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ORIGINAL PAPER

Copper nanoparticles doped in wrinkled fibrous nanosilica as an efficient catalyst for the synthesis of 1,4-disubstituted 1,2,3-triazole in aqueous solution

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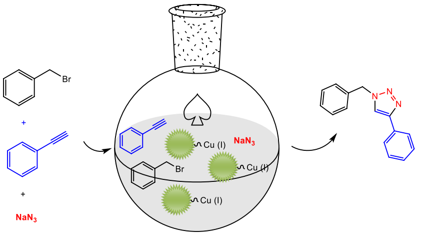
Abstract

A one-pot, highly efficient procedure was reported for the synthesis of 1,4-disubstituted 1,2,3-triazoles in water as a green solvent using Cu@KCC-1-NH-CS_2 as a novel nanoreactor. Different methods such as Fourier transform infrared spectroscopy, field emission scanning electron microscopy, transmission electron microscopy, energy-dispersive X-ray spectroscopy, and adsorption/desorption analysis (BET equation) have been used for the characterization of engineered nanocatalyst. High surface area (133 $m^2\ g^{-1}$) and porous structure of the nanocatalyst, excellent catalytic performance in ecofriendly and green solvent with very short reaction times (5–20 min), easy work up, no use of toxic solvents, and suitable reusability of the catalyst are advantages of this presented work.

Graphic abstract



Keywords Aqueous solution process · Heterogeneous catalyst · Advanced nanomaterials · Dendritic fibrous nanosilica · Nanoreactor

Introduction

1,2,3-Triazoles are one of the vital heterocyclic compounds which extensively applied in various fields such as photographic materials, corrosion inhibition, agro chemicals, pharmaceuticals, dyes, and so on [1, 2]. Due to their biological properties, applications in drug delivery, magnetic resonance imaging, and bimolecular sensors are demanded [3, 4].

Synthesis of substituted 1,2,3-triazoles from the reaction of azide derivatives and terminal alkynes with Huisgen 1,3-dipolar cycloaddition are widely studied by click reactions. In ‘click chemistry’, triazoles synthesis has some advantages such as optimal atom economy, high yield and selectivity, using of water as a solvent, and mild reaction conditions. In common, copper catalysts have been used for the synthesis of 1,4-disubstituted 1,2,3-triazoles using cycloaddition reaction of azides and terminal alkynes. While Cu(I) catalysts provide a consistent way to the synthesis of 1,4-disubstituted 1,2,3-triazoles, but using Ru complex, the 1,5-disubstituted 1,2,3-triazole is a important product [5].

Various copper-containing catalysts such as $CuSO_4$ [6–8], CuI [9, 10], Cu_2O [11], $(PPh_3)_3CuBr$ [12], $CuFe_2O_4$

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nanoparticles, imidazole–Cu(II) [13], Cu–Fe bimetals [14], Cu/C [15], Cu/SiO_2 [16], and polymer-modified Cu/Cu_2O [17] have been used for the synthesis of 1,2,3-triazoles. In general, Cu^+ salt or reduction of Cu^{2+} can be applied as a homogeneous catalyst for the regioselective AAC (alkyne-azide cycloaddition) reaction [18–21].

In recent years, heterogeneous and porous catalysts are highly demanded for various reactions because of their available active sites, high surface area, and simple isolation from the mixture, and the reusability of the nanocatalyst [22, 23]. KAUST Catalysis Center (KCC-1) as a novel wrinkled and dendritic fibrous nanosilica has exceptional physico-chemical properties based on its microsphere structure [24–26]. These types of dendritic nanosilica have accessible pores and channels for rendering of molecules and biomolecules in comparison with other conventional silica mesoporous materials such as Mobil Composition of Matter (MCM), Santa Barbara Amorphous (SBA), and Korea Advanced Institute of Science and Technology (KIT). Moreover, available active sites, high surface area, effective interactions with other organic molecules and facile functionalization and grafting can be performed, because of dendritic and wrinkled structure of KCC-1 [27]. According to this feature, KCC-1 is an exceptional and excellent candidate as a nanocatalyst for various organic synthesis.

Moreover, one of the most significant research area concerning advanced functional materials is surface modification of mesoporous materials [28]. In recent years, fibrous nanosilica (KCC-1), with the low density, broad pore size distribution, high surface area (typically > 700 m^2/g), large pore sizes, ease of surface functionalization, stability and low toxicity with good biocompatibility have been reported by Polshettiwar et al. [29–33]. This nanosilica with dendritic fibrous morphology showed unusual activities as a heterogeneous catalyst [33].

In the present work, a one-pot and highly efficient synthesis of 1,4-disubstituted 1,2,3-triazoles was reported using Cu@KCC-1-NH-CS_2 as a green nanocatalyst in H_2O and 50 °C with high yields and very short reaction times in comparison with other previous studies (Scheme 1). It is vital to point out that, after completion of the reaction the applied nanocatalyst can be separated with simple filtration and used again with minor reduction in its catalytic activity.

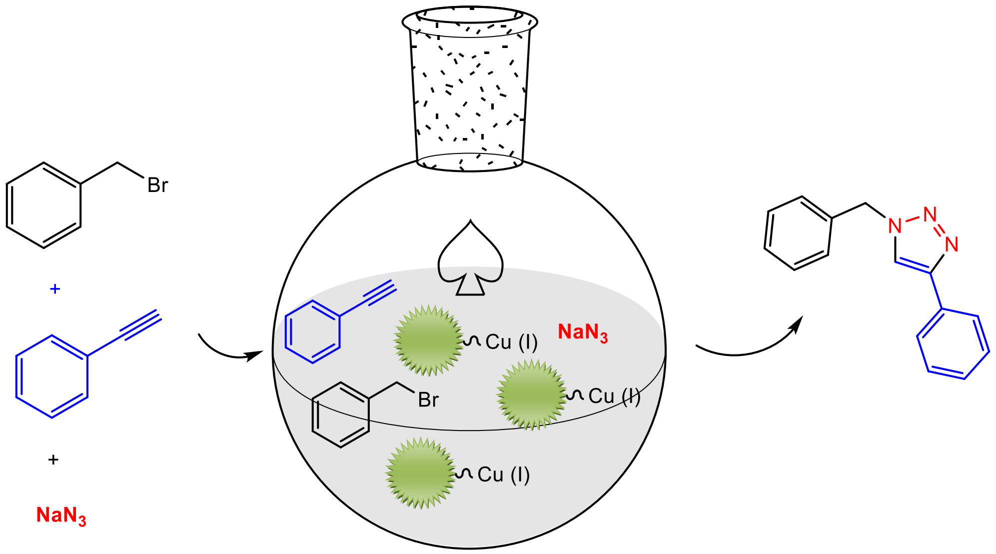
Results and discussion

Characterization of Cu@KCC-1-NH-CS_2

Different methods such as Fourier transform infrared spectroscopy (FT-IR), field emission scanning electron microscopy (FE-SEM), transmission electron microscopy (TEM), energy-dispersive X-ray spectroscopy, adsorption/desorption analysis (Brunauer–Emmett–Teller (BET) equation), and energy-dispersive X-ray spectroscopy (EDX) have been used for the characterization of engineered nanocatalyst. In FT-IR analysis, OH stretching vibration, Si–O symmetric and Si–O asymmetric stretching of Si–OH, and functional groups of Si–O bending, were appeared at, 3424, 1095, 797, and 466 cm^{-1}, respectively (Fig. S1, see supporting information). In addition, two peaks of S–H at 2171 and 2099 cm^{-1} are related to the aggregation of mercapto groups.

The morphology of prepared nanocatalyst was characterized by FE-SEM and TEM analyses. As can be seen in Fig. S2 (see supporting information), wrinkled and fibrous

Scheme 1



morphology and uniform spheres of KCC-1 were confirmed by these analyses.

In addition, EDX data established the existence of nitrogen, carbon, sulfurs, silica, and Cu. The EDX analysis proved that the presence of O, Si, C, N, and Cu characteristic peaks. Furthermore, the presence of copper bands in EDX analysis approves the effective presence of copper(II) complex on the KCC-1-NH-CS_2 channels (Fig. S3, see supporting information).

The porous structure of KCC-1, KCC-1-NH_2, and KCC-1-NH-CS_2 was evaluated by Barrett, Joyner, Halenda (BJH) and BET analyses. From these analyses, specific surface area, mean pore diameter and pore volume of the nano adsorbent were obtained and summarized in Table S1. According to Fig. S4 (see supporting information), pore volumes changed from 1.52 to 1.1 and 0.41 cm^3/g for KCC-1, KCC-1-npr-NH_2, and KCC-1-NH-CS_2. Furthermore, specific surface area was found as 133.0, 367, 617 m^2/g for KCC-1-NH-CS_2, KCC-1-NH_2, and KCC-1, respectively. In addition, the mean pore diameter distribution of KCC-1-NH_2-CS_2, KCC-1-NH_2, and KCC-1 was achieved as 12.69, 11.9, and 9.9, respectively (Table S1, see supporting information).

General synthesis procedure

At first, to find the optimum reaction conditions for the synthesis of 1,2,3-triazole derivatives, 1 mmol of benzyl bromide (**1**), 1 mmol phenylacetylene (**2**), and 1.1 mmol sodium azide (**3**) was selected as a model reaction (Scheme 2).

The catalytic behavior of Cu@KCC-1-NH-CS_2 was compared with other dendritic fibrous nanosilica such as KCC-1, KCC-1-NH_2, and KCC-1-NH-CS_2. No product has been formed when the model reaction was done in water solution (H_2O) and 50 °C in the nonexistence of the nanocatalyst after 140 min. In addition, using KCC-1-NH_2, KCC-1-NH-CS_2 as a catalyst in H_2O and 50 °C after 140 min (Table S2, entries 2, 3; see supporting information) no significant products were detected. It was found that the reaction in the presence of 0.01 g Cu@KCC-1-NH-CS_2 at 50 °C and after 5 min lead to products with a 100% yield (Table S2, entry 4). However, the excellent yield of 1,4-disubstituted 1,2,3-triazoles was observed in the presence of 0.01 g of Cu@KCC-1-NH-CS_2 and sodium ascorbate as a catalyst in 5 min. Finally, after several screening tests by the mentioned nanocatalysts with different functional groups such as amine and dithiocarbamate, Cu@KCC-1-NH-CS_2 (0.01 g) containing Cu^{2+} as a Lewis acid shows shorter reaction times and a higher yields for the model reaction in H_2O and 50 °C (Table S2, entry 5).

In continuously, the EtOH and EtOH/H_2O were selected as a solvent for the click reaction of sodium azide, organic bromides, and terminal alkynes in the presence of Cu@KCC-1-NH-CS_2 and sodium ascorbate as a catalyst. The good results were obtained in the presence of EtOH and EtOH/H_2O (yield: 100%, time: 5 min). Surprisingly, the similar results were obtained in aqueous solution (H_2O) as a green and suitable solvent. Hence, the water which gives the best results in this reaction in very short reaction times and high yields was used as a candidate solvent for this reaction in the existence of Cu@KCC-1-NH-CS_2 as an innovative nanoreactor.

In addition, these reactions were performed between 5 and 20 min. Moreover, TLC results established that no by-product was formed during the reaction. In addition, the reaction completed with electron-withdrawing and electron-donating groups which lead to giving various 1,4-disubstituted 1,2,3-triazole derivatives in 95–100% yields. According to 3, the proposed strategy is universal and comprises a variety of functional groups (Table 1).

Because of the high surface are (133 m^2/g) of the designed nanocatalyst (Cu@KCC-NH-CS_2) and suitable functional groups, the reactants easily can be adsorbed on the surface and channels of the nanocatalyst. Therefore, the interactions between reactants increase significantly. Therefore, each nanometer pore can act as a separate nanoreactor which the reactants quickly transferred to these cavities and changed to the appropriate products in high yields and short reaction times. It should be noted that in the present work, each nanoreactor with Cu^+ species provides the complete reaction requirements for the click reaction. Figure 1 shows this phenomenon.

Scheme 2

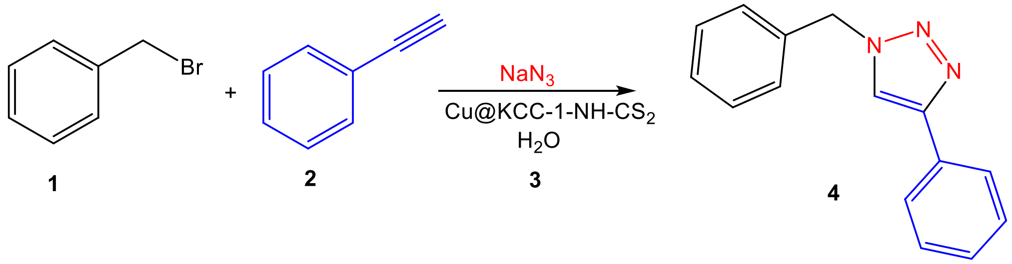
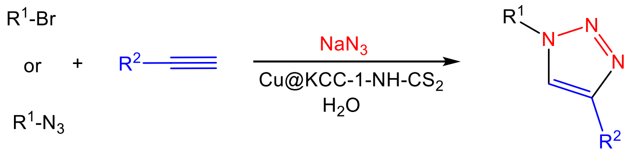
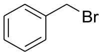
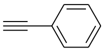
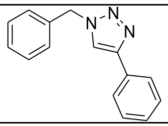
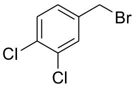
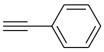
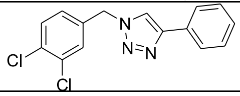
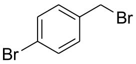
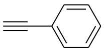
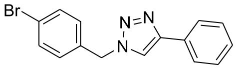
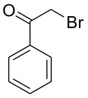
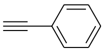
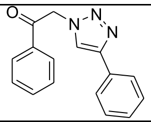
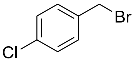
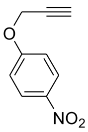
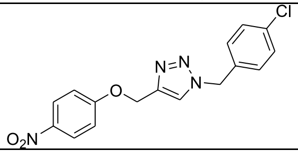
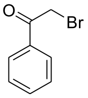
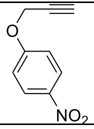
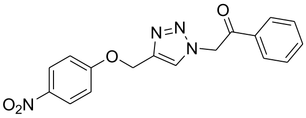
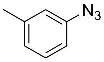
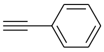
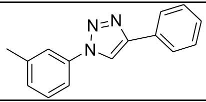
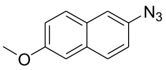
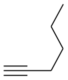
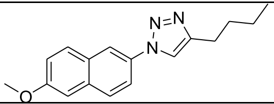
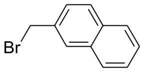
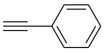
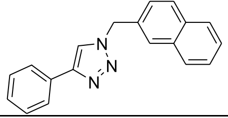


Table 1 Comparison of the yield and reaction time of 1,2,3-triazole products



1 2 3 4

Entry	Aryl halide	Alkyne	Product	Time / min	Yield /%
4a				5	98
4b				5	100
4c				5	99
4d				7	98
4e				5	95
4f				8	95
4g				10	96
4h				20	92
4i				15	93

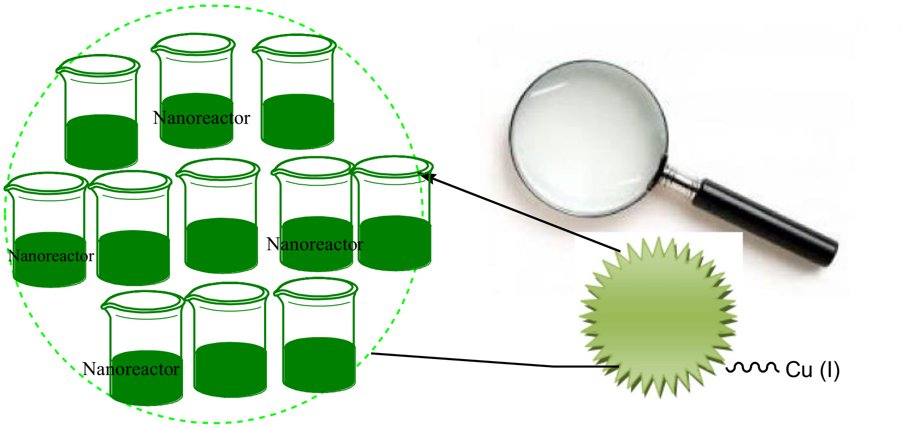


Fig. 1 The role of each pore of Cu@KCC-1-NH-CS$_2$ as individual nonreactor

The catalytically active Cu(I) species which supported on the functionalized KCC-1 can be produced from the Cu(II) salts in the presence of sodium ascorbate as a reducing agent. It is necessary to mention that Cu@KCC-1-NH-CS_2 with high surface area, high pore volume and porosity cause to achieve the product in shorter reaction time and with higher yields in comparison with other reports [6, 35–40].

To complete our discussion, obtained results of the present work were compared with other methods which reported in the previous reports and summarized in the Table 2. According to Table 2, in the previous reports, some toxic solvents have been used to extract the product from the reaction mixture. Due to the high yield of the reaction, the pure product can be easily obtained by the addition of low amount of ethanol to the mixture of reaction and the nanocatalyst separated by simple filtration. Therefore, there is no need to use toxic solvents for the extraction of products from the reaction mixture. These are the critical properties of the current report. In addition, short reaction time is another property of the applied nanocatalyst to speed up the reaction.

Reusability of the catalyst

For commercial applications, the reusability of nanocatalysts is so essential. Hence, the recyclability of the nanocatalyst was evaluated for the one-pot synthesis of 1,4-disubstituted 1,2,3-triazoles, using the reaction of sodium azide, an organic bromide, and a terminal alkyne in aqueous solution (water). In this method, filtration was used for the separation of the nanocatalyst from the reaction mixture and then washed with water and ethanol. Therefore, the separated nanocatalyst was air-dried carefully before being used in subsequent runs. As can be seen in Fig. S5 (see supporting information), it was established that under mild reaction conditions, this green nanocatalyst indicated recyclable behavior 5 times with minor reduction in its catalytic activity. It is important to point out that, this catalyst loosing activity after five times. In recent years, numerous porous solids with ordered structure-silica-based mesoporous materials (MCM, SBA, KCC-1, and others) have been synthesized through surfactant directed self-assembly and applied as a stable support to anchor various functional groups and active species to catalyze various organic transformations. Usually, a continuous decrease in yields was observed after each runs in all cases because of the particle aggregation, leaching of active species and, decrease in active surface area [41].

Conclusion

In summary, the results indicated that Cu@KCC-1-NH-CS_2 is an efficient and green nanoreactor to the synthesis of 1,4-disubstituted 1,2,3-triazole from the one-pot reaction of bromo derivatives, alkynes, and azide in water at 50 °C. Outstanding catalytic behavior in aqueous solution with high surface area and porous structure, very short reaction times, excellent yields, easy work up, and no use of toxic solvents, suitable reusability of the catalyst, using of ecofriendly and green solvent are advantages of this work. Excellent catalytic performance in aqueous media with high surface area (133 $m^2\ g^{-1}$) and porous structure, very short reaction times (5–20 min), easy work up, and no use of toxic solvents, reusability of the catalyst, using of ecofriendly and green solvent are advantages of this work. In addition, under mild reaction conditions, this green nanocatalyst indicated recyclable behavior five times with minor reduction in its catalytic activity.

Experimental

Cetyltrimethylammonium bromide (as a surfactant) and employed solvents during synthesis procedures were purchased from Merck, Germany. Tetraethyl orthosilicate (TEOS) and (3-aminopropyl)triethoxysilane (APTES) and some of the solvents used in the present study (DMSO, toluene, and hexanol) were bought from Sigma-Aldrich. In this study, deionized water was prepared in our laboratory.

Table 2 Organic figure of merit for the copper-catalyzed click reaction

Entry	Catalyst	Condition	Time/temp	Yield/%	References
1	PS-C22-CuI (0.6 mol%)	H_2O	15 h/r.t.	99	[35]
2	Copper(II) sulfate supported on alumina (Cu/Al_2O_3, 10 mol%)	Solvent-free	60 min/r.t.	96	[6]
3	Cu/SiO_2 composite	Water, MW	10 min/70 °C	92	[36]
4	Copper(I) (10 mol%) and amino acid ionic liquid (AAIL) in [BMIM]BF_4	[BMIM]BF_4	10 h/60 °C	88	[37]
5	Reduced graphene oxide-supported Cu-Cu_2O (5 mol%)	Water	2.0 h/r.t.	96	[38]
6	Cellulose supported poly(hydroxamic acid) copper complex (0.05 mol%)	Sodium ascorbate	2.5 h/70 °C	96	[39]
7	[CuBr(PPh_3)$_3$] (0.5 mol%)	Neat	7–24 h/H_2O, r.t.	99	[40]
8	Cu@KCC-1-NH-CS_2, sodium ascorbate (0.17 mol%)	Water	2 min/50 °C	100	This work

Materials and reagents no further purified and they were used as purchased.

Philip Harris C4954718 was used to the measurement of the melting points. FT-IR was recorded by Thermo Nicolet Nexus 670 FT-IR. Bruker Avance AQS 300 MHz spectrometer at 300 and 75.5 MHz were used to the recording of ^{1}H and ^{13}C NMR spectra, respectively. $CDCl_3$ as solvent relative to TMS as the internal standard was used to the recognition of chemical shifts. Elemental analyses were performed using a Leco Analyzer 932. TLC (thin-layer chromatography) on silica plates (Merck's Co.) was used to the monitoring of the reaction progress.

Synthesis of KCC-1-NH-CS_2

The synthesis of KCC-1 and functionalization of its surface with aminopropyl groups to generate KCC-NH_2 is according to the method offered by our research group [34]. For functionalization of KCC-1-NH_2 by dithiocarbamate, 90 mg KCC-1-NH_2, 100 mg CS_2, and 1% of Et_3N in 20 cm^3 acetonitrile were mixed and the obtained solution was stirred overnight. After that, the solvent of mixture was eliminated by the evaporator, and the obtained powder was washed by water and acetonitrile solution for the removal of Et_3N.

Synthesis of Cu@KCC-1-NH-CS_2

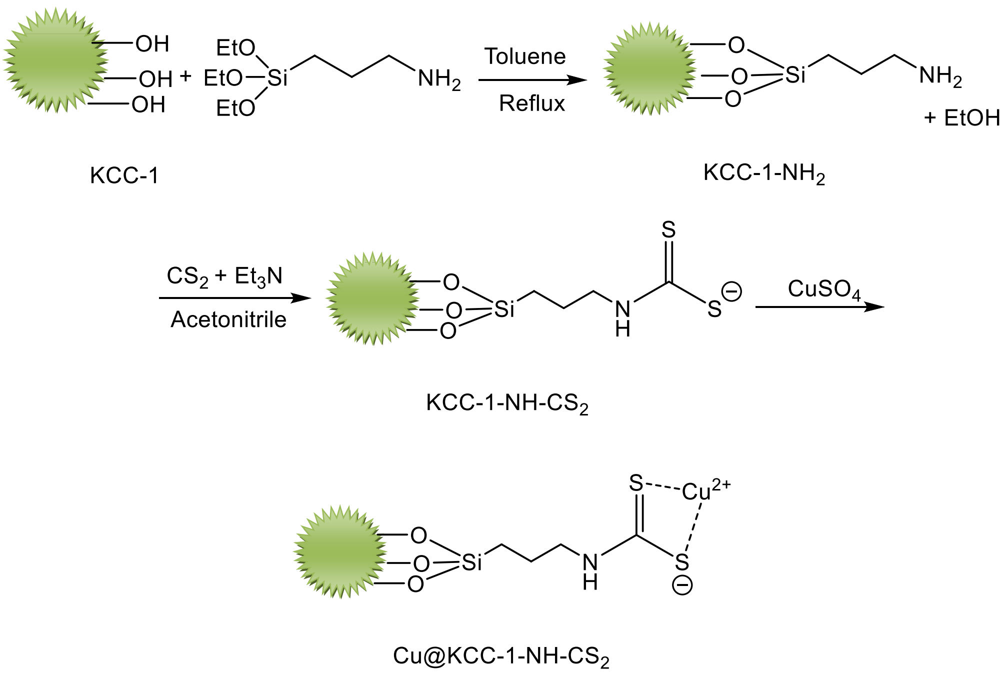
A mixture of copper(II) sulfate solution (1 M, 25 cm^3 in deionized water) and KCC-1-NH-CS_2 was stirred at 60 °C for 15 h and the modified nanocatalyst was successively separated by centrifuge and washed with 10 cm^3 water. Finally, obtained catalyst dried in vacuum at room temperature gave the Cu@KCC-1-NH-CS_2. Scheme 3 represented the synthesis procedure.

Synthesis procedure of 1-benzyl-4-phenyl-1*H*-1,2,3-triazole derivatives: an universal approach

For the synthesis of 1,2,3-triazole derivatives, 1.1 mmol of sodium azide, 1.0 mmol of benzyl bromide, and 1.0 mmol of terminal alkynes were included in a 25.0 cm^3 round-bottomed flask containing 0.01 g Cu@KCC-1-NH-CS_2 as a catalyst and 5 cm^3 H_2O as a solvent. Then, the mixture was heated at 50 °C. After completing the reaction which monitored with TLC (EtOAc, *n*-hexane 1:3), 5 cm^3 of EtOH was included in the mixture and heated at 50 °C. After that, the nanocatalyst was separated by filtration and the crystallization of the product started immediately after cooling the solution. At the end, the separated nanocatalyst was washed using acetone/water (1:1) solution and reused for the next runs without significant reduction in its catalytic activity.

It is important to point out that in two cases (**4g** and **4h**) direct reaction of organic azides and alkynes was performed.

Scheme 3



1-Benzyl-4-phenyl-1*H*-1,2,3-triazole (4a) M.p.: 110–112 °C (Ref. [42] 112–115 °C).

1-(3,4-Dichlorobenzyl)-4-phenyl-1*H*-1,2,3-triazole (4b) M.p.: 137–139 °C (Ref. [42] 137–141 °C).

1-(4-Bromobenzyl)-4-phenyl-1*H*-1,2,3-triazole (4c) M.p.: 135–137 °C (Ref. [42] 136–139 °C).

1-Phenyl-2-(4-phenyl-1,2,3-triazol-1-yl)ethanone (4d) M.p.: 143–146 °C (Ref. [42] 144–146 °C).

1-(4-Chlorobenzyl)-4-(4-nitrophenoxymethyl)-1*H*-1,2,3-triazole (4e, $C_{16}H_{13}ClN_4O_3$) ^{1}H NMR (400 MHz, $CDCl_3$): $\delta = 8.34$ (s, 1H), 8.20 (d, $J = 9.2$ Hz, 4H), 7.25 (d, $J = 9.2$ Hz, 4H), 7.45 (d, $J = 8.4$ Hz, 4H), 7.35 (d, $J = 8.4$ Hz, 4H), 5.63 (s, 2H), 5.31 (s, 2H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): $\delta = 163.7$, 142.5, 141.5, 135.3, 133.3, 130.4, 129.2, 126.3, 125.5, 115.7, 62.3, 52.5 ppm.

2-[4-(4-Nitrophenoxymethyl)-1,2,3-triazol-1-yl]-1-phenylethanone (4f, $C_{17}H_{14}N_4O_4$) ^{1}H NMR (400 MHz, DMSO-d_6): $\delta = 5.39$ (s, 2H), 6.23 (s, 2H), 7.60–8.09 (m, 5H), 7.30 (d, $J = 9.2$ Hz, 4H), 8.24 (d, 4H, $J = 9.2$ Hz), 8.26 (s, 1H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): $\delta = 192.5$, 163.8, 142.1, 141.5, 134.7, 134.5, 129.4, 128.6, 127.1, 126.3, 115.8, 62.3, 56.4 ppm.

4-Phenyl-1-(*m*-tolyl)-1*H*-1,2,3-triazole (4g, $C_{15}H_{13}N_3$) ^{1}H NMR (400 MHz, $CDCl_3$): $\delta = 2.49$ (s, 3H), 7.30 (s, 1H), 7.37–7.41 (m, 3H), 7.58–7.60 (m, 1H), 7.65 (s, 1H), 7.92–7.95 (m, 2H), 8.20 (s, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): $\delta = 21.45$, 117.62, 117.65, 121.23, 125.86, 128.39, 128.92, 129.54, 129.56, 130.32, 137.03, 140.04, 148.31 ppm.

4-Butyl-1-(6-methoxynaphthalen-2-yl)-1*H*-1,2,3-triazole (4h, $C_{17}H_{19}N_3O$) ^{1}H NMR (400 MHz, $CDCl_3$): $\delta = 0.99$ (t, $J = 7.6$ Hz, 3H), 1.47 (sex, $J = 7.6$ Hz, 2H), 1.77 (quin, $J = 7.6$ Hz, 2H), 2.85 (t, $J = 7.6$ Hz, 2H), 3.97 (s, 3H), 7.21 (d, $J = 2.4$ Hz, 1H), 7.25 (dd, $J = 2.4$, 9.0 Hz, 1H), 7.81–7.85 (m, 2H), 7.87–7.90 (m, 2H), 8.08 (d, $J = 1.6$ Hz, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): $\delta = 13.87$, 22.36, 25.42, 31.56, 55.42, 105.80, 118.29, 118.96, 119.59, 120.35, 128.46, 128.57, 129.65, 134.11, 134.96, 149.16, 158.38 ppm.

1-(Naphthalen-2-ylmethyl)-4-phenyl-1*H*-1,2,3-triazole (4i, $C_{19}H_{15}N_3$) ^{1}H NMR (400 MHz, $CDCl_3$): $\delta = 6.02$ (2H, s), 7.25–7.26 (2H, m), 7.33–7.34 (2H, m), 7.47–7.53 (4H, m), 7.72–7.74 (2H, m), 7.90 (2H, d, $J = 5.2$ Hz), 8.00 (1H, d, $J = 5.2$ Hz) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): $\delta = 52.8$, 119.8, 123.3, 125.7, 126.0, 126.8, 127.8, 128.3, 128.5, 129.1, 129.3, 130.2, 130.5, 130.9, 131.6, 134.3 ppm.

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