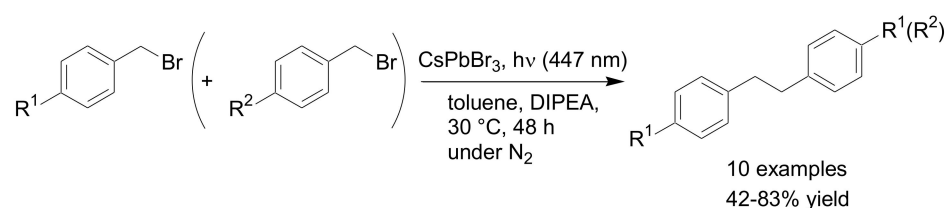
Scheme 37. C(sp³)-H activation catalyzed by Pd NPs. Reproduced from [373], Thieme: 2018.



Scheme 38. Synthesis of indanone derivative, 3-allylidene-2(3H)-oxindoles, and 3-allylidene-2(3H)-benzofuranones catalyzed by binaphthyl-stabilized Pd NPs. Reproduced from [376], ACS: 2020, and [377,378], Wiley-VCH: 2019,2017.

Ćirić-Marjanović et al. have reported the oxidative polymerization using $\text{H}_2\text{O}_2/\text{Fe}_3\text{O}_4$ NPs in an oxidant/catalyst system. Ammonium peroxydisulfate (APS) was used as an initiator in these systems [383,384]. Ultrahigh molecular weight poly(methyl methacrylate) was synthesized using 2-bromoisobutyric acid ethyl ester (EBiB) in the presence of Pd NPs. The polymerization was initiated by the radicals produced from the reaction of EBiB with Pd NPs [385].

Galian, Lloret-Fillol, and Pérez-Prieto et al. found that colloidal CsPdBr_3 perovskite NPs are suitable as photosensitizers for photoredox catalytic homo- and cross-coupling of benzyl bromides at room temperature with TON up to 17500 (Scheme 39) [386].



Scheme 39. Homo- and cross-coupling of benzyl bromides catalyzed by CsPbBr₃ perovskite. Reproduced from [386], RSC: 2020.

3. Conclusions and Perspective

C–C bond forming reactions have been widely utilized to synthesize many kinds of functional molecules such as biomaterials and natural products, fine chemicals, and medicines. On a small scale, these reactions are generally taken place with homogeneous catalysts utilizing the eminent advantages of high activity and selectivity. However, the homogeneous catalysts are not generally appropriate for industrialization because of their problems encountered in higher cost, stability, separation, and reusability. On the other hand, recyclable heterogeneous catalysts have some disadvantages, such as low activity and selectivity, the requirement of severe reaction conditions, and the leaching of metal species.

Metal NPs are expected to improve the above limits in industrialization. This review has outlined recent advances in metal NPs, which were used in the C–C bond forming reactions. Many types of support, such as inorganic materials, magnetically recoverable materials, porous materials, organic–inorganic composites, carbon materials, polymers and surfactants have been utilized to develop the metal NP catalysts. In each support, the excellent metal NPs which proceeded the C–C bond forming reactions, even at room temperature, or showed the photocatalytic activity, have been developed. In addition, most of them showed a high recyclability, and metal NP-catalyzed regio- and stereoselective reactions have been also developed. However, most of the reactions outlined in this review have been performed with catalysts at a mole percent loading (0.1 mol%–10 mol%). For industrial-scale applications, the use of an extremely low loading of catalysts is of great importance. For most catalysts (especially in the case of Pd NPs), the catalytic activity has been confirmed in well-known coupling reactions such as Suzuki–Miyaura, Mizoroki–Heck, Hiyama, Stille, Ullmann, and Sonogashira coupling reactions. In other words, new reactions catalyzed by metal NPs are not well developed. Considerable efforts over the last few decades have led to the development of metal NP catalysts which overcome various drawbacks such as reactivity and reusability. I hope that metal NPs will make a significant contribution to industry in the near future by further developments such as the metal NPs with a reactivity and selectivity that surpasses those of homogeneous catalysts, the new reaction peculiar to metal NPs, and the reaction systems with extremely low loadings of catalysts.

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Conflicts of Interest: The author declare no conflict of interest.

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