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Solvothermal Synthesis of Cuprous Oxide Microsphere and its Application as Catalyst for Synthesis of β-Hydroxy Triazole

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In this article, the solvothermal synthesis of spherical cuprous oxide microsphere in glycerol-water (1:1) using copper(II) acetate as copper precursor is reported. The synthesized cuprous oxide microparticles was characterized by powder X-ray diffraction (PXRD) and scanning electron microscopic technique (SEM). The material was successfully used as a catalyst for the synthesis of β-hydroxy-1,2,3-triazole in water *via* a one-pot multicomponent reaction.

Keywords: Cuprous oxide, Copper(II) acetate, 1,2,3-Triazole, One-pot multicomponent reaction.

INTRODUCTION

The cuprous oxide (Cu_2O) is a p-type semiconductor with a bandgap of 1.9-2.2 eV and absorbs light in the visible range of the electromagnetic spectrum. It is mainly used as an active material in water splitting [1], solar energy conversion [2], gas sensing [3], hydrogen evolution [4], catalysis [5], antimicrobial activity [6], *etc*. Due to the economical availability of copper precursors and low toxicity, the synthesis of Cu-based materials as a functional material has been pursued actively throughout the world.

The Cu_2O-based materials are mostly synthesized by the reduction of Cu(II) to Cu(I) by making use of reducing agents in an aqueous medium or other solvent. The most commonly used reducing agents are ascorbic acid [7], hydroxylamine [8], hydrazine [9], polyhydroxy alcohols [10], glucose [11], *etc*. Several researchers reported various methods for the synthesis of Cu_2O of different morphology by playing with reaction parameters. Chen *et al.* [12] reported the hydrothermal methods for the synthesis of truncated octahedral and quasi-spherical Cu_2O microcrystals from $CuCl_2$. Bagherzadeh *et al.* [13] synthesized Cu_2O with cubic, octahedra, spherical and truncated rhombic dodecahedral morphology by varying the concentration of the reducing agent. Aguilar & Rosas [14] synthesized Cu_2O with cubic, octahedral, truncated octahedral and cubic truncated morphologies by reducing the aqueous solution of cupric chloride with sodium borohydride.

After the invention of click reaction by Sharpless *et al.* [15] and Meldal *et al*. [16], Cu(I)-based materials have been used as an efficient catalyst for the synthesis of regioselective synthesis of 1,4-disubstituted 1,2,3-triazole *via* cycloaddition of organic azide and terminal acetylene. Since then, Cu-based azide-alkyne cycloaddition (CuAAC) has been applied for the synthesis of pharmaceuticals [17], agrochemicals [18], dendrimers [19], polymers [20] and many other functional materials. Synthesis of β-hydroxy-triazole is mainly achieved *via in situ* azidolysis of epoxides in the presence of terminal alkynes and different copper-based catalysts in water. One-pot multicomponent click reaction for the synthesis of triazole from epoxide has been reported using heterogeneous catalysts such as CuI [21], Cu(I)@phosphorated SiO_2 [22], copper ferrite nanoparticles [23], Cu/Cu_2O nanoparticles [24] and Cu(I) coordination complex [25].

All the above Cu-based material shows excellent catalytic properties towards azide-alkyne cycloaddition reaction, however, for the synthesis of all these copper-based catalysts there is a requirement for the multiple reaction constituents. The synthesis of catalysts with similar efficiency with simple procedures with fewer numbers of reaction constituents is greener and more sustainable. In this study, we synthesized Cu_2O micro-

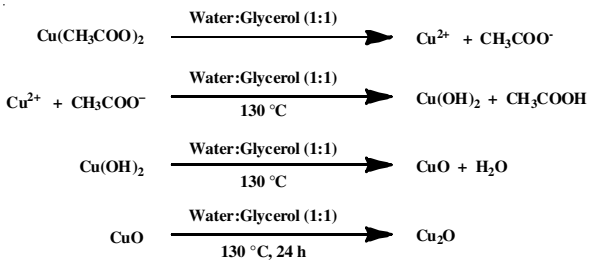
sphere by simple solvothermal methods by heating copper(II) acetate solution in a water-glycerol (1:1) mixture. Further, synthesized Cu_2O microsphere was used as an active catalyst for the synthesis of β-hydroxy-1,2,3-triazole *via* one-pot multi-component click reaction at room temperature in an aqueous medium with good to excellent yields.

EXPERIMENTAL

Copper(II) acetate, glycerol and the common solvents (hexane & ethyl acetate) were purchased from Loba Chemie, India. The organic precursors and deuterated solvents were obtained from Sigma-Aldrich and used without further purification. Distilled water was used as a solvent to carry out the organic reactions.

Characterization: The powder X-ray diffraction (PXRD) of Cu_2O microsphere was recorded using Bruker D8 Advance instruments with Cu*K*α (λ = 1.5406 Å) radiation in the reflection mode. The SEM image of Cu_2O microscope was recorded by FEI Quanta 200 scanning electron microscope. The β-hydroxy-triazole compounds were characterized by recording ^{1}H NMR and ^{13}C NMR using Bruker Avance 400 MHz NMR instrument.

Synthesis of Cu_2O micropheres: Copper(II) acetate (363 mg, 2 mmol) dissolved in 50 mL distilled water was added to 50 mL glycerol. The reaction mixture was heated at 130 ºC in an oil bath for 24 h with continuous stirring. After completion of the reaction, the reaction mixture was allowed to cool down to room temperature and Cu_2O microspheres were isolated by centrifugation, washed with water twice and dried in open air (**Scheme-I**).



Scheme-I: Proposed reaction mechanism for the synthesis of Cu_2O microsphere

General procedure for the synthesis of β-hydroxy-1,2,3-triazoles (2a-j): Epoxide (1 mmol, 1 equiv.), NaN_3 (1.5 mmol, 1.5 equiv.), terminal acetylene (1.2 mmol, 1.5 equiv.) and 10 mg of Cu_2O were added to a 50 mL round bottom flask containing 2 mL water. The reaction mixture was stirred at around on a magnetic stirrer for 24 h at room temperature. The progress of the reaction was monitored by visual inspection of the formation of solid product or by thin layer chromatography (TLC). After the completion of reaction, the product was extracted in ethyl acetate (3 × 5 mL). The solution was evaporated to dryness and purified by column chromatography (**Scheme-II**).

2-Phenyl-2-(4-phenyl-1*H*-1,2,3-triazol-1-yl)ethan-1-ol (2a): White solid, ^{1}H NMR ($CDCl_3$, 400 MHz) δ ppm: 7.74-7.72 (d, 2H, *J* = 8 Hz), 7.69 (s, 1H), 7.39-7.35 (m, 5H), 7.31-7.29 (m, 1H), 7.28-7.24 (m, 2H), 5.68-5.65 (m, 1H), 4.65-4.59 (m, 1H), 4.23-4.19 (m, 1H), 3.69 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ ppm: 147.80, 136.17, 130.30, 129.28, 129.12, 128.95, 128.41, 127.27, 125.79, 120.70, 67.45, 65.22.

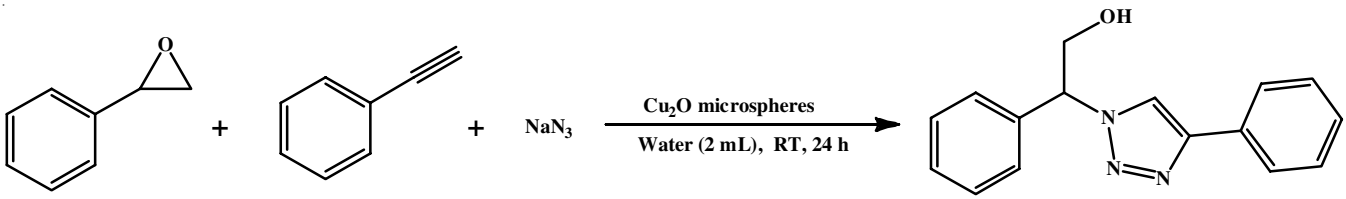
2-Phenyl-2-(4-*p*-tolyl-1*H*-1,2,3-triazol-1-yl)ethanol (2b): White solid, ^{1}H NMR ($CDCl_3$, 400 MHz) δ ppm: 7.67-7.65 (d, *J* = 8 Hz, 3H), 7.41-7.37 (m, 3H), 7.26-7.24 (m, 2H), 7.21-7.19 (d, *J* = 8 Hz, 2H), 5.67-5.64 (dd, *J* = 8Hz, 4Hz, 1H), 4.66-4.59 (m, 1H), 4.24-4.18 (m, 1H), 3.40 (br, 1H) 2.36 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ ppm: 148.02, 138.33, 136.23, 129.65, 129.33, 129.17, 127.54, 127.26, 125.75, 120.35, 67.38, 65.39, 21.43.

2-(4-Phenyl-1*H*-1,2,3-triazol-1-yl)cyclohexan-1-ol (2c): White solid, ^{1}H NMR (400 MHz, $CDCl_3$) δ ppm: 7.71 (s, 1H), 7.63-7.61 (d, *J* = 8 Hz, 2H), 7.34-7.28 (m, 3H), 4.14-4.11 (m, 3H), 2.24-2.16 (m, 2H), 2.02-1.96 (m, 1H), 1.90-1.88 (m, 2H), 1.52-1.39 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ ppm: 146.66, 130.35, 128.85, 128.11, 125.55, 119.90, 72.67, 67.39, 33.87, 31.64, 24.92, 24.19.

2-(4-(*p*-Tolyl)-1*H*-1,2,3-triazol-1-yl)cyclohexan-1-ol (2d): White solid, ^{1}H NMR (400 MHz, $CDCl_3$) δ ppm: 7.70 (s, 1H), 7.58-7.56 (d, *J* = 8 Hz, 2H), 7.18-7.16 (d, *J* = 8 Hz, 2H), 4.15-4.12 (m, 1H), 4.09-4.06 (m, 1H), 3.70 (br, 1H), 2.36 (s, 3H), 2.21 (m, 2H), 1.98-1.88 (m, 3H), 1.51-1.44 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ ppm: 147.33, 138.03, 129.58, 127.69, 125.61, 119.25, 72.75, 67.06, 33.76, 31.71, 24.89, 24.14, 21.44.

1-Phenoxy-3-(4-phenyl-1*H*-1,2,3-triazol-1-yl)propan-2-ol (2e): White solid, ^{1}H NMR (400 MHz, $CDCl_3$) δ ppm: 7.83 (s, 1H), 7.69-7.67 (d, *J* = 8 Hz, 2H), 7.36-7.32 (t, *J* = 16 Hz, 2H), 7.29-7.24 (m, 3H), 6.97-6.93 (t, *J* = 16 Hz, 1H), 6.89-6.87 (d, J = 8 Hz, 2H), 4.71-4.65 (m, 1H), 4.53-4.49 (m, 2H), 4.02-3.95 (m, 2H), 3.81 (br, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ ppm: 158.22, 147.72, 130.33, 129.78, 128.97, 128.34, 125.76, 121.71, 121.51, 114,68, 69.03, 68.96, 53.28.

1-(Allyloxy)-3-(4-phenyl-1*H*-1,2,3-triazol-1-yl)propan-2-ol (2f): White solid, ^{1}H NMR (400 MHz, $CDCl_3$) δ ppm: 7.88 (s, 1H), 7.78-7.76 (d, *J* = 8 Hz, 2H), 7.40-7.38 (t, 2H), 7.33-7.32 (t, 1H), 5.92-5.87 (m, 1H), 5.30-5.26 (d, 1H), 5.23-5.20 (d, 1H), 4.61-4.58 (d, 1H), 4.46-4.41 (m, 1H), 4.28 (s, 1H), 4.02 (s, 2H), 3.54-3.52 (m, 1H), 3.44-3.40 (m, 1H), 3.29 (br, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ ppm: 147.71, 134.12, 130.53, 128.95, 128.27, 125.76, 121.34, 117.96, 72.59, 71.03, 69.39, 53.17.



Scheme-II: Synthesis of β-hydroxy-1,2,3-triazole in the presence of Cu_2O microspheres

1-(Allyloxy)-3-(4-(*p*-tolyl)-1*H*-1,2,3-triazol-1-yl)-propan-2-ol (2g): White solid, ^{1}H NMR (400 MHz, $CDCl_3$) δ ppm: 7.82 (s, 1H), 7.63-7.61 (d, *J* = 8 Hz, 2H), 7.19-7.17 (d, *J* = 8 Hz, 2H),5.92-5.86 (m, 1H), 5.30-5.26 (d, *J* = 16 Hz, 1H), 5.22-5.20 (d, *J* = 8 Hz, 1H), 4.60-4.56 (d, *J* = 16 Hz, 1H), 4.43-4.38 (m, 1H), 4.28 (s, 1H), 4.02 (s, 2H), 3.66 (s, 1H), 3.54-3.50 (m, 1H), 3.46-3.44 (m, 1H), 2.36 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ ppm: 147.64, 138.05, 134.17, 129.57, 127.63, 125.61, 121.03, 117.86, 72.55, 71.11, 69.32, 53.26, 21.40.

1-(4-Phenyl-1*H*-1,2,3-triazol-1-yl)hexan-2-ol (2h): White solid, ^{1}H NMR (400 MHz, $CDCl_3$) δ ppm: 7.84 (s, 1H), 7.76-7.74 (d, *J* = 8 Hz, 2H), 7.39-7.31 (m, 3H), 4.51-4.48 (d, *J* = 8 Hz, 1H), 4.28-4.22 (m, 1H), 4.13 (s, 1H), 2.92 (s, 1H), 1.54-1.25 (m, 6H), 0.94-0.90 (t, *J* = 8 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ ppm: 147.58, 130.51, 128.96, 128.27, 125.74, 121.22, 70.72, 34.26, 27.70, 22.70, 14.13.

1-(4-(*p*-Tolyl)-1*H*-1,2,3-triazol-1-yl)hexan-2-ol (2i): White solid, ^{1}H NMR (400 MHz, $CDCl_3$) δ ppm: 7.78 (s, 1H), 7.63-7.61 (d, *J* = 8 Hz, 2H), 7.20-7.18 (d, *J* = 8 Hz, 2H), 4.49-4.46 (d, 1H), 4.25-4.20 (m, 1H), 4.12 (s, 1H), 3.07 (s, 1H), 2.36 (s, 3H), 1.68-1.37 (m, 6H), 0.93-0.992 (t, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ ppm: 147.16, 138.09, 129.62, 127.67, 125.64, 120.90, 70.67, 56.33, 34.27, 27.71, 22.70, 21.42, 14.12.

1-(*tert*-Butoxy)-3-(4-phenyl-1*H*-1,2,3-triazol-1-yl)propan-2-ol (2j): White solid, ^{1}H NMR (400 MHz, $CDCl_3$) δ ppm: 7.91 (s, 1H), 7.80-7.78 (d, *J* = 8 Hz, 2H), 7.41-7.32 (m, 3H), 4.60-4.57 (d, *J* = 8 Hz, 1H), 4.44-4.41 (m, 1H), 4.16 (s, 1H) 3.46-3.44 (m, 1H), 3.34-3.31 (m, 1H), 1.18 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ ppm: 147.73, 130.70, 128.95, 128.22, 125.80, 121.25, 73.85, 69.75, 62.75, 53.24, 27.99.

RESULTS AND DISCUSSION

In this synthetic procedure, the usage of hazardous chemicals such as water-soluble polymeric compound (*e.g.* PVP) and surfactants was avoided. However, the synthesis of Cu_2O microsphere was performed following a reported procedure with small modification [10]. In the optimized method, copper(II) acetate was used as a Cu^{2+} precursor, where it was heated at 130 ºC in water-glycerol (1:1) as solvent for 24 h with continuous stirring. As the reaction proceeded with time, a slow formation of black particles was observed. This may be due to ion exchange reaction between water and acetate ions, resulting in the formation of acetic acid and cupric hydroxide. As the temperature was high, immediately after the formation of cupric hydroxide it decomposed to cupric oxide which was observed as black particles. Slowly with continuous heating, the black colour particle changes to a yellow colour which is due to the formation of Cu_2O microsphere (**Scheme-I**). At high temperatures, glycerol acts as a reducing agent and reduces Cu(II) to Cu(I).

Powder X-ray studies: The PXRD of the synthesized Cu_2O microspheres shows the intensities of the dynamic reflections from (110), (111), (200), (220), (311) and (222) planes are proportional to the corresponding Bragg intensities (Fig. 1a). The obtained XRD results are consistent with the values reported in the literature and the standard JCPDS No. 05-0667 [26]. Scanning electron microscopic image (SEM) showed that the particles are spherical with an approximate diameter of 5-8 µm (Fig. 1b).

Catalytic studies: The reaction of styrene oxide, sodium azide and phenylacetylene was chosen as a model reaction. In the absence of catalyst no desired products were obtained after 48 h of stirring at room temperature in an aqueous medium (Table-1). Then we focused on the use of synthesized Cu_2O microspheres as catalyst to achieve the target. The reaction was carried out with different mole percentages of the catalyst at room temperature for 24 h. The results show that the reaction was effective with 10 mol% of Cu_2O microspheres where 95% of the desired product was isolated after extraction and purification *via* column chromatography.

Under the same reaction condition, the click reaction was extended to various epoxides and good to excellent yields were obtained (Table-2). Different regioselectivity was observed in different epoxides. In case of styrene oxide, the nucleophilic attack by azide was at more substituted carbon (Table-2, **2a**

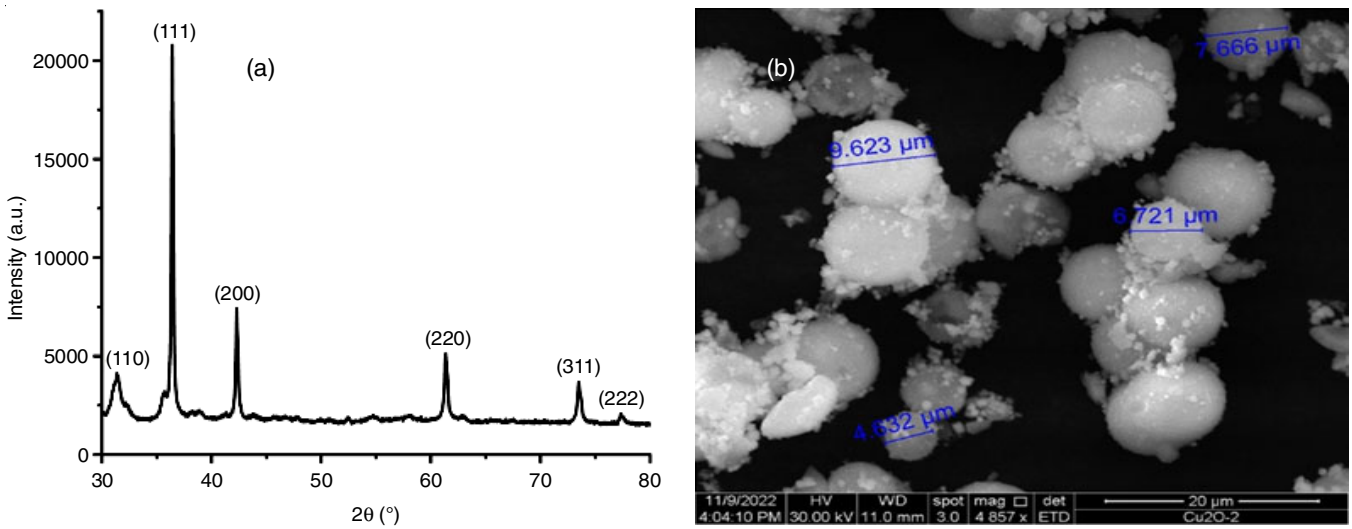


Fig. 1. (a) Powder X-ray diffraction of Cu_2O microspheres and (b) SEM image of Cu_2O microspheres

TABLE-1
OPTIMIZATION OF REACTION CONDITIONS FOR THE SYNTHESIS OF β-HYDROXY-1,2,3-TRIAZOLE

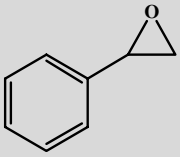
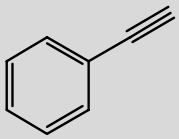
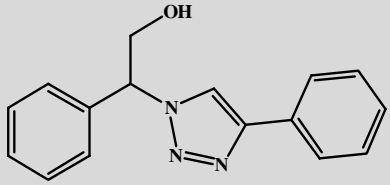
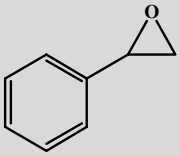
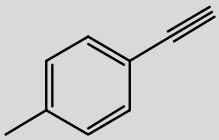
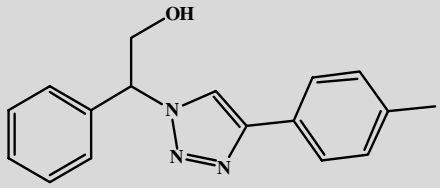
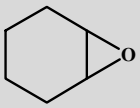
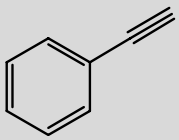
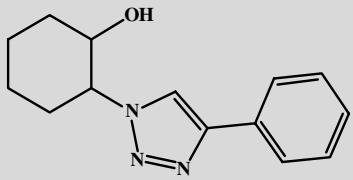
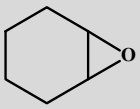
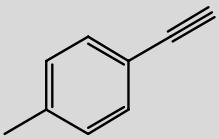
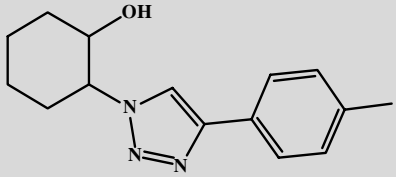
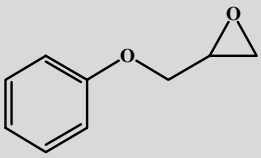
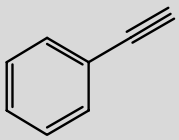
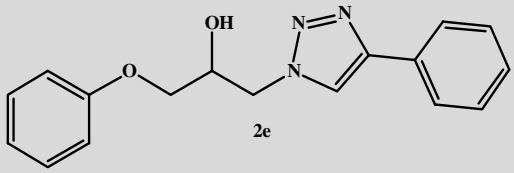
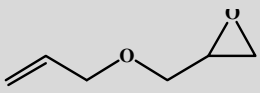
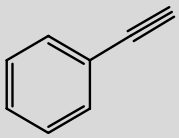
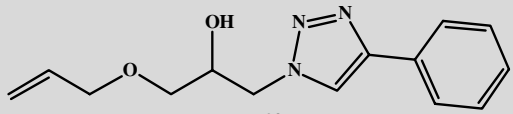
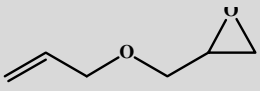
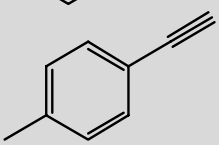
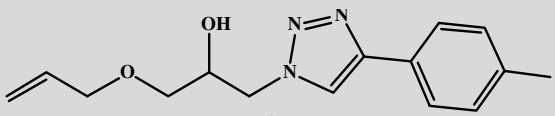
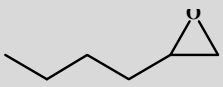
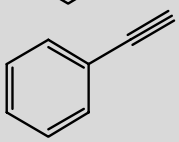
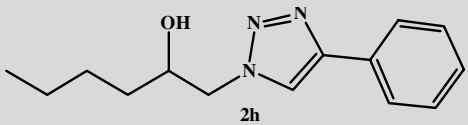
Entry	Cu_2O microspheres (mol%)	Isolated yield (%)
1	–	0
2	1	17
3	5	59
4	10	95

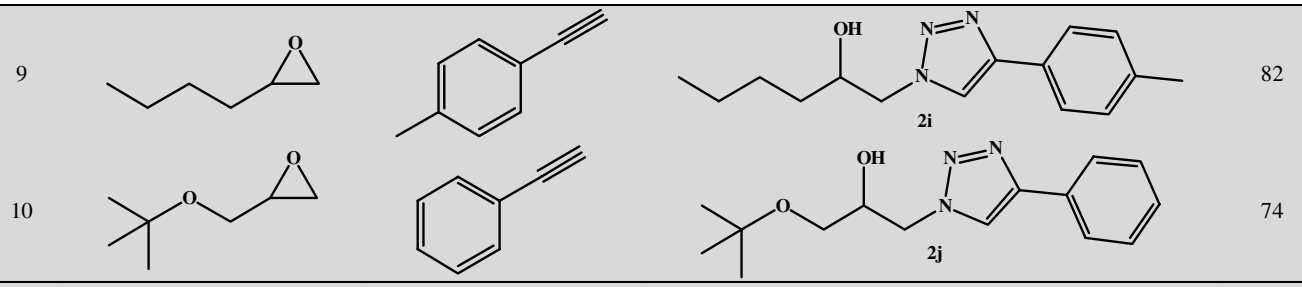
and **2b**) whereas in the case of aliphatic epoxide, the preferential attack on less hindered carbon (Table-2, **2e-2j**) was observed.

Conclusion

In this study, we synthesized Cu_2O microspheres *via* simple solvothermal methods by making use of green solvents and reagents. The catalytic activity of Cu_2O microspheres was explored towards the synthesis of β-hydroxy-1,2,3-triazole

TABLE-2
SYNTHESIS OF 1,4-DISUBSTITUTED β-HYDROXY-1,2,3-TRIAZOLE

Entry	Epoxide	Acetylene	Product	Yield (%)[a]
1			 **2a**	95
2			 **2b**	93
3			 **2c**	81
4			 **2d**	80
5			 **2e**	87
6			 **2f**	79
7			 **2g**	75
8			 **2h**	80



Reaction condition: Epoxide (1 mmol), sodium azide (1.2 mmol), acetylene (1.2 mmol), Cu_2O microspheres (10 mol%), water (2 mL), room temperature, 24 h; [a]Isolated yield

via a one-pot multicomponent click reaction in water at room temperature.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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