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Review Article

Recent Development in the Synthesis of 1,2,3-Triazole Derivatives Using Nanocatalysts

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Abstract

1,2,3-Triazole is an aromatic, five-membered, π-excessive heterocycle comprised of three regular nitrogen atoms. 1,2,3-Triazoles possess a broad spectrum of bioactivities such as anticancer, antimicrobial, anti-inflammatory, antidiabetic, antiviral, and anti-HIV activity. Several compounds containing 1,2,3-triazole-moiety have been employed as drugs in the market. In organic synthesis, these heterocycles also play an important role as building blocks for different transformations. Furthermore, applications of 1,2,3-triazole derivatives as agrochemicals, corrosion retardants, polymers, optical brighteners, photostabilizers, pigments and metal chelators have also been well reported. Due to a wide range of applications, the synthesis of 1,2,3-triazoles has attracted tremendous research interest of chemists and a huge number of studies on the synthesis of these heterocycles have been published over the years. In this review article, we focus on the use of nanocatalysts for the synthesis of 1,2,3-triazoles. 70 studies on the synthesis of 1,2,3-triazoles using nanocatalyst from 2014 have been collected and analyzed. We also try to describe reaction mechanisms as much as we can. The study might be useful for chemists who work in heterocyclic synthesis or medicinal chemistry.

Keywords: Recyclability; terminal alkynes; boronic acids; click reaction; regioselectivity

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1. Introduction

1,2,3-Triazoles are aromatic five-membered ring heterocycles containing three successive nitrogen atoms. These heterocycles widely appeared as synthetic sources. The 1,2,3-Triazoles possess a diverse range of bioactivities such as anticancer [1-3], antimicrobial [4,5], anti-inflammatory [6,7], antidiabetic [8,9], antiviral [10,11], and anti-HIV activity [12,13]. Several 1,2,3-triazole-based compounds have been employed as drugs in the market (Figure 1). This subclass of heterocycle also plays an important role in organic synthesis by participating in different transformations such as the transannulation, triazole ring opening, triazole directing group assisted C-H activation and the C-H activation-based triazolation [14-26]. Several compound containing 1,2,3-triazole moiety can have multiple coordination modes with transition metals [27-29]. In material field, the use of 1,2,3-triazoles as agrochemicals, corrosion retardants, polymers, optical brighteners, photostabilizers, pigments and metal chelators have also well documented [30-36]. With such a wide range of applications, the synthesis of 1,2,3-triazoles has drawn great attention of chemists and a huge number of studies on the synthesis of 1,2,3-triazoles have been reported over the years. The synthesis of 1,2,3-triazoles has also well summarized in several review articles, however

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none of them specialized on 1,2,3-triazole synthesis using nanucatalyst [37-39]. In this review article, we focus on the use of nanocatalysts for the synthesis of 1,2,3-triazoles in a decade. References have been collected from various resources such as Google Scholar, SciFinder, and PubMed. 70 studies on the synthesis of 1,2,3-triazoles using nanocatalyst from 2014 have been collected and analyzed. We also try to described reaction mechanisms as much as we can. The use of nanomaterials as catalysts for the synthesis of other heterocycles were well summarized in the literature [40-43]. The study might be useful for chemists who work in heterocyclic synthesis or medicinal chemistry.

2. Copper Nanomaterials as Catalysts for the Synthesis of 1,2,3 -triazoles

In 2014, Albadi *et al.* described the synthesis of CuO/ZnO nanoparticles by co-precipitation method and characterization of the material by BET surface area, XRD, SEM, TEM and EDS analysis. The material then was used as efficient recyclable catalyst catalysts for the synthesis of 1,2,3- triazoles from benzyl halides, sodium azide, and terminal alkynes (**Scheme 1**) [44]. Simple work-up and purification, short reaction time, excellent yields of products are the main attractive features of the triazole synthesis. Surprisingly, when 4-methylphenylacetylene and benzyl halides with electron-withdrawing groups at para positions were employed, 1,2,3-triazoles with an alkynyl substituent at 5-position were produced in good yields. Furthermore, the catalyst could be reused for 5 consecutive runs without any significant decrease in reaction yield. Huang *et al.* investigated a facile method to access 1,4-disubstituted-1,2,3-triazoles by three-component reaction of alkyl halides, sodium azide with terminal alkynes using nano-copper particles. The nanoparticles were synthesized by reducing $CuSO_4.5H_2O$ with hydrazine hydrate in ethylene glycol. A wide range of products was obtained in moderate to excellent yields under mild conditions. Aliphatic alkynes failed to form the target product under the standard conditions. The nanocatalyst could be reused at least three times without considerable deactivation (**Scheme 2**) [45]. Mechanistically, alkyne reacts with catalyst to generate the alkynyl copper(I) (**A**). Then, this intermediate undergoes cycloaddition with organic azides formed in situ to provide the intermediate **B**. The Intermediate **B** is converted to 1,4-disubstituted 1,2,3-triazoles with the regeneration of the nanocatalyst.

In 2015, Nasrollahzadeh *et al.* employed copper NPs immobilized on carbon (C/Cu NPs) as an efficient catalyst for the preparation of 1,2,3-triazoles from organic halides, terminal alkynes, and sodium azide. Seven products were obtained in good to excellent yields using water as solvent. The use of iodobenzen failed to form the desired product. The catalyst remained highly efficient after five runs, although low catalyst loading was peformed (**Scheme 3**) [47]. The synthesis could be efficiently expanded to 15 mmol scale. Jafari *et al.* demonstrated the synthesis of Fe_3O_4@SiO_2–ABT/$Cu(OAc)_2$ and the employment of this

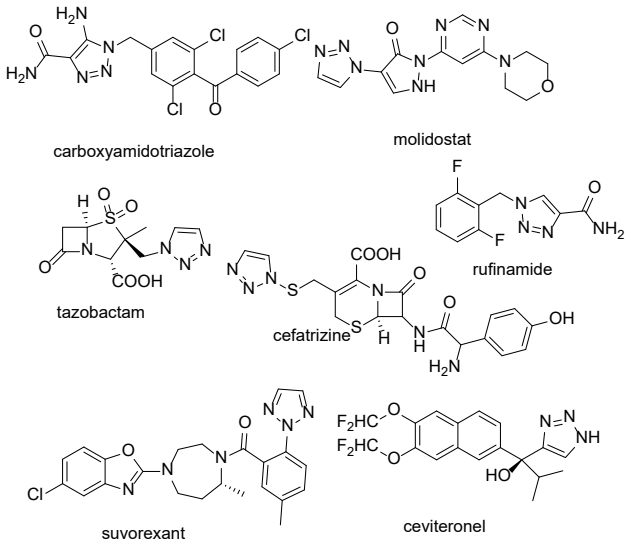
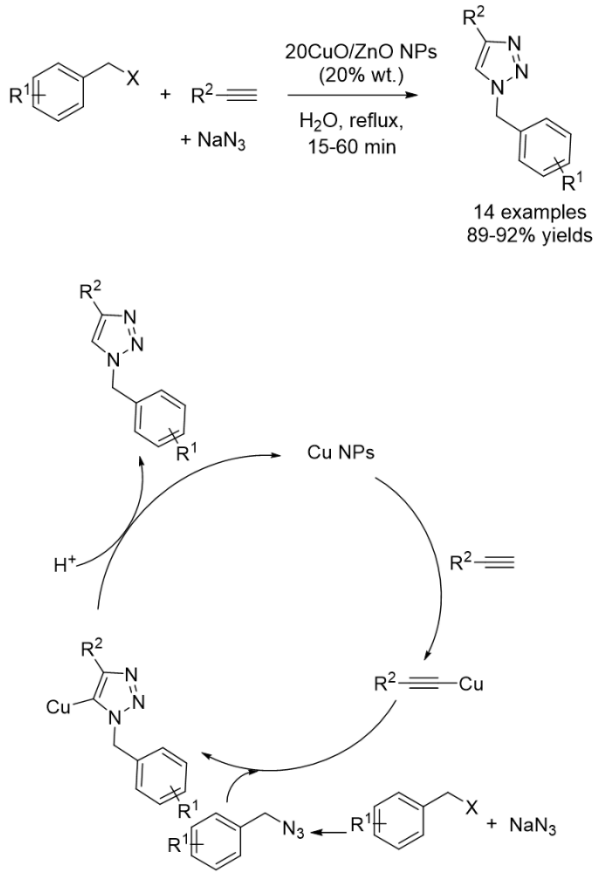


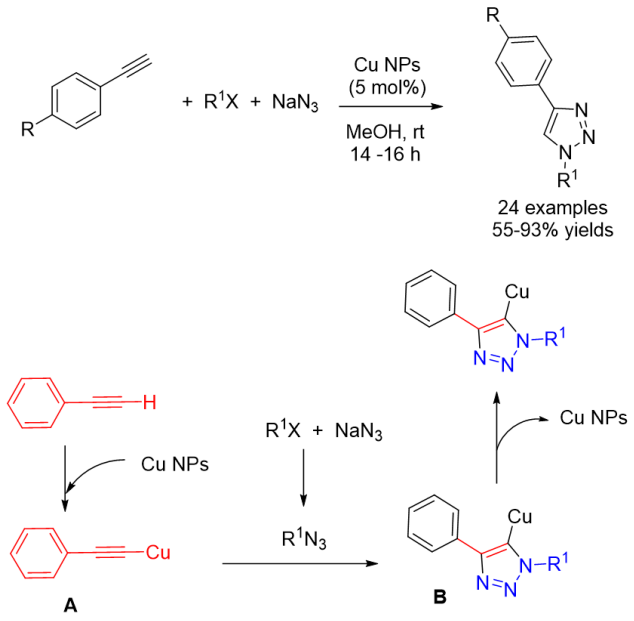
Figure 1. Several drugs containing 1,2,3-triazole moiety.



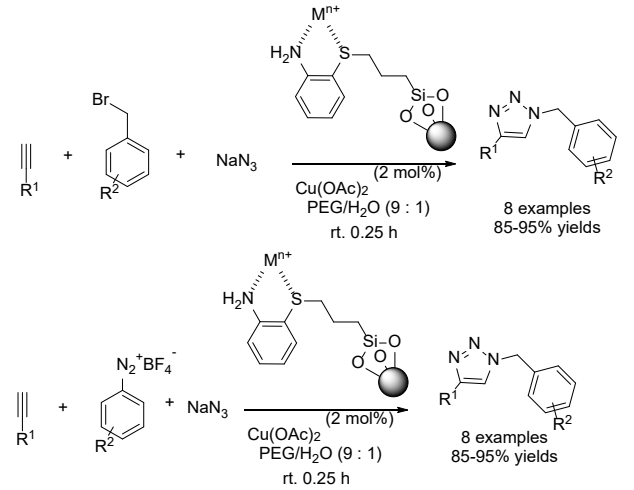
Scheme 1. 1,2,3-triazole synthesis described by Albadi *et al.* [44].

nanoparticle for the assembly of 1-aryl 1,2,3-triazoles from terminal alkynes, benzyl bromides and sodium azide under mild reaction conditions. Advantages of the synthesis include high efficiency, simple catalyst recover and efficient reuse, short reaction time, and good functional group tolerance (**Scheme 4**) [48]. Negligible amount of leaching of the metal and a long life span of the catalyst are the other advantages of the methods. The synthesis was also worked perfectly for the three-component reaction of diazonium salts, terminal acetylenes, and sodium azide.

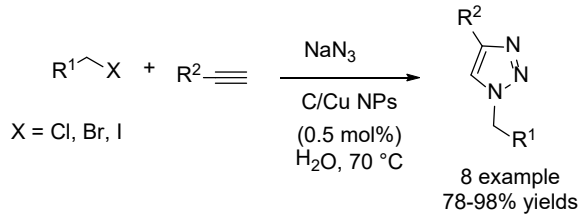
Kumar *et al*. developed a facile, efficient and environmentally-friendly approach for the synthesis of 1,4-diaryl-1,2,3-triazoles by reaction of boronic acids, sodium azide, and acetylenes using simply prepared copper sulphate immobilized on chitosan as a catalyst. The synthesis was performed in water at room temperature. A diverse series of products was delivered with high efficiency and the catalyst could be simply recycled by decanting and reused without significant loss of catalytic activity up to five cycles thank to its stability (confirmed by SEM analysis) (**Scheme 5**) [49]. Gangaprasad *et al*. demonstrated a versatile strategy to prepare 1,2,3-triazoles *via* oxidative cycloaddition between alkenes and organic azides using the commercially available heterogeneous copper oxide nanoparticles. A broad range of products was provided in high yields. Alkyl azides gave products in lower yield than benzyl and aromatic azides. The catalyst was efficiently recovered from the reaction mixture by a sequence of centrifugation, washing with ethyl acetate, and drying. Then the catalyst could be reused for four cycles without any significant decrease in reaction yields (**Scheme 6**) [50].



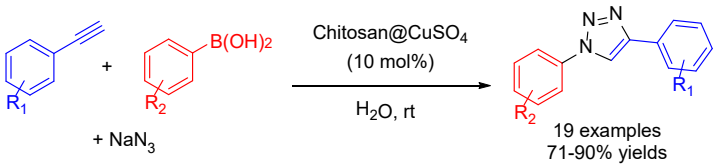
Scheme 2. 1,2,3- triazole synthesis via three-component reaction.



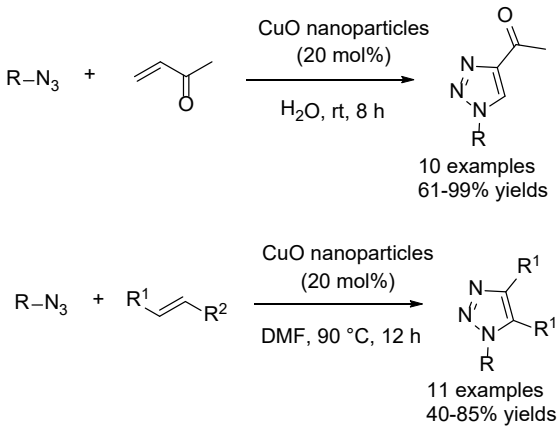
Scheme 4. Versatile synthesis of triazoles under Fe_3O_4@SiO_2–ABT/$Cu(OAc)_2$ catalyst.



Scheme 3. 1,2,3- triazole synthesis using copper NPs immobilized on carbon as a catalyst.



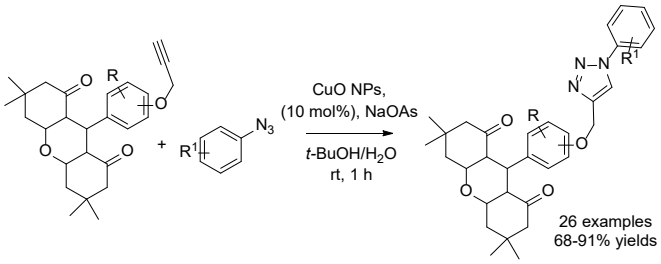
Scheme 5. Mild and efficient preparation of 1,4-diaryl-1,2,3-triazoles.



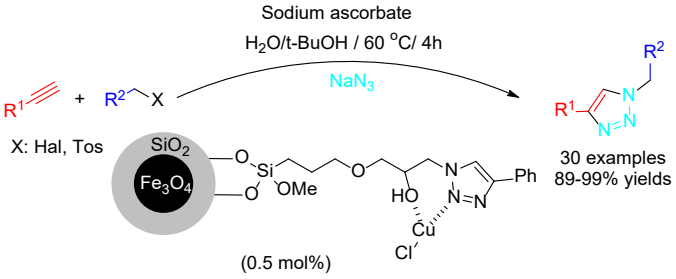
Scheme 6. Synthesis of 1,2,3-triazoles via oxidative cycloaddition.

Iniyavan *et al.* discovered a novel methodology for the preparation of xanthene substituted 1,2,3-triazoles through 1,3-dipolar cycloaddition of aromatic azides with acetylenic xanthenes using copper oxide nanoparticles as a catalyst. The catalyst was prepared from $Cu(OAc)_2$ by simple co-precipitation method and characterized by SEM, EDS, TEM, and XDR analyses. The reaction worked well with both electron-withdrawing and electron-donating groups at aryl azides. Mild reaction conditions, high efficiency, short reaction time, reusability of the catalyst, and broad substrate scope are the salient features of the synthesis (**Scheme 7**) [51]. Mnasri *et al.* described the synthesis of Cu-doped rod-shaped mesoporous silica nanoparticles (Cu-RMSN) by the co-condensation of (3-aminopropyl)triethoxysilane and tetraethyl orthosilicate as a silicon source and the characterization of the material by SEM and TEM studies. The nanomaterial then was employed as a catalyst to prepare 1,4-dibustituted-1,2,3-triazole derivatives by a microwave-assisted three-component 1,3-dipolar cycloaddition reaction of terminal alkynes, bromides, and sodium azide. Seven products were isolated in good to excellent yields with low catalyst loading (**Scheme 8**) [52]. In addition, the nanocatalyst remained highly active after ten runs providing products in good yield (88%) thank to its high stability and negligible metal leaching. Moghaddam *et al.* illustrated the synthesis and application of [MNPs@FGly][Cl] as an efficient catalyst for the synthesis of 1,2,3-triazoles from terminal alkynes, NaN_3, and halides or tosylates. In all cases, reactions for alkyl chlorides were faster than the corresponding bromides/tosylates. The reaction was also affected by steric hindrance on the alkyl halide component resulting lower reaction rate. Both linear and aromatic were compatible for the synthesis. A huge library of products was achieved in excellent yields. The AAS experiments showed that there was no remarkable metal leaching even after 10 cycles of the reactions and this explained the excellent reusability of the catalyst (**Scheme 9**) [53]. All the reactions were achieved with high regioselectivity, with the exclusive formation of 1,4-regioisomers.

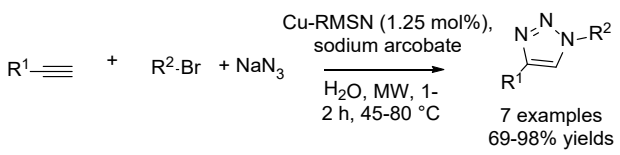
In 2016, Safa *et al.* disclosed a protocol for the construction of 1,4-disusbtitude 1,2,3-triazoles by 1,3-dipolar cycloaddition of benzyl halides, terminal alkynes, and sodium azide under ultrasound conditions using $LaCu_{0.7}Mn_{0.3}O_3$ as a nanocatalyst. The nanocatalyst was prepared by the sol-gel method and its structure was characterized by XRD and SEM. The catalyst could be recovered by simple filtration and reused at least five times without any significant loss of its catalytic activity. A diverse array of products was achieved in excellent yields under mild conditions in aqueous media in a short reaction time (5 minutes) (**Scheme 10**) [54]. Moghaddam *et al.* reported the synthesis of a novel copper nanomaterial [MNPs@APTA][Cl_2] and the characterization of the nanomaterial by SEM, TEM, AAS, FT-IR, TGA, and EDS analyses. The material was then applied for the assembly of triazole from phenylacetylenes, benzyl bromides, and sodium azide. A huge library of products was achieved with superior efficiency using water as solvent (**Scheme 11**). AAS analysis indicated that there was no significant metal leaching of the catalyst the catalyst after 10 cycles. This could explain the excellent reusability of the catalyst for ten times with high efficiency of the reaction [55]. In all cases, excellent regioselectivity with exclusive formation of 1,4-



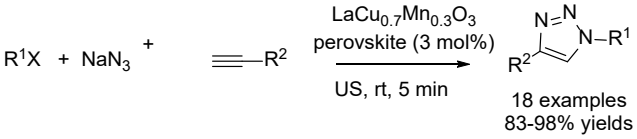
Scheme 7. Preparation of xanthene substituted 1,2,3-triazoles.



Scheme 9. 1,2,3-triazole synthesis using [MNPs@FGly][Cl] catalyst.



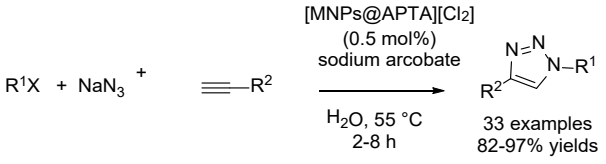
Scheme 8. Cu-RMSN-catalyzed synthesis of 1,2,3-triazoles.



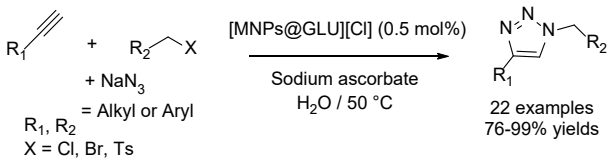
Scheme 10. $LaCu_{0.7}Mn_{0.3}O_3$ as an efficient catalyst for the construction of 1,4-disusbtitude 1,2,3-triazoles.

regioisomers was obtained. Steric hindrance on the halide counterpart could lower reaction rates. Aliphatic alkynes were more inactive than aromatic alkynes, and the reactions need longer times as compared with aromatic alkynes.

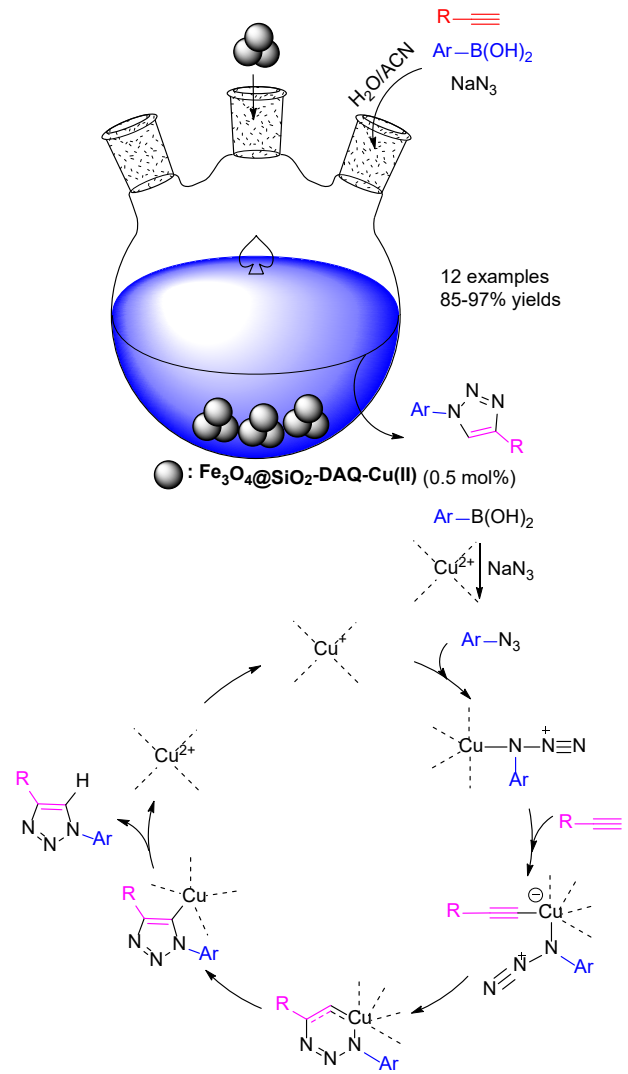
Moghaddam *et al.* presented the synthesis of [MNPs@GLU][Cl] nanomaterial by the immobilization of Cu^{2+} onto glucose on Fe_3O_4. The substance was characterized by FT-IR, TGA, CHN, SEM, EDX, and AAS methods. Subsequently, the nanomaterial was used for the 1,3-dipolar cycloaddition of alkynes, alkyl halides, and sodium azide to access 1,2,3-triazoles. Mild reaction conditions, the use of water as green solvent, high efficiency, catalyst recyclability, low catalyst loading, and broad substrate scope are the merits of the triazole synthesis (**Scheme 12**) [56]. AAS analysis of the reused catalyst after seven cycles indicated there was no considerable metal leaching of the catalyst. The reactions of alkyl chlorides needed longer time than those of alkyl bromides and tosylates did. Increasing the steric hindrance of alkyl halides led to the decrease in rate of the reaction. Zahmatkesh *et al.* employed 1,4-dihydroxyanthraquinonecopper(II) supported on superparamagnetic Fe_3O_4@SiO_2 (Fe_3O_4@SiO_2DAQCu(II)) as a catalyst for the preparation of 1,2,3-triazoles from NaN_3, terminal alkynes, and aryl boronic acids. The catalyst could be separated from solution in a short period under an external magnetic field and reused for six runs without any significant loss of its catalytic activity (**Scheme 13**) [57]. ICP experiment revealed that there was only 2.67 wt.% (0.0061 mmol/g) of the total amount of the original copper species leaching into solution during the course of the reaction after six cycles. A plausible reaction mechanism explaining the role of the catalyst was also proposed. Nasrollahzadeh *et al.* prepared copper nanoparticles (Cu NPs) by reduction of $CuCl_2{\cdot}2H_2O$ solution using flavonoid and other phenolics as a main ingredients in aqueous extract of leaves of *Otostegia persica*. After characterizing the structure by TEM, FT-IR and XRD, the nanoparticles were employed as a catalyst to synthesized 1,4-disubstituted 1,2,3-triazoles from halides, sodium azide and terminal alkynes. Various triazole derivatives were obtained in good yields under mild conditions. The catalyst could be simply recovered by centrifugation and reused at least five cycles without significant loss of its activity (**Scheme 14**) [58]. Benzyl bromide showed better reactivity than corresponding benzyl chloride under optimal conditions.



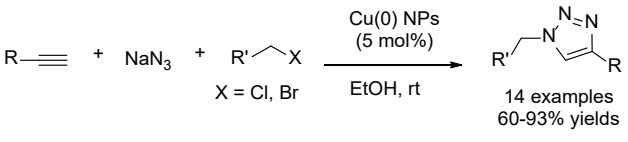
Scheme 11. 1,2,3-triazole synthesis using [MNPs@APTA][Cl_2] catalyst.



Scheme 12. [MNPs@GLU][Cl]-catalyzed three-component reaction for the construction of 1,2,3-triazoles.

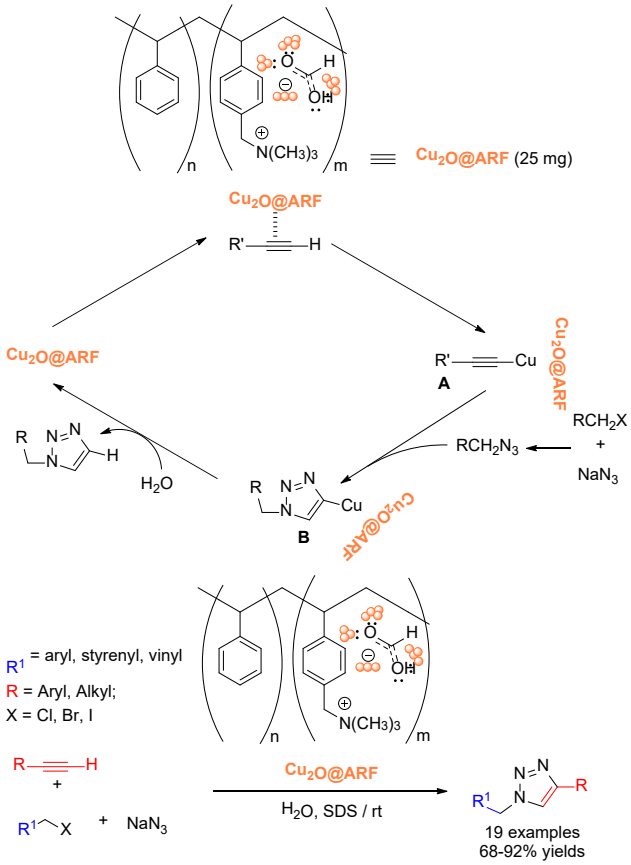


Scheme 13. Preparation of 1,2,3-triazoles from NaN_3, terminal alkynes, and boronic acids.



Scheme 14. Synthesis of 1,2,3-triazoles using copper nanoparticles under mild conditions.

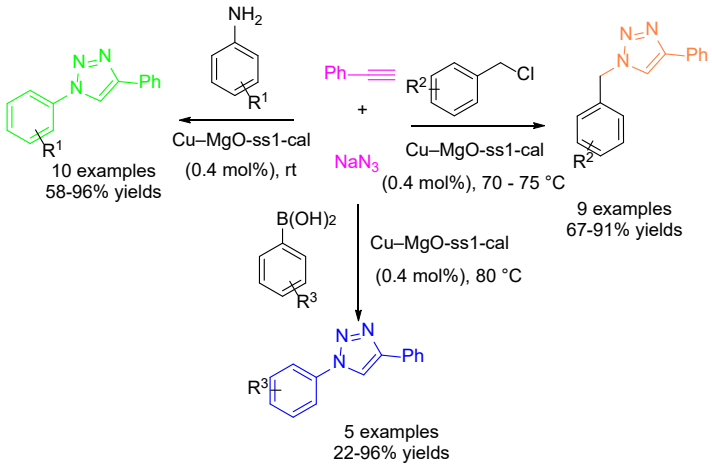
In 2017, Ghosh *et al*. introduced the synthesis of immobilization of stabilized Cu_2O nanoparticles and the characterization of the material by FT–IR, XRD, XPS, HR–TEM, and ICP–AES analyses. The Cu_2O nanoparticles then were utilized as catalyst for the construction of 1,2,3-triazoles through a three component "click" reaction of alkyl halide, terminal alkyne, and sodium azide. The triazole synthesis was performed in water at room temperature. The reaction of aliphatic alkyne needed longer time than aromatic alkyne. A wide range of products was furnished in good to excellent yields. The XRD analysis revealed that structure of recovered catalysts was found to be almost similar to the fresh one. This exxplained why the catalytic activity remains almost unchanged in consecutive fourth runs (**Scheme 15**) [59]. In comparison with previous on-water triazole synthesis using other Cu(I)-based catalytic systems, the TOF of this method was found to be significantly high. Mechanistically, terminal alkyne reacts copper catalyst forms alkynyl copper intermediate, which undergoes cycloaddition with alkyl azide generated *in situ* from alkyl halide and sodium azide. Finally, protonation of the C-Cu bond furnishes the desired triazole product. Roy *et al*. illustrated the synthesis of various morphologies



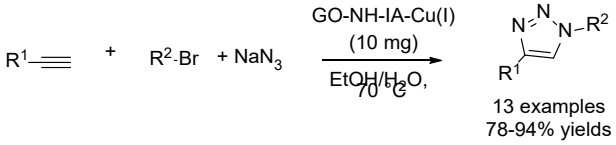
Scheme 15. Synthesis of 1,2,3-triazoles via click reaction of alkyl halide, terminal alkyne, and sodium azide.

of self-assembled Cu-MgO nanocrystal line materials by single step hydrothermal route as well as wet impregnation method from pre-synthesized MgO. The Cu-MgO nanomaterial was a versatile catalyst for the synthesis of triazole by three component reaction of phenyl acetylenes, sodium azide with benzyl halides or anilines or phenyl boronic acids. In most cases, products were afforded in high yields. After the fifth run, a little disorderedness in assembly of the catalysts was observed in SEM image of the reused catalyst and this explained the excellent reusability of the catalyst. In addition, each reaction had high TON value.

Naeimi *et al*. reported the synthesis of functionalized graphene oxide with copper(I) (GONH-IA-Cu(I)) and its characterization by FT-IR, XRD, FE-SEM, EDX methods. The nanomaterial was examined for the one-pot preparation of 1,2,3-triazoles through 1,3-dipolar cycloaddition reaction between terminal alkynes, halides and NaN_3. Various products were isolated in high yields and the catalyst still showed excellent catalytic activity even for the fifth cycle (**Scheme 17**) [61]. The structure of the reused catalyst did not change much according to the SEM analysis. Naeimi and Ansarian described the preparation of a tetradentate nitrogen ligand on the surface of functionalized GO@PTA-Cu and the characterization of this material by FT-IR, SEM, TGA, ICP analyses. The nanomaterial was

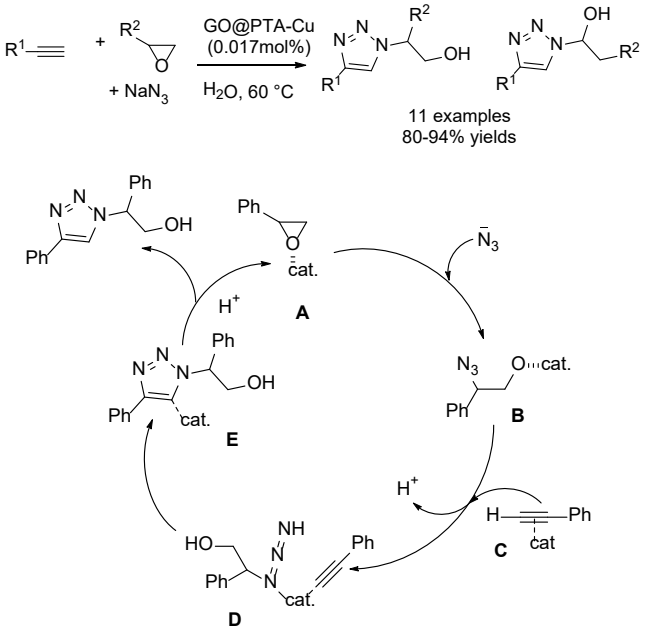


Scheme 16. Versatile synthesis of 1,2,3-triazoles using Cu-MgO nanocatalyst.



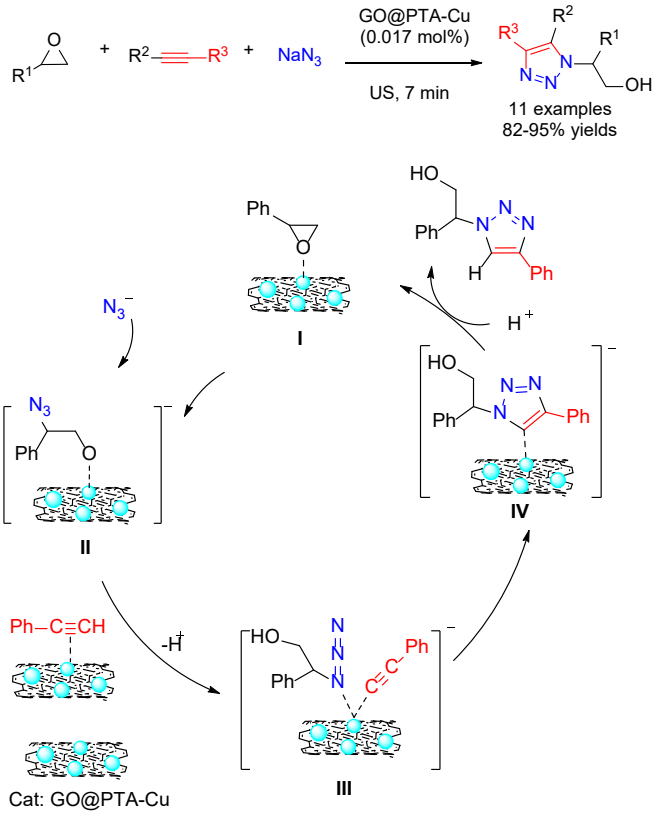
Scheme 17. GONH-IA-Cu(I)-catalyzed synthesis of 1,2,3-triazoles.

then employed as a catalyst to access ß-hydroxy-1,2,3-triazoles by three-component reaction of terminal alkynes, oxiranes, and sodium azides. Products were provided in good to excellent yields and in good regioselective manner. Reactions of aliphatic epoxides gave lower yields than those of aryl-substituted epoxides. A reaction mechanism was also proposed to explain the role of the nanocatalyst (**Scheme 18**) [62]. SEM image of the reused catalyst did not show any considerable change in the morphology indicating the excellent reusability of the catalytic activity with recycling. These authors also performed GO@PTA-Cu-catalyzed triazole synthesis under ultrasound conditions. Desired products were achieved in good to excellent yields. The catalyst was simply recovered by filtration, washing, and drying then was efficiently reused for five runs (**Scheme 19**) [63]. Short reaction time, the use of water as a green solvet, and high TOF and TON values are other merits of the synthesis. A reaction mechanism was also presented, in which GO@PTA-Cu has a two-fold catalytic impress as a bifunctional catalyst. Initially, epoxide reacts with sodium azide in the presence of GO@PTA-Cu catalyst generates organoazide **II** (detected by TLC). Phenylacetylene interreacts with GO@PTA-Cu provides the copper **I** acetylide, which reacts with the organoazide **II** to yield the intermediate **III**. Cyclization of the intermediate **III** resulted in the intermediate **IV**, which undergoes protonation to furnish the ß-hydroxy-1,2,3-triazole product. For the reactions of aliphatic epoxides, a decrease in yield and increase in reaction time were observed in comparison with aromatic epoxides.

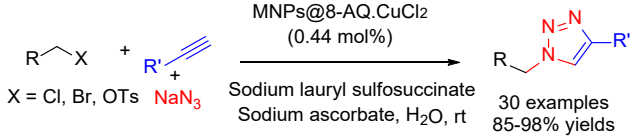


Scheme 18. 1,2,3-triazole synthesis from terminal alkynes, sodium azide, and epoxides.

Mogaddam *et al.* synthesized copper(II) chloride immobilized on 8-aminoquinoline functionalized magnetic Fe_3O_4@SiO_2 (MNPs@8-AQ.$CuCl_2$) and characterized the structure by FT-IR, XRD, SEM, TEM, EDX, TGA, and AAS. The material showed excellent catalytic activity for the synthesis of 1,4-disubstituted-1H-1,2,3-triazole *via* one-pot three-component of benzyl halides, terminal alkynes, and sodium azide. Salient features of the synthesis include broad substrate scope (30 products), mild reaction conditions, catalyst recyclability, the use of water as solvent, scalability, low catalyst loading and excellent efficiency (**Scheme 20**) [64]. The AAS analysis revealed that there was no significant metal leaching of the catalyst after five cycles. Reaction of alkyl tosylate and alkyl bromide gave slight better yields in shorter time than the corresponding chloride. Chetia *et al.* fabricated copper nanoparticles supported on nanocellulose (CuNPs/NC) and applied this material as catalyst to access 1,2,3-triazoles. 14 triazole derivatives



Scheme 19. GO@PTA-Cu-catalyzed triazole synthesis under ultrasound conditions.

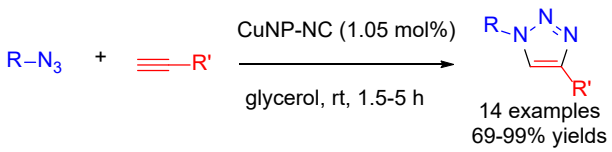


Scheme 20. Triazole synthesis using MNPs@8-AQ.$CuCl_2$ catalyst.

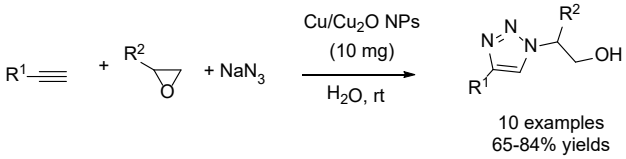
were afforded in high efficiency under mild conditions. The nanocatalyst could be reused for five times with negligible loss of catalytic activity (**Scheme 21**) [65]. ICP analysis confirmed that there was no significant leaching of copper species from the reused catalyst during the recycling test.

In 2018, Esmaeili-Shahri *et al.* synthesized Cu/Cu_2O nanomaterial by a simple ball mill reduction of CuO with $NaBH_4$ using a ball-to-powder weight ratio of 50:1 under air atmosphere at room temperature. The material was characterized by SEM, EDS, FT-IR and X-ray diffraction. The nanomaterial exhibited significant catalytic activity for the synthesis of 1,2,3-triazoles from sodium azide, epoxides and terminal alkynes. The triazole synthesis was performed in water at room temperature. The nanocatalyst still remained highly active for the third cycle (**Scheme 22**) [66]. The reactions of aliphatic epoxides delivered a single regioisomer by exclusive attack at more hindered carbon atom. The regioselectivity decreased in the case of aromatic epoxides but more substituted regioisomers were obtained. Nayal *et al.* discovered novel approach for the synthesis of 1,4-disubstituted-1,2,3-triazoles using heterogeneous copper(I) iodide nanoparticles (CuI NPs) as a catalyst. Ten products were afforded in excellent yields using water as a solvent (**Scheme 23**) [67].

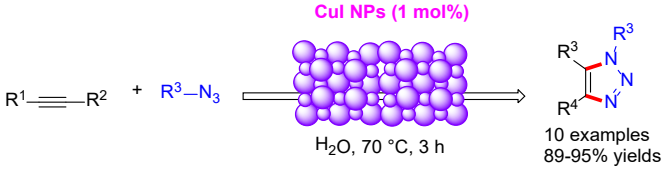
Dolatkhah *et al.* prepared Cu–IG@Fe_3O_4 nanomaterial by the copper immobilization onto magnetic isinglass. The nanomaterial was characterized by FT-IR, TGA, AAS, SEM, EDS, VSM, XPS, and XRD measurements. This material was applied as a nano bio-catalyst for the synthesis of 1,2,3-triazoles *via* 1,3-dipolar cycloaddition reaction of benzyl halides, phenyl acetylenes, and sodium azide. A diverse series of products was generated in good to excellent yields using water as a solvent (**Scheme 24**) [68]. FT-IR, XRD and FESEM analyses revealed that structure of the catalyst after these five runs was similar to the fresh one indicating the excellent reusability of the catalyst. Gupta *et al.* demonstrated the preparation of dual synergistic PdCu@rGO nanocatalyst and characterization of the material by XRD, Raman, FT-IR and XPS methods. The catalyst was suitable for several transformations including Suzuki, Heck reactions and click reactions. For the triazole synthesis, ten products were provided with excellent efficiency (**Scheme 25**) [69]. TEM, SEM, EDX and FT-IR analyses confirmed slight changes of reused catalyst after seven runs in comparison to the fresh one indicating the excellent reuse of the catalyst. Raj *et al.* utilized commercially available CuO nanoparticles as an efficient catalyst for the assembly of 1,2,3-triazoles through azide-olefin cycloaddition of bromoalkenes and organic azides. A wide range of products was delivered in good to excellent yields with good catalyst reuse (four times) (**Scheme 26**) [70].



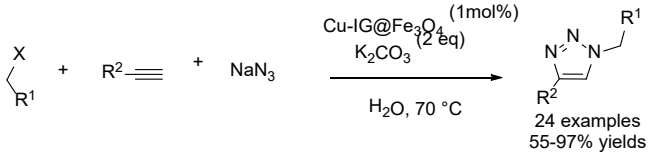
Scheme 21. Click synthesis of 1,2,3-triazoles demonstrated by Chetia *et al.* [65].



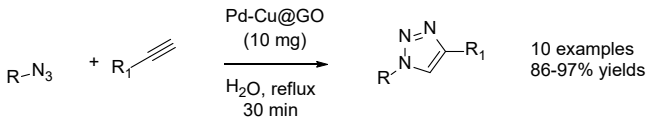
Scheme 22. Cu/Cu_2O nanomaterial as catalyst for the synthesis of 1,2,3-triazoles.



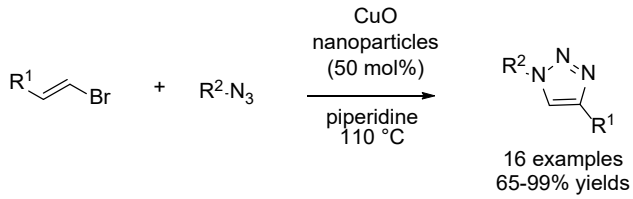
Scheme 23. CuI NPs-catalyzed click reaction between alkynes and azides.



Scheme 24. Cu–IG@Fe_3O_4 nanomaterial as a catalyst for the synthesis of 1,2,3-triazoles.

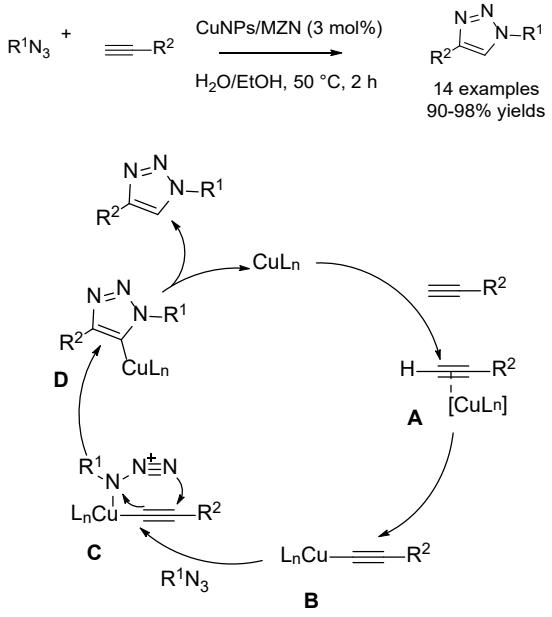


Scheme 25. Efficient synthesis of 1,2,3-triazoles under PdCu@rGO nanocatalyst.

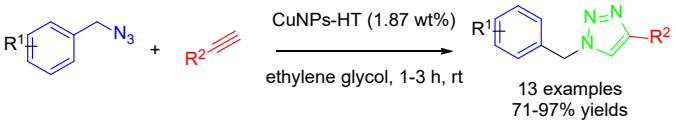


Scheme 26. Synthesis of 1,2,3-triazoles from bromoalkenes and organic azides.

In 2019, Siuki *et al*. illustrated the assembly of copper nanoparticle-loaded natural zeolite (CuNPs/ MZN) and its characterization by FT-IR, CHN, TGA, ICP, XRD, SEM, and TEM methods. The nanomaterial then was employed as a catalyst for the construction of 1,2,3-triazoles from aryl azide and terminal alkynes under ultrasonic conditions. Various triazoles were furnished in excellent yields. The TEM photograph of the recovered nanocatalyst revealed that the structure of the catalyst remained almost the same after five reuses and this explained the efficient reusability of the catalyst (**Scheme 27**) [71]. A plausible reaction mechanism was also proposed. Initially, a terminal alkyne is coordinated to CuNPs supported on zeolite to form copper–alkylidine complex **B** is formed on the surface of the nanocomposite thank to high alkynophilicity of Cu metal for terminal alkynes. Subsequently, azide attacks the complex followed **B** by intramolecular cyclization to furnish the desired product. Chetia *et al*. developed hydrotalcite supported copper nano particles as a catalyst for the azide-alkyne cycloaddition reaction to access 1,4-disubstituted-1,2,3-triazoles. Attractive features of the synthesis include low catalyst loading, mild reaction conditions, the use of ethylene glycol as solvent, high efficiency, and recyclability of the catalyst (four cycles due to minute copper leaching) (**Scheme 28**) [72].

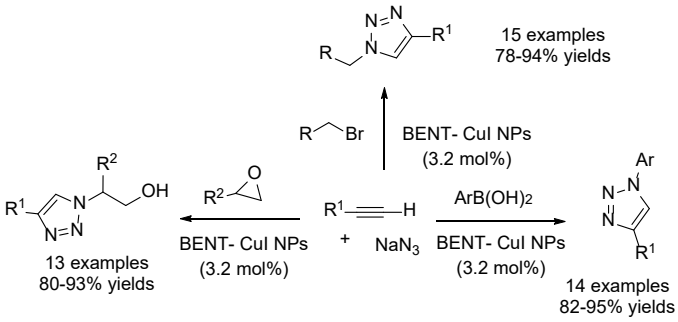


Scheme 27. CuNPs/ MZN-catalyzed efficient synthesis of 1,2,3-triazoles.

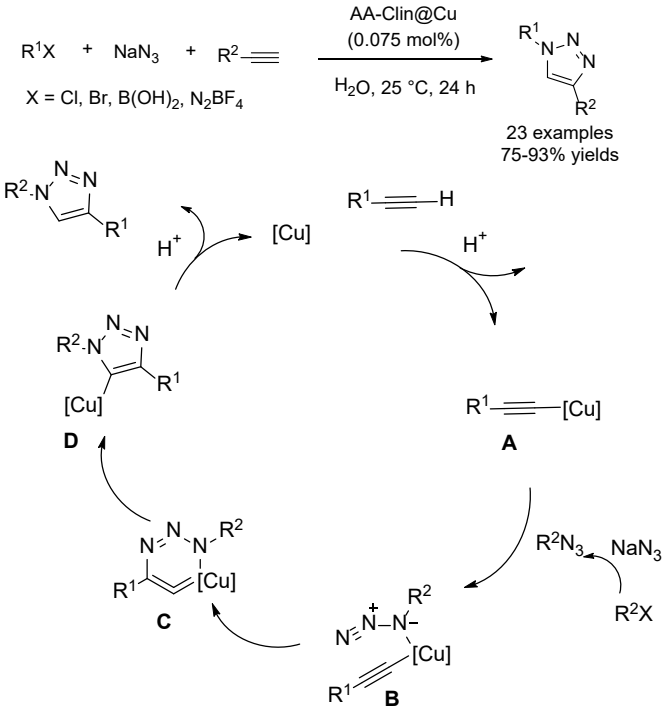


Scheme 28. Mild synthesis of 1,2,3-triazoles using CuNPs-HT catalyst.

Chavan *et al*. illustrated the preparation Bentonite clay supported CuI nanoparticles (BENT-CuINPs) and characterization of the material by ICP-AES, EDS, SEM, XRD, TEM as well as BET techniques. The nanomaterial then was used as a versatile catalyst for the synthesis of 1,2,3–triazoles through three different routes. A diverse library of products was achieved in good to excellent yields. The SEM and TEM images of reused catalyst did not show any change in the morphology of the catalyst (after five cycles) and this explained the excellent catalytic activity of the recycled nanocatalyst (**Scheme 29**). For the reaction of epoxides, the reaction showed excellent regioselectivity with the exclusive formation of one product [73] Gholinejad *et al*. prepared acid activated clinochlore supported copper nanoparticles (AA-Clin@Cu) by immobilization of copper sulfate on clinochlore followed by *in situ* chemical reduction. The resulting nanoparticle was characterized by SEM, TEM, TGA, FTIR, XRD, XPS, and EDX analyses. Then it was employed as an efficient



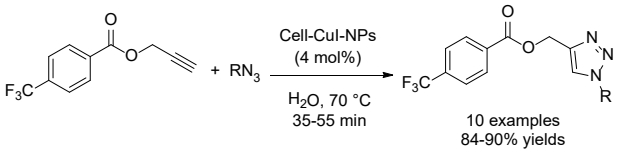
Scheme 29. Versatile and efficient synthesis of 1,2,3–triazoles using BENT-CuINPs catalyst.



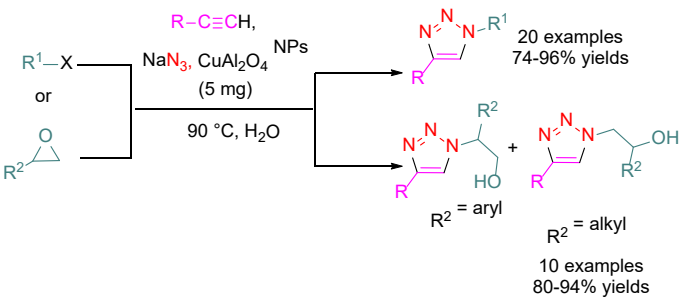
Scheme 30. AA-Clin@Cu as a catalyst for 1,2,3–triazole synthesis.

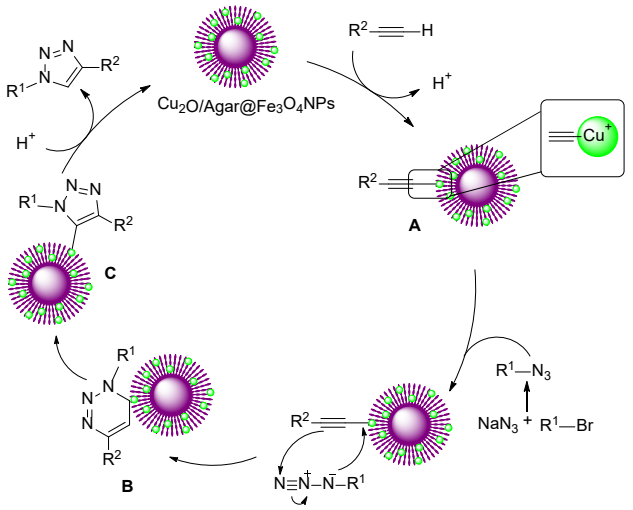
catalyst for the construction of 1,2,3-triazoles from halide or boronic acids, sodium azide and alkynes under mild conditions. A diverse array of products was afforded in good to excellent yields under mild conditions (**Scheme 30**). XPS study of the structure of the reused catalyst after the four runs showed very similar pattern toward the fresh catalyst indicating the efficient reusability of the catalyst. In addition, the reaction could be efficiently applied in gram scale [74]. Mechanistically, terminal alkyne coordinates with Cu species to generate [Cu] acetylidine complex **A**, which reacts with organic azides to give [Cu]–triazolide complex **D**. Finally, protonation and demetallation of [Cu]–triazolide furnishes desired 1,2,3-triazole.

Maleki *et al.* prepared Cu_2O/Agar@Fe_3O_4 by supporting copper(I) oxide nanoparticles on magnetic agar and characterized the material by SEM, TEM, EDS, FT–IR, TGA, XRD, VSM, and ICP techniques. The nanomaterial then was successfully applied as a catalyst for the synthesis of 1,4–disubstituted 1,2,3–triazoles by three-component reaction of alkyl halides, sodium azide, and alkynes. Ten products were finished in good to excellent yields. The ICP analysis revealed that only 6% of Cu was leached into the reaction media after five consecutive runs. This results proved the excellent reuse of the catalyst (**Scheme 31**) [75]. Mechanistically, the catalyst reacts with a terminal alkyne to form copper acetylide **A**, which undergoes 1,3-dipolar cyclization with an organic azide (generated *in situ* from organic halide and sodium azide) to generate the intermediate **C** *via* the six-membered intermediate **B**. Finally, protonation of **C** provides 1,2,3-triazole products and regenerates of Cu_2O/Agar@Fe_3O_4 nanocatalyst. Deswal *et al.* investigated a protocol to access 1,2,3-triazoles starting from 4-trifluoromethylbenzoic acid prop-2-ynyl ester 2 with aryl and benzyl azides using cellulose supported-copper(I)-nanoparticles as a catalyst. The synthesis was performed in water under mild conditions and gave products in good to excellent yields (**Scheme 32**) [76]. Most of the synthesized triazoles displayed strong antimicrobial activity against tested strains. Khalili *et al.* accomplished the synthesis of 1,4-disubstituted 1,2,3-triazoles by reaction of terminal alkynes, sodium azide, and halides or oxiranes using copper aluminate nanoparticles as a catalyst. A wide range of products was furnished in high yields. Low catalyst loading, the use of water as a green solvent, and the recyclability of the catalyst (over six consecutive runs) are the other merits of the synthesis (**Scheme 33**). Inductively coupled plasma analysis indicated that only negligible amounts of copper leached from the catalyst surface during each cycle. This confirmed the remarkable reusability of the nanocatalyst [77].

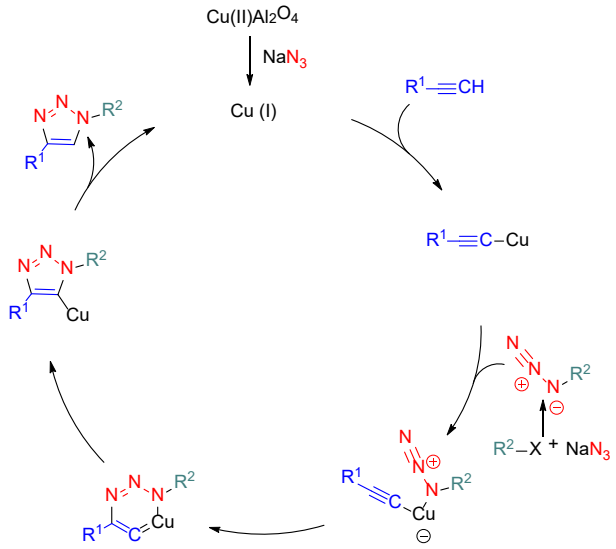


Scheme 32. Triazole synthesis using Cell-CuI-NPs catalyst.





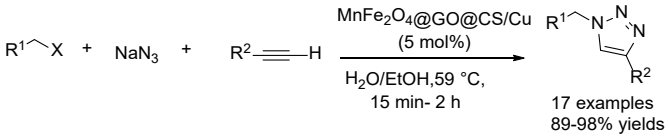
Scheme 31. Cu_2O/Agar@Fe_3O_4-catalyzed synthesis of 1,2,3-triazoles.



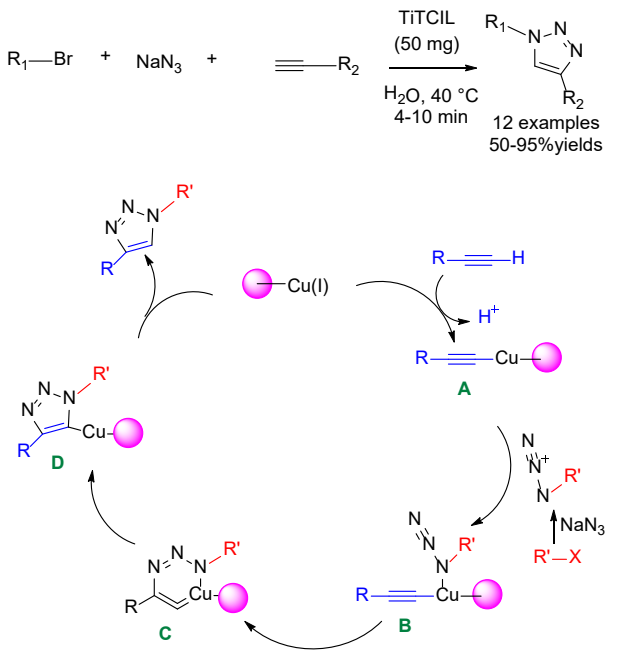
Scheme 33. Versatile synthesis of 1,2,3-triazoles using $CuAl_2O_4$ NPs catalyst.

For the reaction of epoxides, excellent regioselectivity was observed. A tentative reaction mechanism was also illustrated. Mahdavinasa *et al*. examined a strategy towards 1,4-disubstituted 1,2,3-triazoles through reaction of primary halides, NaN_3 and terminal alkynes using $MnFe_2O_4$@GO@CS/Cu nanocatalyst. A wide range of products was delivered in excellent yields. The nanocatalyst still exhibited efficient activity even after nine runs due to insignificant metal leaching (**Scheme 34**) [78].

In 2020, Shamar *et al*. reported the preparation of 1-butyl-4methylpyridinium tetrafluoroborate coated $CuFe_2O_4$ over L -tyrosine functionalized titania nanospheres (IL@$CuFe_2O_4$-L-Tyr-TiO_2/Titin) and its characterization by FTIR, HR-TEM, FE-SEM, EDX, VSM, P-XRD, XPS, photoluminescence spectroscopy and Raman spectroscopy analyses. The material was then utilized as a catalyst for the synthesis of 1,4-disubstituted-1,2,3-triazoles *via* click reaction under mild conditions. For aliphatic bromides, the click reaction was viable but the yields of products were low. High efficiency, the use of water as solvent, short reaction times, and recyclability of catalyst are the advantages of the synthesis. The XPS experiments of recycled catalyst revealed



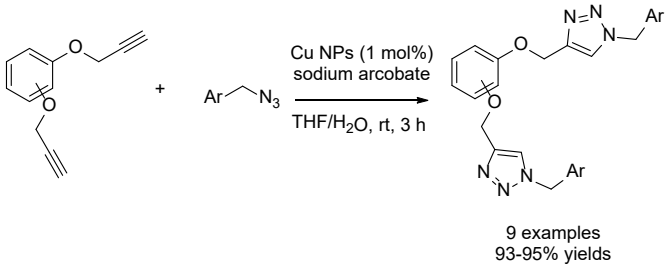
Scheme 34. Triazole synthesis accomplished by Mahdavinasa *et al.* [78].



Scheme 35. TiTCIL-catalyzed synthesis of 1,4-disubstituted-1,2,3-triazoles.

that there was no change in the oxidation states of carbon, nitrogen, oxygen, copper and iron in the recycled catalyst. Mechanistically, terminal alkyne reacts with catalyst to give the acetylide complex A, which binds to azide *via* nitrogen atom adjacent to carbon forming л complex intermediate **B**. Subsequently, intramolecular attack of distant nitrogen of the complex **B** to C-2 carbon of acetylide produces the six-membered Cu(III) metallacycle **C**, which undergoes ring contraction to form triazolyl Cu(I) derivative **D**. Finally, protonation of **D** furnishes the desired triazole and the catalyst was regenerated (**Scheme 35**) [79]. Abiraman *et al*. synthesized sub 1 nm sized poly(acrylic acid)-capped copper nanoparticles (PAACC NPs) using the high-intensity ultrasound sonication method in the presence of mild reducing agent *L*-ascorbic acid. The resulting material was characterized by DRS UV-visible, XPS, PL, FE-SEM, and HR-TEM techniques. Subsequently, the nanomaterial was applied as a catalyst for the assembly of 1,2,3-triazoles *via* click reaction between benzyl azides and diynes. Nine bis-1,2,3-triaoles linked to benzoxazole or benzothiazole were isolated in excellent yields under mild reaction conditions (**Scheme 36**) [80].

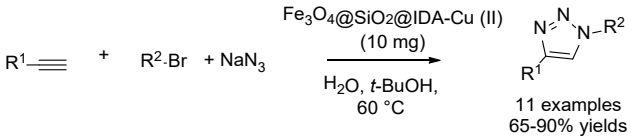
Godarzbod *et al*. synthesized silica-coated magnetic nanoparticles functionalized by iminodiacetic acid (Fe_3O_4@SiO_2@IDA) by sol–gel method and characterized the resulting material by ICP, XRD, EDX, SEM, TGA, and FT-IR analyses. The nanomaterial was employed as catalyst for the construction of 1,4-disubstituted-1,2,3-triazole derivatives *via* an azide–alkyne [3 + 2] cycloaddition reaction. The catalyst could be easily recovered using an external magnetic field and FT-IR spectroscopy analysis indicated that no significant change was observed for the structure of the reused catalyst compared to the fresh one. This could explain why the catalyst could be reused at least five times with negligible loss of its catalytic activity (**Scheme 37**) [81]. Rajabi *et al*. introduced the synthesis of melamine-supported CuO nanoparticles and the application of this nanomaterial as a catalyst for the assembly of 1,2,3-triazoles from halides, phenyl acetylene, and sodium azide. Various products were afforded in excellent yields in short



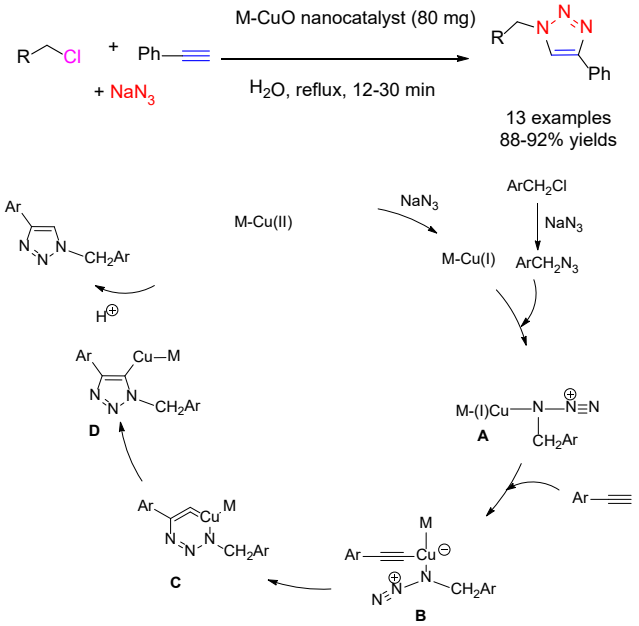
Scheme 36. Synthesis of bis-1,2,3-triazoles.

times. Low leaching amount of copper oxide catalyst 3.3 after fives runs) was examined by ICP-AES. This evidenced that the catalyst could be recycled and reused for six run with insignificant decrease in reaction yields (**Scheme 38**) [82]. A plausible reaction pathway was included. Firstly, Cu(II) is partially reduced to Cu(I) state by treatment with sodium azide and therefore is presented as Cu(II)/Cu(I) mixed valency dinuclear species. This species coordinates with benzyl azide to form the intermediate **A**, which react with phenyl acetylene to give the six-member ring **C** via the intermediate **B**. Ring contraction of **C** delivers the five-member **D**, which undergoes protonysis to furnish the corresponding product along with the regeneration of the catalyst. Nandi *et al*. prepared polyaniline stabilized copper-azide nanoparticle (CANP) and employed the nanomaterial as a catalyst as well as a nitrogen source for the construction of 5-alkynyl 1,4-disubstituted triazoles. A huge library of products was obtained in moderate to excellent yields under mild reaction conditions (**Scheme 39**) [83]. A reaction pathway explaining the role of the catalyst was also proposed. Initially, benzyl bromide reacts with CANP to form a copper-organic azide complex, which further rearranged to the intermediate **A**. Terminal alkyne is deprotonated by triethylamine and then reacts with A to provide a Cu-acetylidine complex which undergoes rearrangement to produce the metallacycle B. Subsequently, ring contraction of **B** leads to the formation of 1,4-disubstituted triazole **C**. In case of terminal alkyne containing electron donating group (EDG), further deprotonation affords the second Cu-acetylidine complex **D**, which finally converted to 5-alkenyl 1,4-disubstituted triazole. In contrast, for alkyne bearing an electron withdrawing group (EWG), a strong rate limiting oxidative addition facilitate the formation of 1,4-disubstituted triazole product.

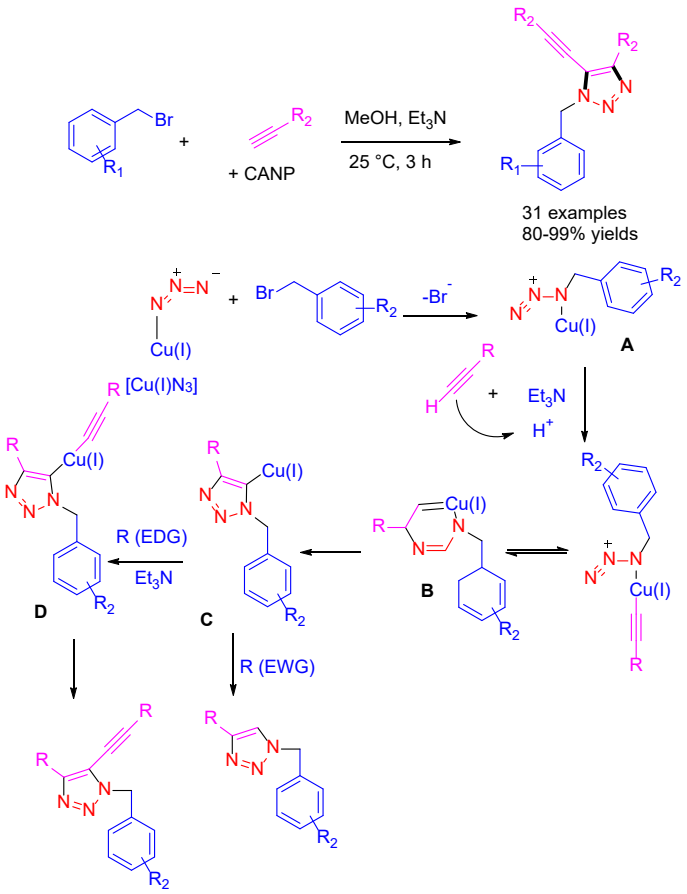
In 2021, Agarwal *et al*. prepared CuS nanoparticles and used this material as a catalyst to prepare 1,2,3-triazoles from aldehyde, nitro compounds, and sodium azide. Ten triazole derivatives were produced in good to excellent yields (**Scheme 40**) [84]. Darroudia *et al*. prepared a novel nanocatalyst (Cu/SiO_2-Pr-NH-Benz) and applied it as a catalyst for click reaction in an aqueous solution to access 1,2,3-triazole derivatives. This method led to the rapid formation of triazoles in good to excellent yields. Reactions of alkyl acetylides resulted in triazole in lower yields and required longer reaction time then aryl acetylides. The catalyst could be reused for six cycles with slight decrease in reaction



Scheme 37. Synthesis of 1,2,3-triazoles using Fe_3O_4@SiO_2@IDA catalysts.



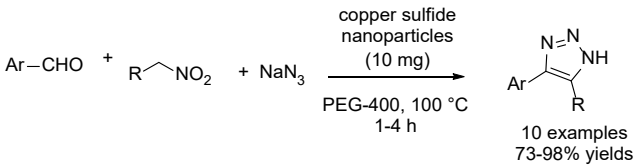
Scheme 38. Melamine-supported CuO nanoparticles as an efficient catalyst to access 1,2,3-triazoles.



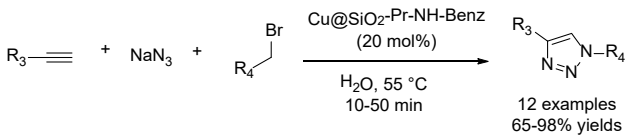
Scheme 39. Synthesis of 5 alkynyl 1,4 disubstituted triazoles.

yields (**Scheme 41**) [85]. FT-IR analysis indicated that no significant changes were observed for the reused catalyst in comparison with the fresh one.

Khoshnoud *et al.* demonstrated the preparation of a novel catalyst Al_2O_3@PCH-Cu(II) and employed the nanocatalyst for the synthesis of 1,4-disubstituted 1,2,3-triazoles. Nine triazole derivatives were furnished in excellent yields. Low decrease in reaction yield after five runs was observed for the reused catalyst (**Scheme 42**) [86]. Arefi *et al.* developed a magnetic metal organic framework with core–shell structure based on HKUST-1 MOF and characterized the material by FT-IR, TEM, SEM, TEM mapping, SEM mapping, EDX, PXRD, TGA, ICP and VSM analyses. The use of this nanomaterial as catalyst for triazole synthesis by three-component reaction of halides, sodium azide, and phenyl acetylenes resulted in triazole derivatives in good to excellent yields. Benzyl halides were more reactive than alkyl halides and phenylacetylene derivatives with no substituent or with electron-withdrawing group gave better yield compared to electron-donating groups. FT-IR, SEM and ICP analyses confirmed that no leaching of Cu or MOF shell structures occurred during the 1,3-dipolar cycloaddition reaction after five cycles. This could explain the excellent reusability of the catalyst to the click reaction **(Scheme 43)** [87]. A tentative reaction mechanism was presented. Initially, phenyl acetylene can coordinate with catalyst at Cu centers to form the Cu–alkylidine intermediate **A**, which react with organic azide (in situ generated from organic halide and sodium azide) to provide the intermediate **B**. Finally, intermediate **B** was converted to the desired product by a sequence of intramolecular cyclization and protonation.

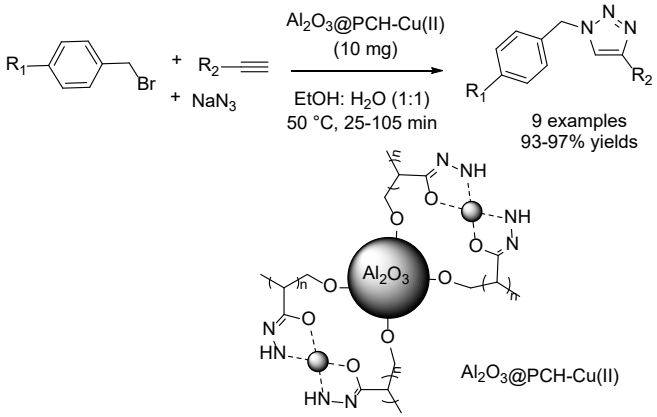


Scheme 40. CuS nanoparticles-catalyzed synthesis of 1,2,3-triazoles.

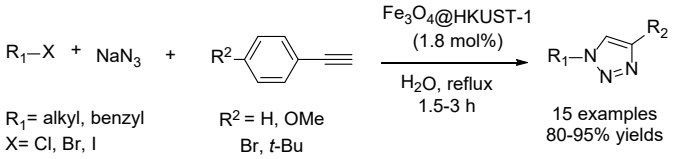


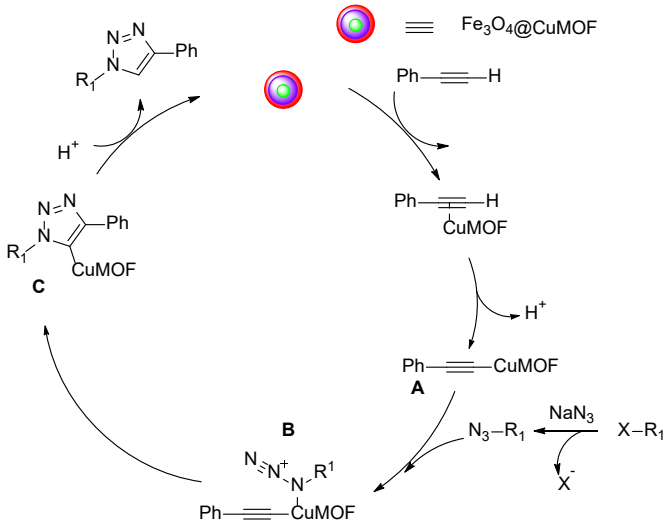
Scheme 41. Cu/SiO_2-Pr-NH-Benz as a catalyst for the synthesis of 1,2,3-triazoles.

In 2022, Velpula *et al.* developed the Cu_2O–Fe_3O_4@TNT nanocatalyst for the synthesis of 1,4-disubstituted 1,2,3 triazoles through three-component reaction of sodium azide, organic halides, and alkynes. The reaction was performed under mild condition and resulted in products in good to excellent yields with broad substrate scope. High efficiency, mild reaction conditions, the use of water as a solvent, and recyclability of the catalyst are the main advantages of the synthesis (**Scheme 44**) [88]. Tajbakhsh *et al.* described the preparation of immobilized Cu(I) in thiosemicarbazide-functionalized *β*-cyclodextrin (Cu@TSC-ß-CD) and the characterization of the nanomaterial by FT-IR, TGA, XRD, SEM, EDS, and ICP-OES measurements. The material then



Scheme 42. Al_2O_3@PCH-Cu(II) as an efficient catalyst for the synthesis of 1,4-disubstituted 1,2,3-triazoles.

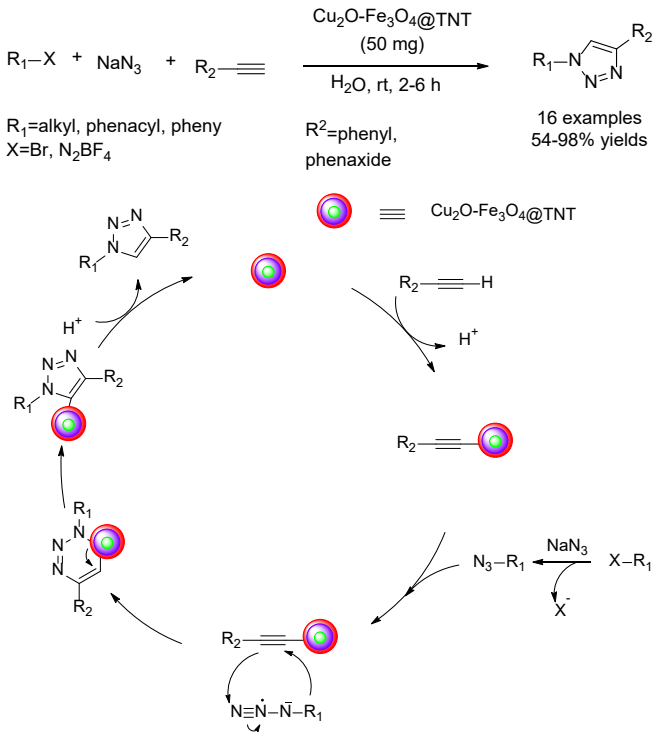




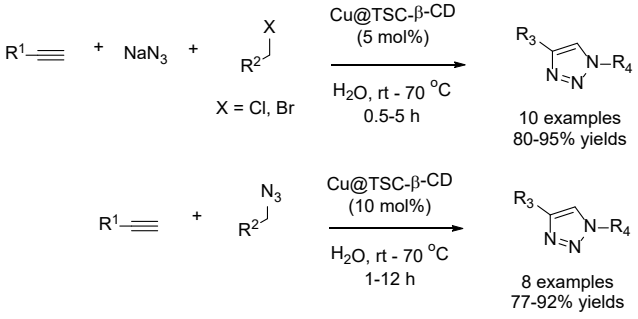
Scheme 43. Fe_3O_4@HKUST-1-catalyzed synthesis of 1,2,3-triazoles.

was successfully applied as a catalyst for the synthesis of 1,4-disubstituted-1,2,3-triazoles *via* alkyne–azide 1,3-dipolar cycloaddition. The synthesis provided products in high yields. The catalyst could be easily recovered by simple anti-solvent precipitation and filtration, and reused at least 7 times without significant drop in reaction yields (**Scheme 45**) [89]. ICP-OES analysis of the filtrate indicated that insignificant amount for the leaching of copper species was detected.

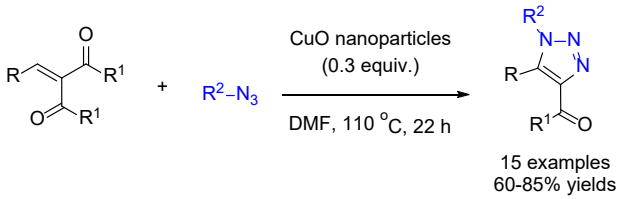
Rengasamy *et al.* employed CuO nano particles as a catalyst for the synthesis of 1,4,5-trisubstituted 1,2,3-triazoles by [3+2] cycloaddition of benzylidene diketones and organic azides. A wide range of products was isolated in high yields and the catalyst still showed good catalytic activity for four additional runs (**Scheme 46**) [90]. Babamoradi *et al.* illustrated the preparation of LDH@CS@PST/Cu nanomaterial and the characterization of the material by HNMR, ^{13}C NMR, FE-SEM, FT-IR, XRD, EDX, ICP-OES, and TGA/DTA methods. The nanomaterial exhibited excellent catalytic activity for the three-component reaction of phenacyl bromides, sodium azide, and phenyl acetylene. Various products were achieved in excellent yields in short time. The catalyst could be simply recovered from the reaction mixture by filtration and washing. The Cu content after eight successive runs was almost similar to the fresh catalyst (5.48 in comparison with 5.54%) indicating negligible copper leaching. This could explain why the catalyst could be reused up to eight cycles with negligible decrease in reaction yields (**Scheme 47**) [91]. The synthesis featured high TON and TOF values. Mechanistically, sodium azide reacts with phenacyl bromide to give azide **1**, which coordinates with CuI nanoparticles loaded on the LDH@CS@PST to generate the intermediate **B**. Finally, the desired product is provided by a sequence of ring



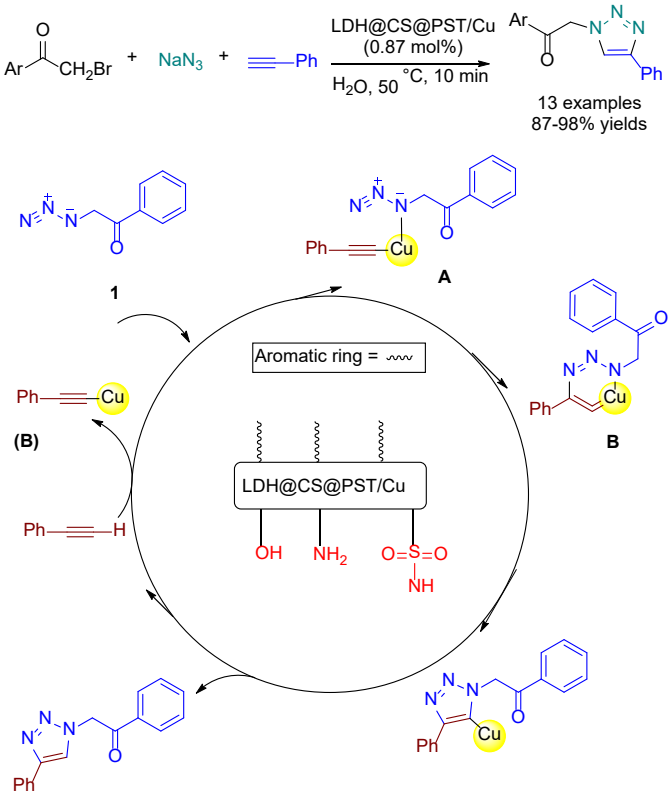
Scheme 44. Synthesis of 1,2,3-triazoles using Cu_2O–Fe_3O_4@TNT nanocatalyst.



Scheme 45. Cu@TSC-β-CD-catalyzed synthesis of 1,2,3-triazoles.

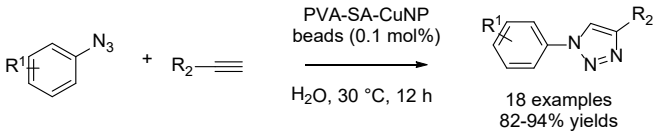


Scheme 46. The use of CuO nano particles as a catalyst for the synthesis of 1,2,3-triazoles.

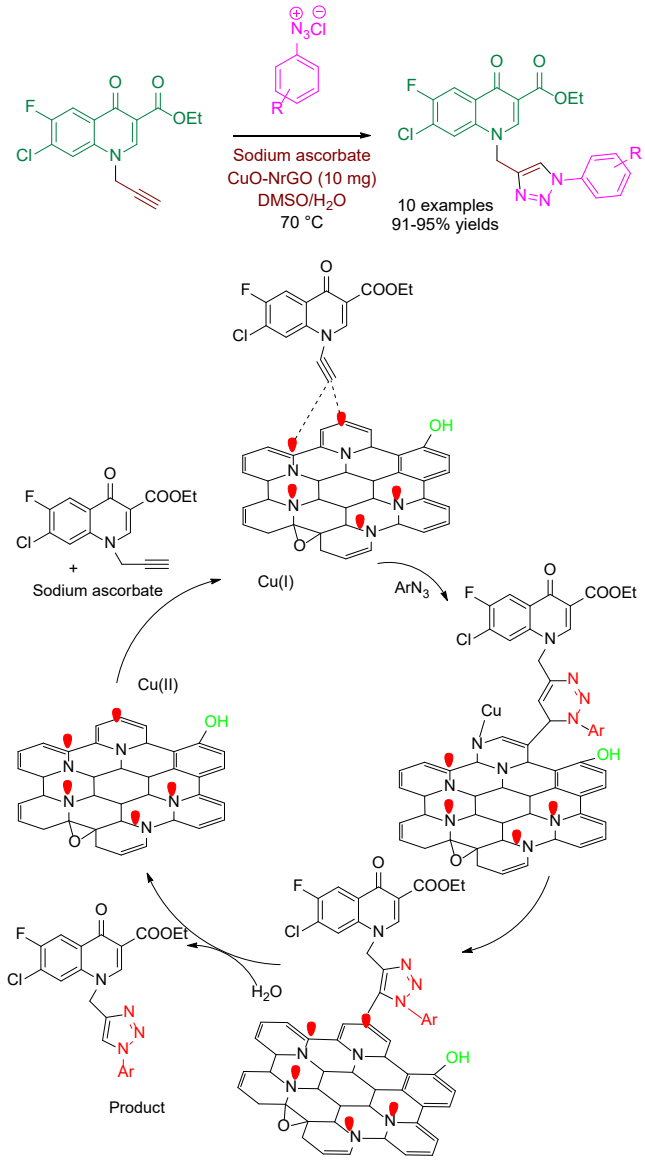


Scheme 47. 1,2,3-triazole synthesis demonstrated by Babamoradi *et al.* [91].

contraction and demetallation. Kumar *et al.* synthesized popper nanoparticles immobilized in polyvinyl alcohol and sodium alginate (PVA-SA-CuNPs) and characterized its structure by FE-SEM, EDX, FE-TEM, TGA, XRD, and UV-vis analyses. The nanomaterial was then employed as a catalyst for the assembly of 1,2,3-triazoles *via* click reaction of phenyl azides and terminal alkynes. A diverse series of products was obtained in good to excellent yields under mild reaction conditions. In addition, the nanocatalyst could be reused for five cycles with negligible decrease in reaction yields (**Scheme 48**) [92].



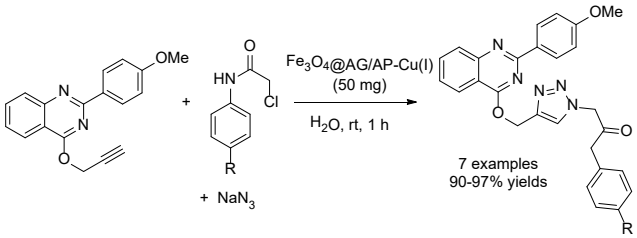
Scheme 48. Efficient triazole synthesis using PVA-SA-CuNPs catalyst.



Scheme 49. CuO-NrGO nanoparticles as an excellent catalyst for triazole synthesis.

In 2023, Singh *et al.* explored a novel method to access 1,2,3-triazole derivatives by reaction between terminal alkynes and azides using CuO-NrGO nanoparticles as a catalyst. All products were achieved in excellent yields. The SEM and EDX analysis of reused nanoparticles indicated that even after use of seven consecutive catalytic cycles, structure of catalyst was almost the same as the fresh one. Agreement with this result, the nanocatalyst could be reused for seven cycles without any significant loss of its activity (**Scheme 49**) [93]. Khaleghi *et al.* synthesized Fe_3O_4@AG/AP-Cu(I) nanocomposite and used this material as catalyst for the preparation of 1,2,3-triazoles through click reaction of terminal alkyne, organic halide, and sodium azide. Excellent yields of products, recyclability of the catalyst, the use water as a green solvent, relatively short reaction time, and mild reaction conditions are the salient features of the synthesis (**Scheme 50**) [94]. FTIR analysis revealed that the structure of recovered catalyst remained intact even after six reaction cycles.

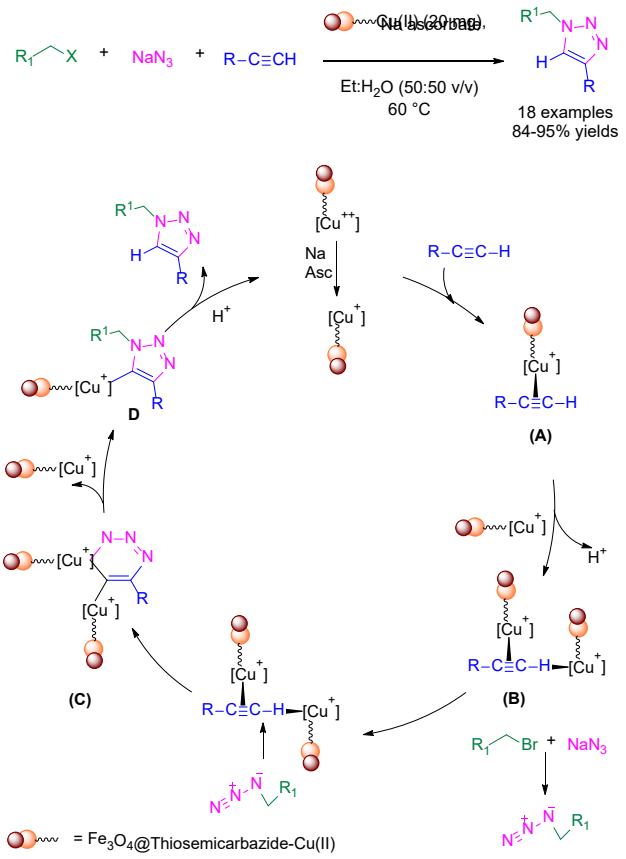
In 2024, Zond *et al.* demonstrated the synthesis of Fe_3O_4@ Thiosemicarbazide-Cu(II) and the characterization of the material by FT-IR, XRD, EDX, TGA-DTA, VSM, TEM, SEM, BET, ICP-AES techniques. The nanomaterial then was utilized as a catalyst for the construction of 1,2,3-triazoles *via* three-component reaction of organic halides, sodium azide, and terminal alkynes. The triazole synthesis showed many salient features such as clean work-up procedure, mild reaction conditions, tremendous yields, short reaction time, catalyst recyclability, and broad substrate scope (**Scheme 51**) [95]. A plausible mechanistic pathway for the synthesis of 1,2,3-triazole catalyzed by Fe_3O_4@Thiosemicarbazide-Cu(II) was suggested. Initially, *in situ* reduction of Cu(II) in the presence of sodium ascorbate generate Cu(I) which reacts with alkyne upon gives Cu(I) acetylide **A**. Subsequently, organic azide (generated *in situ* from sodium azide and halides) undergoes addition to Cu acetylide to form the intermediate **D** *via* the intermediate **C**. Finally, desired product was obtained from the intermediate **D** by protonolysis. García-García *et al.* introduced a novel copper biocatalyst based on



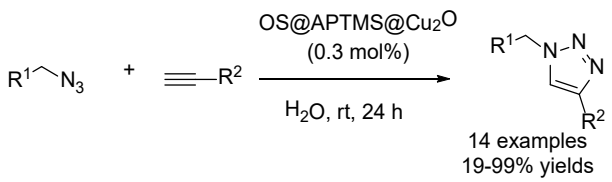
Scheme 50. Fe_3O_4@AG/AP-Cu(I)-catalyzed synthesis of triazole at room temperature.

silanized olive stone (OS) functionalized with copper oxide. The nanomaterial was characterized by XPS, FTIR-ATR, XRF, EM, TGA, moisture content, electrophoretic mobility and PSD analyses. The nanomaterial was used as a catalyst for the synthesis of 1,2,3-triazoles through the click reaction. The synthesis was performed under mild reaction conditions resulting products in high yields (in most cases). Recyclability of catalyst and scalability are the two other advantages of the synthesis (**Scheme 52**) [96].

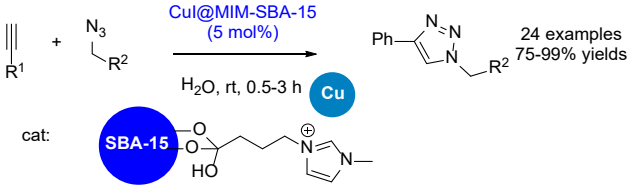
Galvan *et al.* accomplished the construction of 1,4-disubstituted 1,2,3-triazoles from organic azides and alkynes under ultra sound irradiation using CuI@MIM-SBA-15 as a catalyst. A diverse array of products was delivered in moderate to excellent yields under mild reaction conditions. The method was efficiently applied for the multicomponent synthesis of 1,2,3-triazoles from alkynes, bromides, and KN_3 in one step, although the reaction requires longer times. XPS analysis showed negligible change in catalyst structure (compared to the fresh one) and this explained the excellent reusability of the nanocatalyst (**Scheme 53**) [97]. Khaleghi *et al.* prepared Fe_3O_4@CG/CPTMS/OL-Cu, a novel hybrid magnetic material, and characterized its structure by FTIR, XRD, SEM, EDS, and VSM analyses. The nanomaterial showed excellent catalytic activity for the synthesis of triazole from azide and terminal alkynes. High yields of products, the use of water as a green solvent, and catalyst recyclability are the main attractive features of the synthesis (**Scheme 54**) [98]. Ashrafi *et al.* prepared novel $Cu_3Fe_4V_6O_{24}$ nanoparticles (NPs) *via* a facile sol-gel method. After characterizing, the material was applied as a catalyst for azide-alkyne 1,3-dipolar cycloaddition reaction to access triazoles. Various products were isolated in moderate to excellent yields. The catalyst remained highly active for the reaction even for the fifth cycle despite of low catalyst loading (**Scheme 55**) [99].



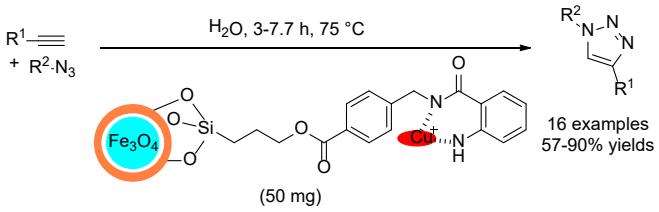
Scheme 51. The use of Fe_3O_4@ Thiosemicarbazide-Cu(II) for the click reaction.



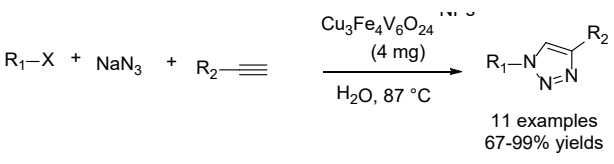
Scheme 52. OS@APTMS@Cu_2O-catalyzed synthesis of 1,2,3-triazoles.



Scheme 53. Efficient synthesis of 1,2,3-triazoles using CuI@MIM-SBA-15 catalyst.

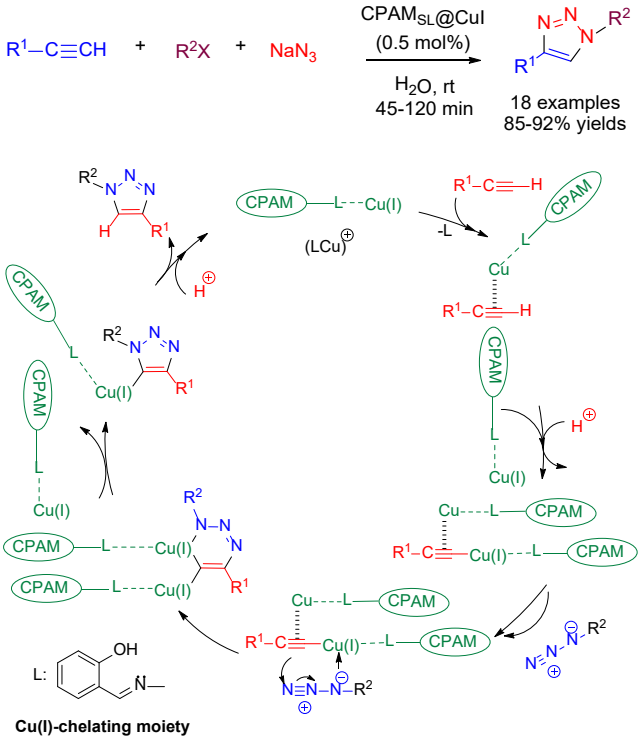


Scheme 54. Fe_3O_4@CG/CPTMS/OL-Cu-catalyzed synthesis of 1,2,3-triazoles in water.



Scheme 55. The use of $Cu_3Fe_4V_6O_{24}$ NPs as a catalyst for 1,2,3-triazole synthesis.

Recently, Rahmatpour *et al.* demonstrated the fabrication of CPAM SL @CuI nanomaterial and its characterization by FTIR, ICP, DR-UV–VIS, FTIR, XRD, FE-SEM, EDAX, TEM, TG/DTGA, and elemental analyses. The nanomaterial then was employed as an efficient catalyst for triazole synthesis from alkyl or benzyl halides, sodium azide, and terminal alkynes. The triazole synthesis featured many advantages such as greener solvent, mild reaction conditions, broad substrate scope, recyclability of the catalyst (for six runs), scalability (10 mmol), and excellent yields of products. A tentative reaction mechanism explaining the role of the catalyst was also demonstrated (**Scheme 56**) [100]. ICP, FE-SEM, FTIR, and XDR analyses confirmed negligible change in catalyst structure even after six cycles. Arjmandzadeh *et al.* synthesized humic acid-based nanomagnetic copper(II) composite and characterized the structure of the material by SEM, TEM, XRD, EDX, EDS-mapping, VSM, TGA, AAS, and FT-IR methods. The nanomaterial then was employed as a catalyst for the synthesis of 1,2,3-triazole derivatives by reaction of halides or oxiranes, sodium azide, and terminal alkynes in water at room temperature. A diverse series of products was isolated in good to excellent yields under mild reaction conditions. The catalyst was easily recovered using an external magnetic field and still showed high catalytic activity for three additional runs (**Scheme 57**) [101]. A presumable reaction mechanism was also illustrated.

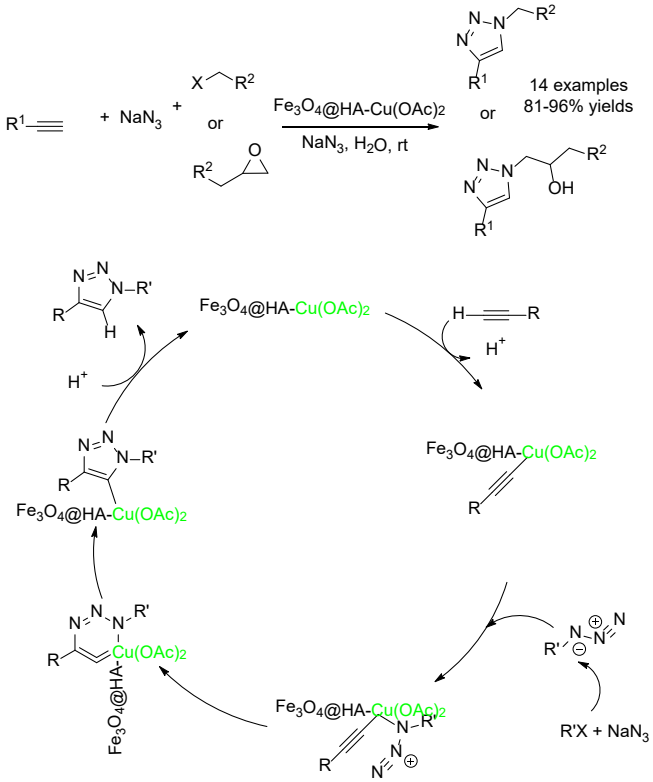


Scheme 56. Preparation of 1,2,3-triazoles using CPAM SL @CuI catalyst.

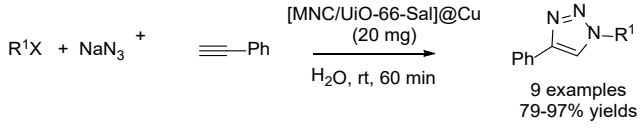
Mohammadi *et al.* fabricated a novel hybrid magnetic nanocatalyst based on magnetic nanocellulose, NH_2-UiO-66 nanocrystals modified with salicylaldehyde, and immobilized copper ions. The nanomaterial then was examined by FTIR, XRD, EDX, TGA, VSM, and FE-SEM analyses. After that, the nanomaterial was applied as a catalyst for 1,2,3-triazole synthesis. Nine products were afforded in in good to excellent yields under mild reaction conditions. The catalytic activity remains almost unchanged after four cycles thank to its stability (confirmed by FTIR and XRD analyses) (**Scheme 58**) [102].

3. 1,2,3-Triazole Synthesis Using Other Nanocatalyts

Paplal *et al.* designed a methodology for the assembly of 1,4-disubstituted triazoles from azides, nitroalkenes or chalcones using Bi_2WO_6 nanoparticles as catalyst in presence of $CuSO_4.5H_2O$ and sodium ascorbate. A diverse range of products was delivered in high yields. The catalyst system (Bi_2WO_6 + $CuSO_4.5H_2O$ +



Scheme 57. Versatile synthesis of 1,2,3-triazoles using Fe_3O_4@HA-Cu(OAc)$_2$ catalyst.

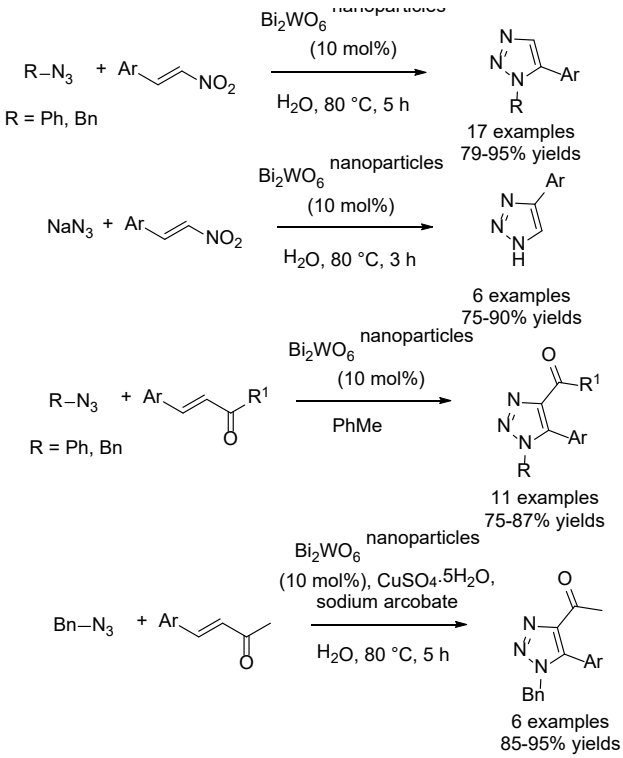


Scheme 58. [MNC/UiO-66-Sal]@Cu-catalyzed synthesis of 1,2,3-triazoles.

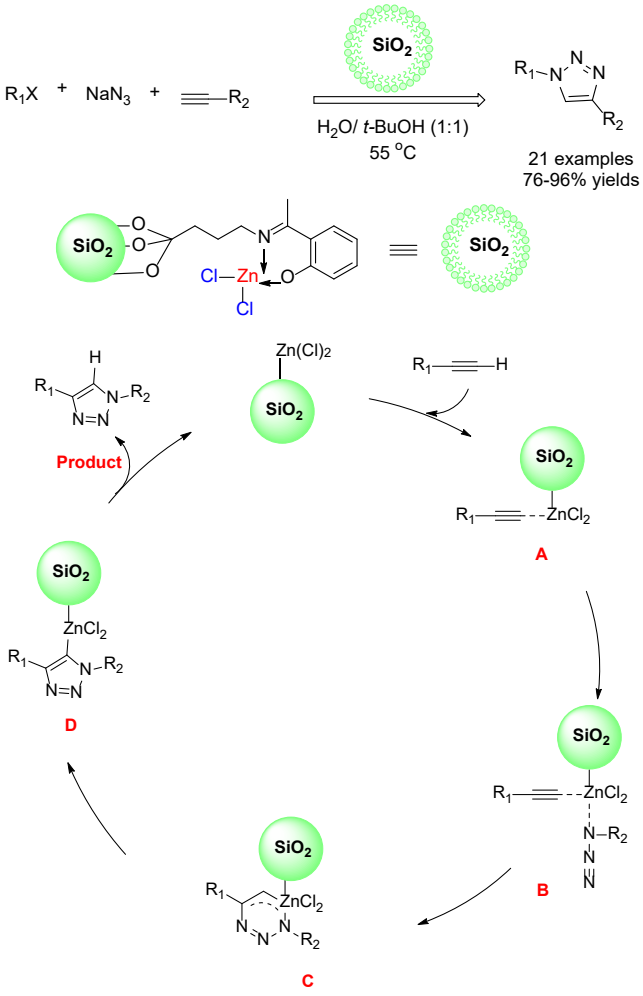
sodium ascorbate) could be reused for five runs without any considerable loss of activity (**Scheme 59**) [103]. XRD analysis indicated no changes in the morphology even of the catalyst even after 5 cycles.

Sharma *et al.* reported the synthesis of SiO_2@APTES@2HAP-Zn nanomaterial and its characterization by ^{13}C CPMAS NMR spectroscopy, FT-IR, XRD, BET surface area analysis, SEM, AAS, ED-XRF, and elemental analysis. The nanomaterial was then successfully applied as a catalyst for triazole synthesis by click reaction of halides, NaN_3, and terminal alkynes. A diverse array of products was provided in good to excellent yields. SEM analysis of the reused catalyst after the sixth run showed no significant structure difference compared to the fresh one and this result explained why the catalyst could be recycled six times without any significant loss in its activity. A plausible reaction pathway describing the role of the catalyst was also suggested (**Scheme 60**) [104]. The synthesis featured high TON value fot every product. Initially, zinc coordinates with terminal alkyne to generate the acetylide complex **A**, which reacts with azide to produce the complex **B**. Cyclization of the complex B produces the triazolide intermediate **C**, which was converted to the heterocyclic complex **D** by ring contraction. Finally, protonolysis of **D** furnishes the desired product and regenerating the active catalyst.

In 2017, Basua *et al.* fabricated a novel silver-nanomaterial by grafting of Ag NPs on the surface of -Al_2O_3 cores and -Fe_2O_3 shells. The material then was utilized as a catalyst for triazole synthesis *via* click reaction. Six products were isolated in excellent yields. The catalyst could be reused for five cycles with negligible drop in reaction yield (**Scheme 61**) [105]. A reaction pathway illustrating the role of the catalyst was included. Paplal *et al.* examined a strategy to access 1,2,3-triazoles *via* oxidative [3 + 2]-cycloaddition reactions of azides with *β*-nitrostyrenes and chalcone derivatives using $Zn(OAc)_2$-*t*-BuOOH or ZnO nanoparticles as catalyst system in aqueous medium. A diverse series of products was achieved in moderate to excellent yields and regioselectivity (**Scheme 62**) [106]. The use of ZnO nanoparticles afforded products in higher yields than $Zn(OAc)_2$-*t*-BuOOH. XRD analysis indicated that structure of ZnO recovered catalyst after 6th cycle was almost the same as the fresh one. Mechanistically, the nitrostyrene is activated by the chelating $Zn(OAc)_2$ and then reacts with azide to give the intermediate I *via* [3 + 2]-cycloaddition



Scheme 59. Bi_2WO_6 nanoparticles as an efficient catalyst for the assembly of 1,4-disubstituted triazoles.

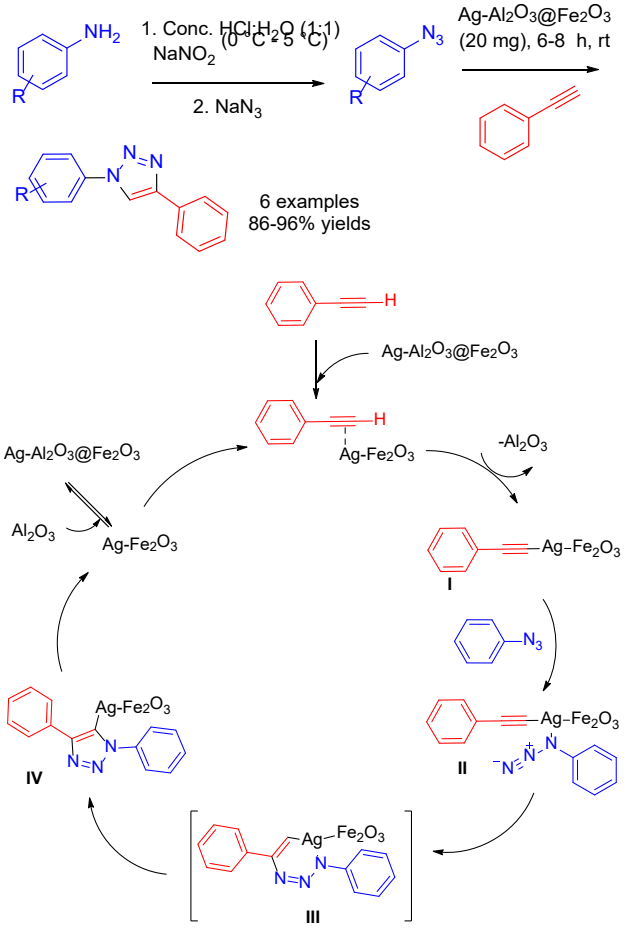


Scheme 60. Synthesis of 1,2,3-triazoles using SiO_2@APTES@2HAP-Zn nanocatalyst.

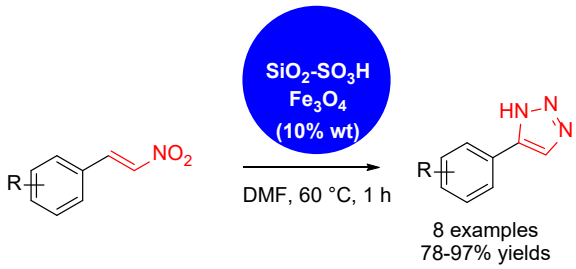
reaction. This intermediate is then oxidized in presence of TBHP to give the desired product.

Jiang *et al.* accomplished an efficient synthesis of 1,2,3-triazoles using Fe_3O_4@SiO_2-SO_3H (MSS-SO_3H) as a catalyst. Various products were furnished in moderate to excellent yields. The MSS-SO_3Zn catalyst could be recovered easily by an external magnet, and reused without significant change in reaction yields (**Scheme 63**) [107]. Garg *et al.* employed silica-supported silver complex, Ag–NHC@SiO_2 to synthesize triazole *via* click reaction of benzyl azides and phenyl acetylene. A wide range of products was obtained in good to excellent yields. Aryl azides with strong electron-withdrawing groups were found to react sluggishly. ICP-AES analysis of the reused catalyst confirmed a tiny amount of decrease in the metal loading and this proved the catalyst reusability for five cycles ((1st cycle 98% to 5th cycle 86%) despite of low catalyst loading (**Scheme 64**) [108].

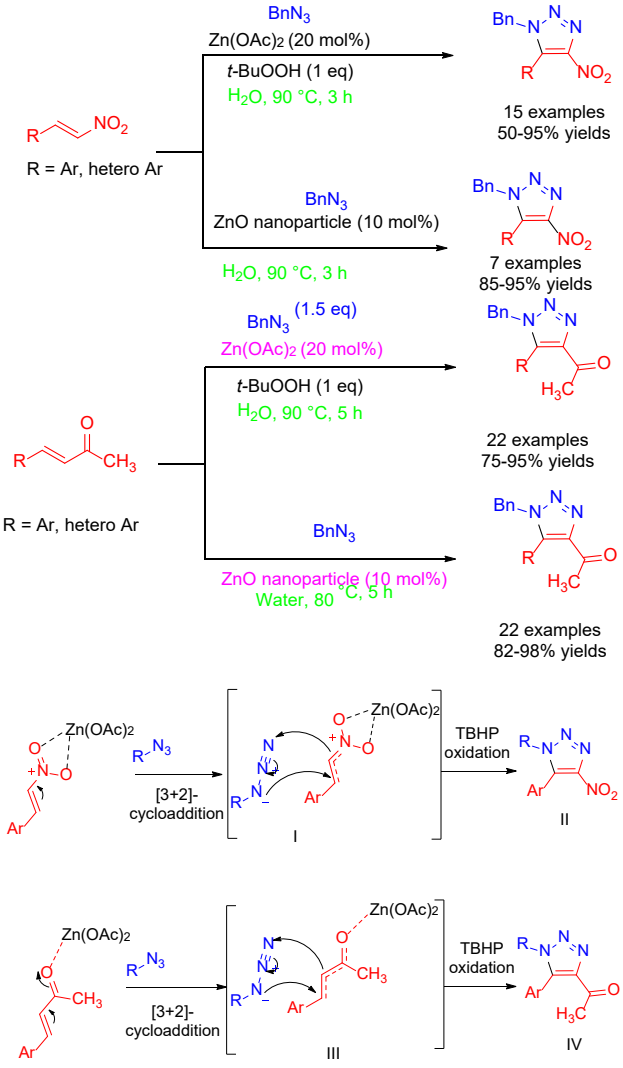
Mirzaei-Mosbat *et al.* prepared Fe_3O_4@SiO_2@Propyl-HMTA nanomaterial and applied the nanomaterial as a catalyst for the assembly of 4-aryl-NH-1,2,3 triazoles *via* one-pot, three-component reaction of aryl aldehydes, nitromethane, and NaN_3. Various products were obtained in good to excellent yields. A tentative



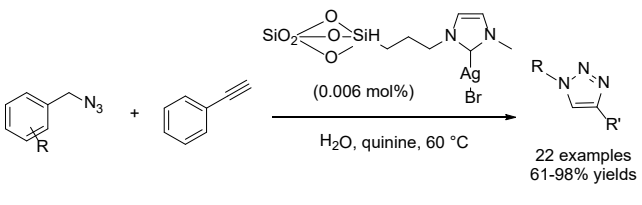
Scheme 61. Ag-Al_2O_3@Fe_2O_3-catalyzed synthesis of triazoles under mild conditions.



Scheme 63. Fe_3O_4@SiO_2-SO_3H-catalyzed synthesis of 1,2,3-triazoles.



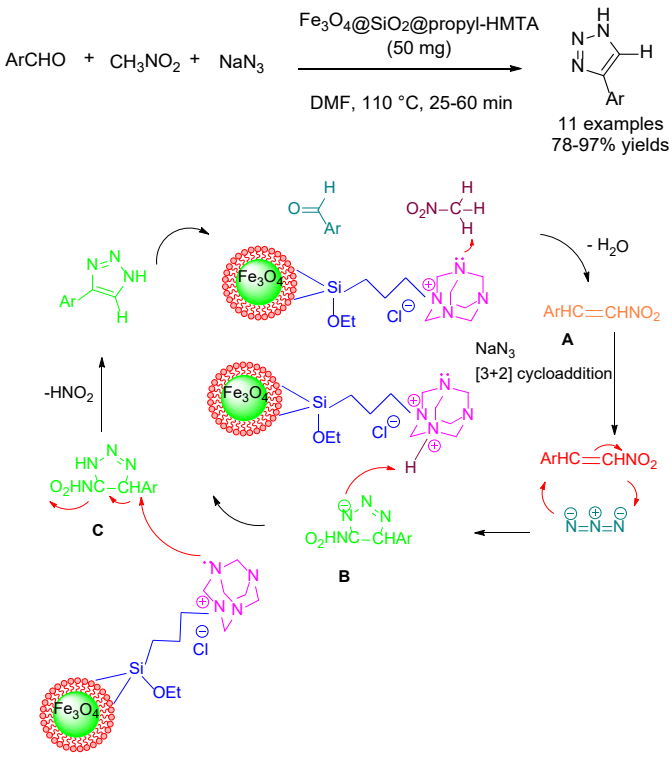
Scheme 62. Synthesis of triazoles completed by Paplal *et al.* [62].



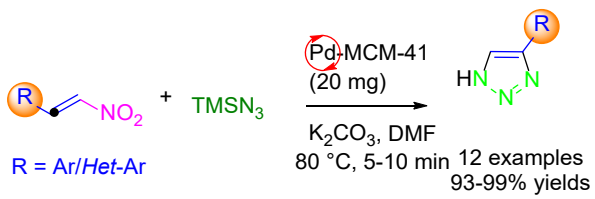
Scheme 64. The use of Ag–NHC@SiO_2 as an efficient catalyst for the synthesis of 1,2,3-triazoles.

reaction pathway explaining the formation of desired products was also included. SEM analysis confirmed no significant change in the morphology of the catalyst after five cycle and this explained the excellent reusability of the catalyst (**Scheme 65**) [109]. A plausible mechanism for the synthesis of 4-aryl-*N*H-1,2,3- triazole derivatives using Fe_3O_4@SiO_2@Propyl-HMTA catalyst was proposed. Initially, activated nitro-methane reacts with aldehyde to form the intermediate **A** (Henry reaction), which then undergoes a [3+2] cycloaddition with azide to give the intermediate **B**. Finally, elimination of HNO_2 by catalyst from **B** furnishes the desired product. Saha *et al.* disclosed a strategy to access triazoles by de-nitrative [3+2] cycloaddition of nitrostyrenes with $TMSN_3$ using mesoporous nano-Pd-MCM-41 catalyst. Products were afforded in excellent yields. The nanocatalyst still showed excellent catalytic activity for eight runs thank to insignificant Pd leaching (confirmed by TEM and AAS analyses) (**Scheme 66**) [110].

Rajabi-Moghaddam *et al.* demonstrated the synthesis of copper(II)-coated magnetic core–shell



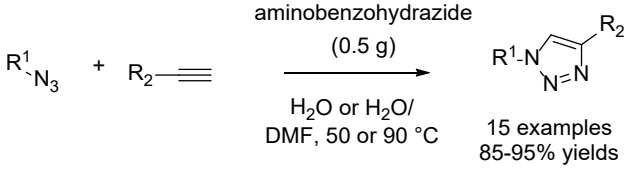
Scheme 65. Synthesis of 4-aryl-NH-1,2,3 triazoles from aryl aldehydes, nitromethane, and sodium azide.



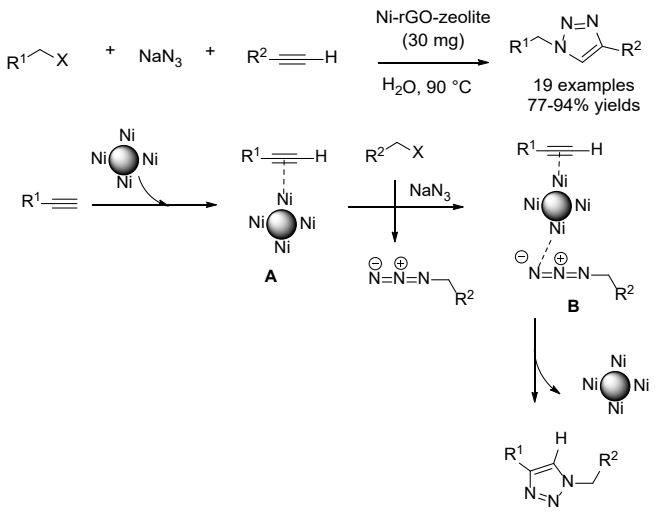
Scheme 66. Synthesis of triazoles from nitrostyrenes and $TMSN_3$.

nanoparticles Fe_3O_4@SiO_2 modified by isatoic anhydride and the characterization of the material by modern physical analyzes. The nanomaterial the was efficiently applied as a catalyst for the preparation of 1,2,3-triazole derivatives *via* the click reaction of azides and terminal alkynes. A diverse series of 1,2,3-triazoles was produced in excellent yields and the catalyst could be reused for five cycles with minimum loss of activity (**Scheme 67**) [111].

Choudhury *et al.* employed Ni-based nanocomposite catalyst (Ni–rGO–zeolite) as a catalyst for the construction of 1,2,3-triazoles by three-component of organic halides, NaN_3, and monosubstituted acetylenes. A diverse range of products was afforded in good to excellent yields. High efficiency, the use of water as solvent, and recyclability of the catalyst are the main attractive features of the synthesis. FT-IR, XRD and Raman analyses revealed that there is no significant leaching of nickel occurred from the nanocomposite during the reaction. A plausible reaction mechanism describing the role of the catalyst was outlined (**Scheme 68**) [112]. Initially, Ni catalyst coordinates to alkyne to generate the complex **A**, which then reacts with the in situ generated organic azide to provide the intermediate **B**. Regioselective cycloaddition of B affords the target triazole along with regeneration of the catalyst.



Scheme 67. Preparation of 1,2,3-triazoles under Fe_3O_4@SiO_2-2-Aminobenzohydrazide catalyst.



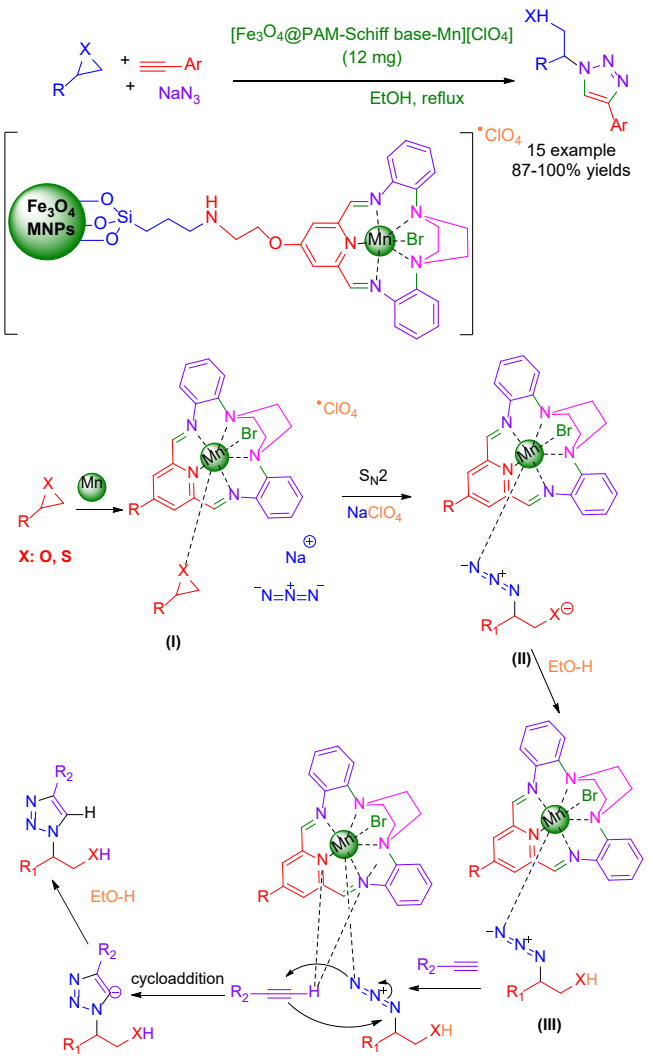
Scheme 68. Ni–rGO–zeolite as an efficient catalyst for the assembly of 1,2,3-triazoles.

Jasim *et al*. synthesized Fe_3O_4@PAM–Schiff-base–Mn][ClO_4] heterogenized complex and characterized its structure using modern physical techniques. The nanomaterial was then applied for the preparation of 1,2,3-triazoles. A broad range of products was provided in excellent yields. It was observed that there was no leaching of Mn from the nanocatalyst after eight cycles. This result evidenced the excellent reuse of the catalyst (**Scheme 69**) [113]. A plausible reaction mechanism based on the Huisgen 1,3-dipolar cycloaddition was also suggested.

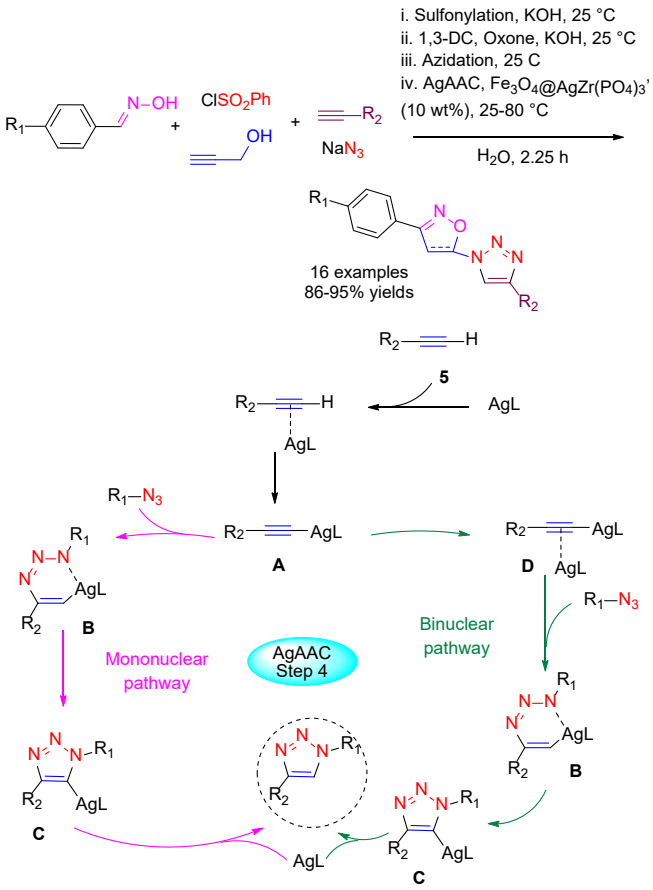
Mokhi *et al*. demonstrated the synthesis of Fe_3O_4@$AgZr_2(PO_4)_3$ nanocomposite and the characterization of the material by FT-IR, SEM, EDX, and XRD analyses. The nanomaterial showed excellent catalytic activity for the assembly of isoxazoline-linked 1,2,3-triazoles. The synthesis proceeded *via* a sequence of sulfonylation, 1,3-dipolar cycloaddition, azidation, and click reaction. A wide range of products was achieved in excellent yields under ultrasound cavitation conditions. The structure of the reused catalyst was reinvestigated after five cycles by using FT-IR spectroscopy, and no considerable change was observed. That is why the nanocatalyst could be reused for five runs without any significant loss of reactivity (**Scheme 70**) [114].

4. Conclusions

In summary, we have described an overview on the synthesis of 1,2,3-triazole derivatives using nanocatalysts in this study. 70 studies from 2014 have been collected and analyzed. We have also tried to describe reaction mechanisms where possible. Most of studies involve the use of nanocatalysts for the click reaction between alkynes and azide. Among these catalysts, copper nanomaterials have been the most common. In most studies, the nanocatalysts could be recovered and reused with high efficiency. In several studies, gram scale synthesis was performed, although industrial implementation remains a big chalange. Due to time constrain and lack of data from published articles, details about reaction mechanisms have not been analyzed. Undoubtedly, the use of nanocatalysts for the assembly of 1,2,3-triazoles will be exploited in the future due to the recyclability of these materials. Besides, continuous-flow click



Scheme 69. Fe_3O_4@PAM–Schiff-base–Mn][ClO_4]-catalyzed synthesis of 1,2,3-triazoles.



Scheme 70. Multistep synthesis of 1,2,3-triazoles.

chemistry, mechanochemical synthesis, photocatalytic and electrocatalytic click reactions, AI-assisted catalyst design may have a bigger contribution to 1,2,3-triazole synthesis.

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CRediT Author Statement

Author Contributions: *I. Nguyen Van Quoc*: Conceptualization, Methodology, Investigation, Resources, Data Curation, Writing, Review and Editing, Supervision; *Nguyen Thi Phuong Thao*: Conceptualization, Methodology, Formal Analysis, Data Curation, Writting Draft Preparation, Visualization; *Dau Xuan Duc*: Investigation, Resources, Writing, Review and Editing, Validation. All authors have read and agreed to the published version of the manuscript.

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