Received: 24 March 2020 | Revised: 18 June 2020 | Accepted: 1 July 2020
DOI: 10.1002/aoc.5959

FULL PAPER

Applied Organometallic Chemistry WILEY

Immobilization of copper nanoparticles on WO_3 with enhanced catalytic activity for the synthesis of 1,2,3-triazoles

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In this study, a heterogeneous catalyst based on copper nanoparticles immobilized on metal oxide, WO_3, was fabricated using an impregnation method as an easy and straightforward nanoparticle synthesis strategy. The successful synthesis of the nanocatalyst was confirmed using various spectroscopic techniques such as X-ray diffraction, scanning electron microscopy, energy dispersive X-ray analysis, and transmission electron microscopy. The catalytic performance of the well-characterized material was evaluated through the azide–alkyne cycloaddition reaction (click reaction) in the aqueous medium. To optimize reaction conditions, different reaction parameters such as nanocatalyst amount, reaction time, temperature, and solvents were studied. Experimental results showed that as-prepared nanocatalyst (Cu/WO_3) could act as an effective and reusable heterogeneous catalyst in water for the synthesis of 1,2,3-triazoles in good-to-excellent yields. In addition, Cu/WO_3 has some advantages such as simple preparation procedure, easy separation, and recyclability for three runs with no remarkable loss of catalytic activity, which is essential from a catalytic application point of view.

KEYWORDS
catalytic activity, click reaction, copper, nanoparticles, WO_3

1 | INTRODUCTION

Rational design and synthesis of structurally complex heterocyclic molecules have attracted much attention from synthetic organic and medicinal chemists.[1] Among them, triazoles,[2] especially, N-substituted 1,2,3-triazoles, as substantial intermediates have more potential utilization in the synthesis of some important drugs with unique biological and pharmaceutical properties such as antiviral, antitubercular,[3] antidiabetic, anticancer,[4] selective β3 adrenergic receptor agonist, and anti-HIV activities.[5] Traditionally, the synthesis of 1,2,3-triazoles was carried out in harsh reaction conditions and often led to the two regioisomers by using asymmetric alkynes.[6] In 2002, Sharpless et al.[7] successfully synthesized these compounds in the presence of catalytic amounts of Cu(I) at ambient temperature with high regioselectivity in a short time. In general, organic reactions in homogenous media have some disadvantages, such as low catalytic performance, difficulty in product purification, and catalyst recyclability.[8] Using heterogeneous versions of catalysts could be considered as an effective approach to reduce these limitations.[9] However, despite various useful advantages of heterogeneous catalysts, their low activity and aggregation tendency are reported to be challenging.[10] The solution to these problems is the immobilization of various types of metal nanoparticles (NPs) on different heterogeneous supports.[11] For example, a broad range of polymeric materials have been used as active solid supports for copper NPs (Cu NPs) in the click synthesis of 1,2,3-triazoles between azides and alkynes.[12] In addition, massive research interests have been devoted to transition metal oxide materials owing to their exceptional physicochemical properties, and over the past two decades, they have been employed extensively as solid supports for various metal NPs.[13] Among all existing transition metal oxides, WO_3—with its excellent physical properties such as piezoelectricity, electrical conductivity, and defect structures—has gained prominence as one of the useful supports for metal NPs.[14] For instance, prepared Cu/WO_3 materials have wide applications in gas sensors and solid-state drives.[15] From a catalytic application point of view, no reports on various organic reactions in the presence of Cu/WO_3 have been reported.[13] Further to our research on the synthesis and applications of various nanomaterials,[16] in this study, WO_3 was synthesized by the impregnation method followed by immobilization of copper nanoparticles (Cu NPs) to prepare Cu/WO_3 nanomaterials, and then, for the first time, the catalytic activity of the synthesized Cu/WO_3 was evaluated in the click reaction.

2 | EXPERIMENTAL

2.1 | Materials and characterization instruments

Hydrochloric acid (HCl, 37%), $Na_2WO_4·2H_2O$, $Cu(NO_3)_2$, sodium azide, hydrazine hydrate, all alkynes (phenyl acetylene and propargyl alcohol), all organic halides (benzyl chloride, benzyl bromide, 2-nitrobenzyl chloride, 4-nitrobenzyl chloride, and 2-methylbenzyl chloride), and solvents were provided by Fluka and Merck (Germany) and utilized with no extra purification. Transmission electron microscopy (TEM, FEI Tecnai 20) was used to determine the structure, shape, and size of the sample. Scanning electron microscopy (SEM) analysis was carried out with Philips CM120 and LEO 1430VP instruments with an energy dispersive X-ray (EDX) spectroscopy. The X-ray powder profiles were recorded using a Bruker D8 ADVANCE (Germany) diffractometer (Cu-Kα radiation).

2.2 | Preparation of WO_3 and immobilized copper nanoparticles (Cu/WO_3)

The synthesis of WO_3 nanoparticles was carried out following our previously reported procedure.[17] In short, in a 25-ml round-bottom flask containing 10 ml H_2O, a certain amount of $Na_2WO_4·2H_2O$ (0.825 g, 2.5 mmol) was added and acidified with a 6 M HCl solution. The precipitated white solid $WO_3·nH_2O$ was washed thrice with water and dried at 50°C. Finally, to prepare WO_3 nanoparticles, the solid $WO_3·nH_2O$ was dehydrated at 500°C. An impregnation strategy was used to immobilize Cu NPs (Cu/WO_3) in the presence of $Cu(NO_3)_2·3H_2O$ as a source of Cu. In short, in a 50-ml round-bottom flask containing 20 ml methanol, WO_3 (0.1 g) and $Cu(NO_3)_2·3H_2O$ (0.2 g) were added and stirred vigorously for 24 h. After impregnation, Cu NPs were synthesized by a reduction of the Cu/WO_3 with 0.5 ml hydrazine hydrate. The final sample was filtered, washed with methanol, and dried at room temperature.

2.3 | General procedure for Cu/WO_3 base azide–alkyne cycloaddition

A round-bottom flask containing 2 ml H_2O was charged with Cu/WO_3 along with a certain amount of alkyne (0.5 mmol), organic halide (0.55 mmol), and NaN_3 (0.55 mmol). The reaction mixture was stirred at 90°C for 12 h. The total conversion of starting materials was monitored using thin-layer chromatography. Filtration was used for catalyst recovery, and the desired products were extracted with EtOAc (2 × 10 ml) without any additional purification steps.

3 | RESULTS AND DISCUSSION

3.1 | Characterization of the catalyst (Cu/WO_3)

The structure and phase formation of the samples were determined using X-ray diffraction (XRD) profiles of WO_3 support and Cu/WO_3 nanocatalyst. As shown in Figure 1, all WO_3 support peaks related to lattice reflection planes are in a good agreement with JC-PDS 01–075-2072.[18] Because a low amount of copper was immobilized on the support, there were no differences between the XRD profiles of WO_3 and Cu/WO_3. On the contrary, copper atoms occupied the lattice position of WO_3, resulting in the absence of copper peaks in the pattern of Cu/WO_3.[17]

The morphology and elemental composition of the synthesized Cu/WO_3 were studied using SEM and EDX analyses, respectively. Figure 2a,b shows the SEM images of Cu/WO_3 with two different magnifications. Spherical-shaped aggregated nanoparticles could be seen easily in Figure 2b. The EDX data are shown in Figure 2c, which

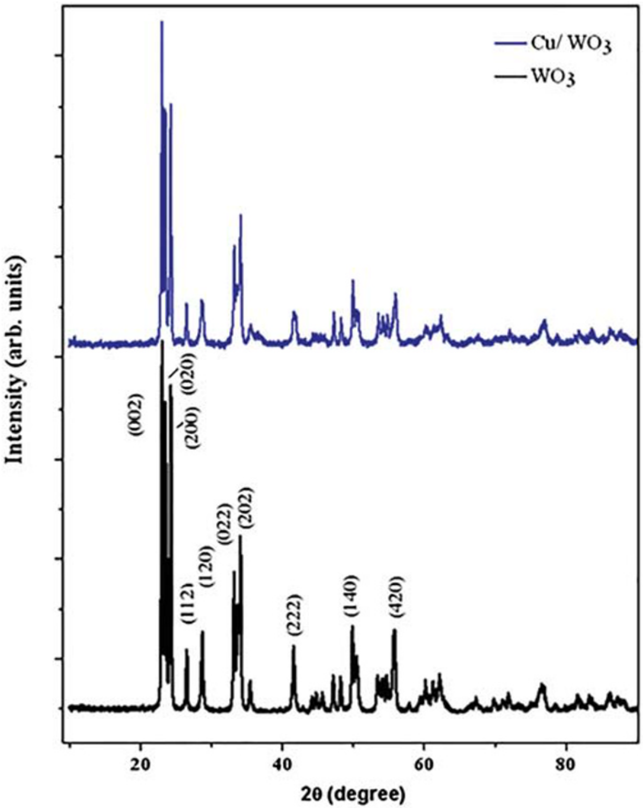


FIGURE 1 X-ray diffraction profiles of WO$_3$ and Cu/WO$_3$ nanoparticles

confirms the presence of Cu and W elements in the structure of Cu/WO$_3$ catalyst. The content of Cu and W was 12.78 and 76.03 wt%, respectively.

Immobilized Cu NPs with a size of about 2–5 nm on WO$_3$ support were observed in TEM image (Figure 3).

3.2 | Catalytic experiments

The catalytic activity of the synthesized Cu/WO$_3$ catalyst was evaluated in the 1,3-dipolar azide–alkyne cycloaddition reaction. For this, the reaction between phenylacetylene and benzyl chloride in the presence of sodium azide was selected as a model reaction.[12b] To optimize the reaction parameters, the model reaction was subjected to various reaction conditions as summarized in Table 1.

Entries 1–5 show the effect of catalyst amount on the yield of model reaction. In the absence of a catalyst (entry 1), there was no progress in the reaction, which demonstrated the need for the Cu/WO$_3$ catalyst to obtain the desired product. The product yield increased from 66 to 82% at 90°C when the amount of catalyst was increased from 1 to 5 mg. When the reaction temperature increased from room temperature to 90°C, there was a remarkable increase in the product yield (entries 5–8). As shown in entry 4 of Table 1, the product, 1-benzyl-4-phenyl-1,2,3-triazole, has the highest yield (82%) at 90°C after 12 h. Based on this result, 90°C was selected as the optimum reaction temperature in all the remaining experiments. Furthermore, the time dependency of the click reaction was evaluated and is presented in Table 1. By running the reaction in less than 12 h, the amount of the isolated desired product was low

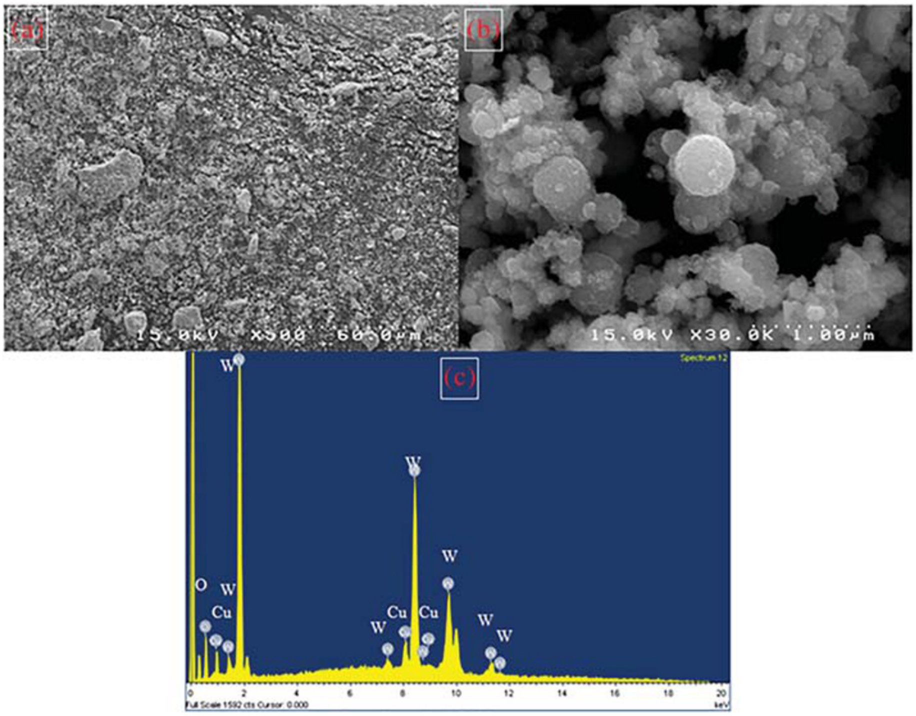


FIGURE 2 (a,b) Scanning electron microscopy images of the Cu/WO$_3$ catalyst with two different magnifications and (c) energy dispersive X-ray analysis of Cu/WO$_3$

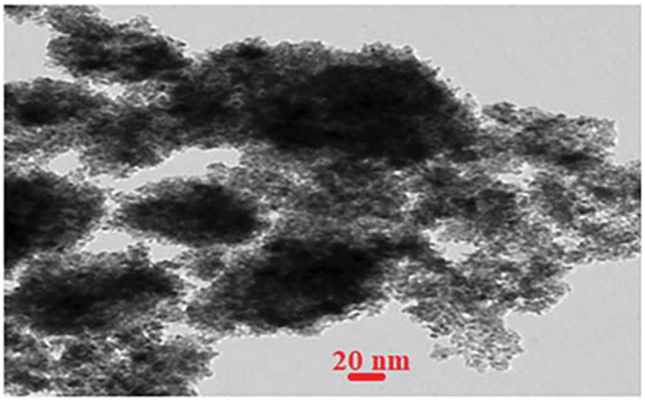
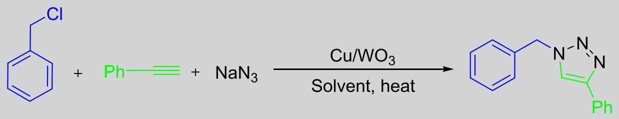


FIGURE 3 Transmission electron microscopy image of the Cu/WO_3 catalyst

(entries 8 and 9). The effect of various solvents on the catalytic activity of Cu/WO_3 was also investigated. In general, solvents as a reaction medium play an important role in increasing the solubility of the starting materials. As shown in Table 1 (entries 9–13), when the click reaction was catalyzed by Cu/WO_3, an increase in the reaction conversion was observed in the order of $H_2O > CH_3CN >$ acetone $> C_2H_5OH >$ toluene. On the contrary, there was no progress in reaction in toluene (entry 12), but in the case of water as a green solvent (entry 4), 82% product yield was achieved. The reason is that sodium azide salt is completely soluble in water, leading to the rapid synthesis of reaction intermediates. Finally, entry 4 was selected as the best optimized condition in which the model click reaction was conducted in the presence of 5 mg Cu/WO_3 catalyst in water at 90°C for 12 h. With this optimized condition, the scope of the general applicability of Cu/WO_3 catalyst was evaluated toward the reaction of different alkynes and substituted benzyl halides. Experimental results are summarized in Table 2. As could be seen, substitution variations on the phenyl ring of benzyl halides were investigated, and all of them yielded the desired products in good-to-excellent yields (entries 1–5). Due to steric hindrance, the *ortho* substitute of the nitro group on benzyl chloride led to a low product yield compared to its *para* substitute (entries 3 and 5). In addition, the product yield of aliphatic alkyne was less than that of phenylacetylene in all experiments (entry 6).

In general, the formation of 1,2,3-tetrazole via the click reaction proceeded smoothly to obtain the corresponding 1,4-disubstituted-1,2,3-triazole derivatives. A tentative stepwise mechanism (Figure 4) similar to those of other Cu(I)-catalyzed azide–alkyne cycloaddition reactions was proposed, involving the generation of the

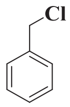
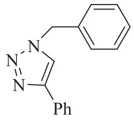
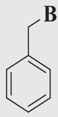
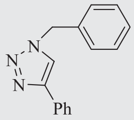
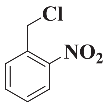
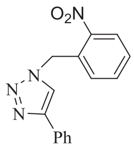
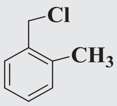
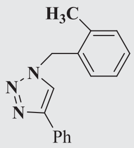
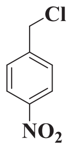
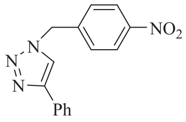
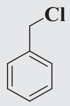
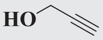
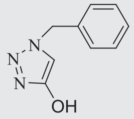
TABLE 1 Results of the azide–alkyne cycloaddition reaction at different reaction conditions



Entry	Catalyst amount (mg)	Solvent	*T* (°C)	Time (h)	Yield (%)[a]
1	0	H_2O	90	12	0
2	1	H_2O	90	12	66
3	3	H_2O	90	12	71
4	5	H_2O	90	12	82
5	5	H_2O	r.t	12	0
6	5	H_2O	40	12	48
7	5	H_2O	70	12	67
8	5	H_2O	90	4	55
9	5	H_2O	90	8	72
10	5	C_2H_5OH	90	12	48
11	5	CH_3CN	90	12	65
12	5	Toluene	90	12	N.R.[b]
13	5	Acetone	90	12	57

[a] Isolated yield.
[b] No reaction.

TABLE 2 Click reaction of different starting materials in the presence of Cu/WO_3 catalyst

Entry	Benzyl halide	Alkyne	Product	Yield (%)	Melting point (°C)^{ref}
1		Ph—≡		82	125–126 (126–127)[19]
2		Ph—≡		91	125–126 (126–127)[20]
3		Ph—≡		84	145–147
4		Ph—≡		70	106–107 (105–106)[21]
5		Ph—≡		95	156–157 (156–157)[22]
6				35	78–79 (78–78.5)[19]

copper(I) acetylide via the insertion of Cu(I) into terminal alkynes followed by the addition of the alkyl–azide intermediate.

To evaluate the reusability of the catalyst, a set of reactions between benzyl chloride, NaN_3, and phenylacetylene were conducted with water as a solvent. At the end of the first run, the used catalyst was removed from the reaction mixture by centrifugation, washed, and dried at 40°C to use for the next run. As shown in Figure 5, the catalyst could be recycled at least thrice without significant yield loss.

To date, many different catalysts with several disadvantages are being used for the synthesis of 1,2,3-triazole derivatives. Table 3 summarizes a comparison of the results obtained for the as-prepared Cu/WO_3 catalytic system with those reported in the literature. Cu(II)–PBS–HPMO and SiO_2–NHC-Cu(I) catalysts (Table 3, entries 1 and 2) were prepared through multistep processes for a

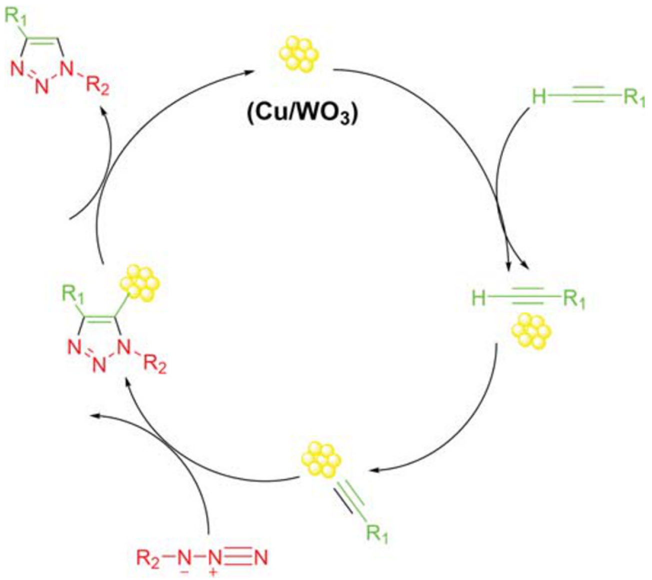


FIGURE 4 Tentative mechanism of azide–alkyne cycloaddition reaction

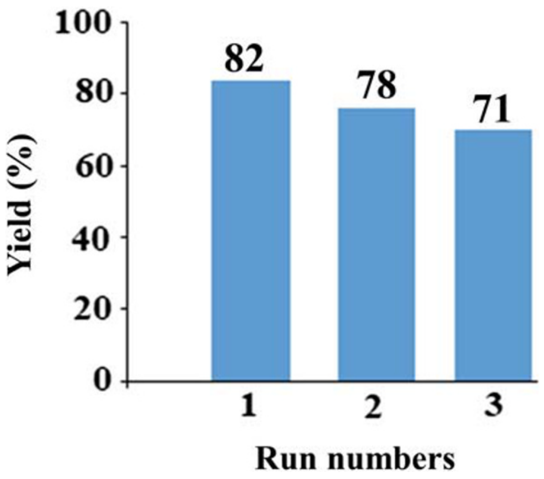


FIGURE 5 Reusability evaluations of the Cu/WO_3 catalyst in the azide–alkyne cycloaddition reaction

long time. In the presence of Cu/Al_2O_3, the reaction was carried out under severe conditions such as ball milling (Table 3, entry 3). PS–C22–CuI and Cu(I)–zeolite catalysts led to the formation of the desired product at a low temperature, but the reaction took a long time (Table 3, entries 4 and 5). Moreover, the fabrication of Cu@βCD–PEG–mesoGO and CuNPs/Mag silica are so hard and expensive (Table 3, entries 6 and 7). In summary, according to Table 3, each reported protocol has its own merits as well as demerits. However, from the point of cost, easy and straightforward catalyst preparation, operational simplicity, and relatively low reaction time, the Cu/WO_3 catalyst synthesized in this study is superior and easily adaptable.

To evaluate the heterogeneity of the synthesized catalyst, a leaching experiment was carried out. In this regard, after the reaction was run for 4 h, the catalyst was separated by centrifugation and then the reaction was continued for 12 h. It was found that no product was obtained, suggesting the high stability of catalyst during the reaction process. Moreover, inductively coupled plasma (ICP) analysis was used to measure the copper content in the recycled Cu/WO_3 catalyst, which showed that the copper contents of the fresh catalyst and the second-run reused catalyst were 12.3 and 16.0 w/w%, respectively. This result indicated that very low leaching amount of copper occurred in the reaction mixture.

4 | CONCLUSIONS

The Cu NPs immobilized on WO_3 support were fabricated and characterized using XRD, SEM–EDX, and TEM. The synthesized novel material was found to be an effective catalyst in the click reaction of alkynes with various benzyl halides in the presence of sodium azide. This reaction could be considered an important

TABLE 3 Comparison of Cu/WO_3 heterogeneous catalyst toward click reaction with other reported catalysts

Entry	Catalyst	Reaction condition	Time (h)	Yield (%)	Ref.
1	Cu(II)–PBS[a]–HPMO	100°C, H_2O	3.5	96	[23]
2	SiO_2–NHC–Cu(I)	80°C, H_2O	6	98	[24]
3	Cu/Al_2O_3	Ball milling, Rt	1	92	[25]
4	PS–C22–CuI[b]	Rt, H_2O	15	99	[26]
5	Cu(I)–zeolite	20°C, MeOH	15	80	[27]
6	Cu@βCD–PEG–mesoGO[c]	25°C, H_2O	1	90	[28]
7	CuNPs/Mag silica	70°C, H_2O	1	98	[29]
8	Cu/WO_3	90°C, H_2O	12	82	This work

[a] Porphyrin-bridged silsesquioxane.

[b] Polystyrene resin-supported copper(I) iodide–cryptand-22 complex.

[c] Copper-supported β-cyclodextrin-functionalized PEGylated mesoporous silica nanoparticle–graphene oxide hybrid.

approach to synthesize 1,2,3-triazole derivatives, which are very substantial intermediates in organic and medicinal chemistry. To optimize the reaction conditions, various parameters such as catalyst amount, solvents, reaction time, and temperature were studied. Finally, the recyclability of the fresh and reused catalysts along with a leaching experiment was also checked for the click reaction.

ACKNOWLEDGMENTS

The authors thank the University of Maragheh for financial support of this work.

AUTHOR CONTRIBUTIONS

Mojtaba Amini: Supervision. **Elham Hajipour:** Conceptualization; methodology. **Keun Hwa Chae:** Software.

AUTHOR CONTRIBUTION

Mojtaba Amini supervised the study, Elham Hajipour conceptualized the study and devised methodology, Ali Akbari wrote the original draft, and Keun Hwa Chae used software to generate data.

FUNDING INFORMATION

The authors thank the University of Maragheh for financial support of this work.

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How to cite this article: Amini M, Hajipour E, Akbari A, Hwa Chae K. Immobilization of copper nanoparticles on WO_3 with enhanced catalytic activity for the synthesis of 1,2,3-triazoles. *Appl Organomet Chem*. 2020;e5959. https://doi.org/10.1002/aoc.5959