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$CuFe_2O_4$ nanoparticles catalyze the reaction of alkynes and nitrones for the synthesis of 2-azetidinones†

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$CuFe_2O_4$ nanoparticles acted as a highly efficient heterogeneous catalyst in the reaction of alkynes and nitrones (Kinugasa reaction) for the synthesis of various 2-azetidinones. In all cases, the reactions proceeded conveniently under mild conditions with good-to-excellent yields and with a wide range of functional-group tolerance. The catalyst could be separated readily using an external magnet.

Introduction

Besides the extensive medical applications with high importance of β-lactam antibiotics,[1,2] β-lactams (2-azetidinones) also have other biological activities.[3–5] Due to biological application of 2-azetidinones and their utility as synthetic intermediates,[6,7] several methods for the preparation of 2-azetidinones have been presented.[8–13] The applicable methodologies for the synthesis of 2-azetidinones include the Kinugasa reaction,[14–16] Staudinger reaction[17–20] ([2+2] ketene-imine cycloaddition) and metallo-ester enolate-imine cyclocondensation.[21]

The Kinugasa reaction is the direct synthesis of β-lactams from copper acetylides and nitrones.[22] It provides some advantages, including optimal atom economy and employment of readily accessible starting materials.[23]

In 1972, Kinugasa and Hashimoto reported, for the first time, a simple one-pot reaction between terminal acetylene and a nitrone to produce a β-lactam in the presence of copper(I) and pyridine.[24] The early product of the 1,3-dipolar cycloaddition reaction is a metallated isoxazoline intermediate, which can subsequently undergo rearrangement into the β-lactam ring.[25,26]

Basak and coworkers reported the synthesis of functionalized 2-azetidinones by the Kinugasa reaction in the presence of $CuSO_4{\cdot}5H_2O$ or $Cu(OAc)_2{\cdot}H_2O$ and sodium ascorbate as a reducing agent.[27] Tang and coworkers found that $Cu(ClO_4)_2{\cdot}6H_2O$ without a reducing agent, instead of sensitive Cu(I) salts, could catalyze the Kinugasa reaction very well in air.[28] Also, $CuBr_2$ and $Cu(OTf)_2$ have been used as catalysts for this reaction by other research teams.[29]

Recently, magnetic nanoparticles (MNPs) have been used widely in various biological applications, such as magnetic resonance imaging and in organic reactions, because of low toxicity, easy preparation without the need for a filtration step, large surface area, reusability by magnetic separation, and increased efficiency of catalytic activity.[30,31]

Copper ferrite ($CuFe_2O_4$) nanoparticles are inexpensive, air-stable, magnetically separable, and economical catalysts. Scientists have reported several applications in organic reactions: reduction of nitrophenol; C–O cross-coupling of phenols with aryl halides; synthesis of aryl azides and 1,4-diaryl-1,2,3-triazoles; synthesis of diaryl/aryl alkyl sulfides *via* cross-coupling; the azide–alkyne 'Click' reaction; C–N cross-coupling; sugar diacylation; A^3 coupling; Biginelli condensation using $CuFe_2O_4$ as a heterogeneous catalyst.[32–36]

Application of magnetic $CuFe_2O_4$ nanoparticles in the Kinugasa reaction has not been reported. Herein, for the first time, the efficient catalytic activity of magnetic copper ferrite ($CuFe_2O_4$) nanoparticles for the synthesis of 2-azetidinones by the Kinugasa reaction is reported.

Results and discussion

Nitrones were synthesized by the reaction of nitro compounds and aldehydes in the presence of zinc powder and acetic acid according to a reported procedure.[37] $CuFe_2O_4$ nanoparticles were prepared readily using $Fe(NO_3)_3{\cdot}9H_2O$, $Cu(NO_3)_2{\cdot}2H_2O$ and NaOH with a modified method according to the literature.[38] The powder X-ray diffraction pattern of the sample is shown in Fig. 1. The spectrum is similar to that reported previously,[38] and is consistent with standard JCPDS file number 34-0425. Furthermore, the $CuFe_2O_4$ nanoparticles were characterized by transmission electron microscopy (TEM) (Fig. 1). TEM demonstrated

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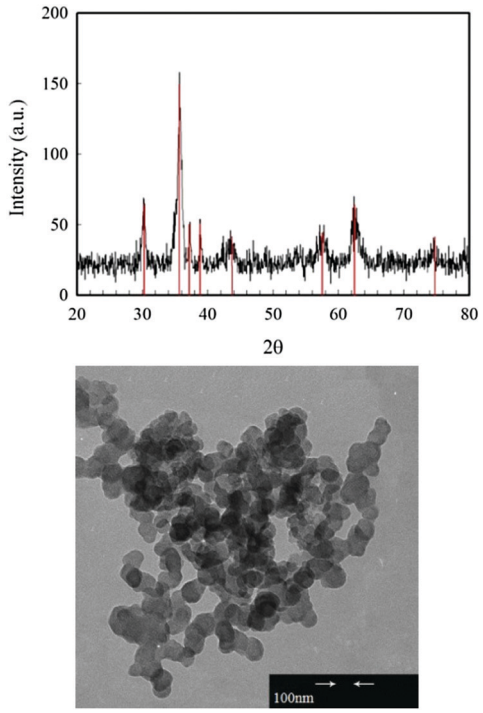


Fig. 1 X-ray diffraction pattern (up) and TEM images (down) of $CuFe_2O_4$ nanoparticles.

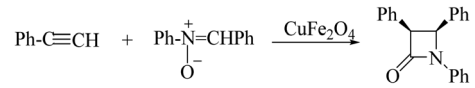
the spherical shape and nanometer size of synthesized $CuFe_2O_4$ nanoparticles.

Then, $CuFe_2O_4$ nanoparticles were employed as a heterogeneous catalyst to catalyze the reaction of phenylacetylene and nitrone **2a** in DMF in the presence of K_2CO_3 as a model reaction. The target product 2-azetidinone **3a** was obtained in 43% yield in 21 h (Table 1, entry 2), whereas 2-azetidinone **3a** was not obtained in the absence of $CuFe_2O_4$ nanoparticles. The latter were separated readily by a magnetic field. Optimization of solvents (DMF, DMSO, CH_3CN, THF and toluene) led us to find the highest yield when using CH_3CN (entry 4). Among the tested bases (K_2CO_3, Et_3N, pyridine and Et_2NH) in CH_3CN as the solvent, the yield of product **3a** in the presence of Et_2NH was the highest (entry 9). These results suggested that the yield increased when the base was changed to an organic base. To evaluate and optimize the amount of catalyst, 10–20 mol% of $CuFe_2O_4$ nanoparticles was examined with Et_2NH as the base (entry 9, 11, 12). Among them, 15 mol% of the catalyst seemed to be the most efficient amount for this reaction. The reaction at a lower temperature (0 °C) or higher temperature (50 °C) resulted in a decreased yield of **3a**. In consideration of these results, 15 mol% of $CuFe_2O_4$ nanoparticles in the presence of Et_2NH at room temperature in CH_3CN was chosen as the best condition for further work (entry 11).

We wished to establish the scope of this catalyst. Having the optimized reaction condition in hand (Table 1, entry 11), various substituents on alkynes and nitrones were examined (Scheme 1, Table 2).

Various aromatic alkynes and nitrones bearing electron-donating and electron-withdrawing substituents underwent

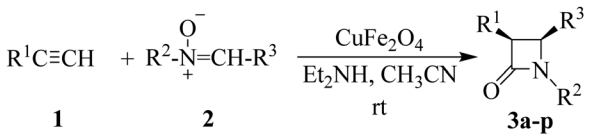
Table 1 Optimization of reaction conditions in the Kinugasa reaction using $CuFe_2O_4$ nanoparticles



1a **2a** **3a**

Entry	Amount of $CuFe_2O_4$ (mol%)	Solvent	Base	Time (h)	Yield of **3a** (%)
1	—	DMF	K_2CO_3	26	—
2	10	DMF	K_2CO_3	21	43
3	10	DMSO	K_2CO_3	24	14
4	10	CH_3CN	K_2CO_3	18	51
5	10	THF	K_2CO_3	20	48
6	10	Toluene	K_2CO_3	29	—
7	10	Toluene	Et_3N	23	—
8	10	CH_3CN	Et_3N	18	61
9	10	CH_3CN	Et_2NH	18	68
10	10	CH_3CN	Pyridine	18	53
11	15	CH_3CN	Et_2NH	18	77
12	20	CH_3CN	Et_2NH	18	71
13	15	CH_3CN	Et_2NH	24	73
14	15	CH_3CN	Et_2NH	18	57[a]
15	15	CH_3CN	Et_2NH	18	44[b]

[a] Reaction was carried out at 0 °C to rt. [b] Reaction was carried out at 50 °C.



1 **2** **3a-p**

Scheme 1 Synthesis of 2-azetidinones **3a–p** using $CuFe_2O_4$.

this $CuFe_2O_4$ nanoparticles-catalyzed reaction to obtain substituted-2-azetidinones in good-to-excellent yields. Also, aliphatic derivatives of the starting materials afforded the corresponding 2-azetidinones in appropriate yields. The results shown in Table 2 indicated that all reactions proceeded under mild conditions at room temperature to gain stereoselective *cis* 2-azetidinones. Purification of products was undertaken by simple crystallization from ethyl acetate.

The structure of 2-azetidinones **3a–n** was deduced from their spectral data (^{1}H nuclear magnetic resonance (NMR), ^{13}C NMR and infrared (IR)). ^{1}H NMR spectra showed two characteristic peaks at about δ 3.5 ppm and 5.20 ppm for H-3 and H-4 protons, respectively. The coupling constants of H-3 and H-4 protons were $J \geq 5.7$ Hz, thereby confirming the *cis* stereochemistry of 2-azetidinones **3a–n** ($J_{3,4} > 4.0$ Hz for the *cis* stereoisomer and $J_{3,4} \leq 3.0$ Hz for the *trans* stereoisomer).[46,47] The signals in ^{13}C NMR spectra at around δ 161.0 ppm were related to the carbonyl group of the 2-azetidinone ring. Also, the carbonyl groups were seen as sharp and characteristic peaks at $\sim$1750 cm^{-1} in the IR spectra.

After completion of the reaction, the nanomagnetic catalyst was separated by an external magnet (Fig. 2), washed with distilled water and EtOAc, and then dried. Further characterization by TEM showed no change in the surface morphology of the used catalyst (Fig. 2). The reusability of the catalyst was also

Table 2 Synthesis of 2-azetidinones **3a–p** using $CuFe_2O_4$

Entry	R^1	R^2	R^3	Product	Isolated yield (%)	M.p. °C (Lit. m.p. °C)
1	Ph	Ph	Ph	**3a**	77	177–179 (172–175)[39]
2	Ph	Ph	2-Naphthyl	**3b**	75	159–161 (170)[40]
3	Ph	4-$MeOC_6H_4$	Ph	**3c**	72	139–140 (138–140)[41]
4	CH_2OH	4-ClC_6H_4	4-$MeOC_6H_4$	**3d**	75	114–116
5	Ph	Ph	4-$MeOC_6H_4$	**3e**	79	110–112 (106–107)[42]
6	CH_2OH	Ph	PhCH=CH	**3f**	63	141–143 (143–144)[16]
7	Ph	4-ClC_6H_4	Ph	**3g**	69	163–165 (160–161)[41]
8	CH_2OH	Ph	4-$MeOC_6H_4$	**3h**	58	104–106 (102)[43]
9	CH_2OH	Ph	Ph	**3i**	54	129–131 (128)[43]
10	CH_2OH	Ph	2-Furyl	**3j**	57	117–119 (122)[43]
11	CH_2Br	Ph	PhCH=CH	**3k**	51	187–189 (185–188)[44]
12	Ph	4-MeC_6H_4	4-MeC_6H_4	**3l**	73	197–199 (201–202)[45]
13	CH_2OH	4-ClC_6H_4	Ph	**3m**	77	172–175
14	Bbutyl	4-ClC_6H_4	Ph	**3n**	68	167–169
15	CH_2OH	Ph	4-ClC_6H_4	**3o**	61	151–153
16	Butyl	Ph	4-ClC_6H_4	**3p**	55	157–159

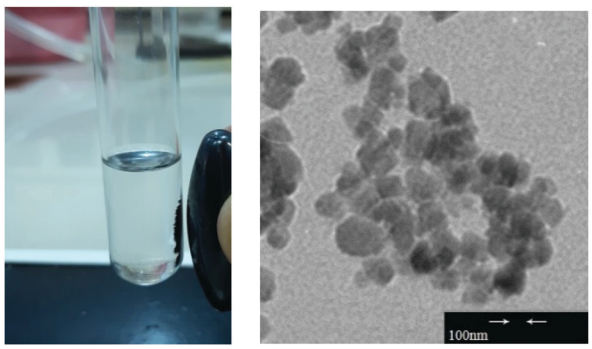


Fig. 2 Magnetic recoverability of $CuFe_2O_4$ nanocatalyst (left) and TEM image of the used $CuFe_2O_4$ nanoparticles (right).

Table 3 Recycling of $CuFe_2O_4$ (15 mol%) for the synthesis of **3a**

Number of cycles	1	2	3	4
Yield (%)	77	75	75	74

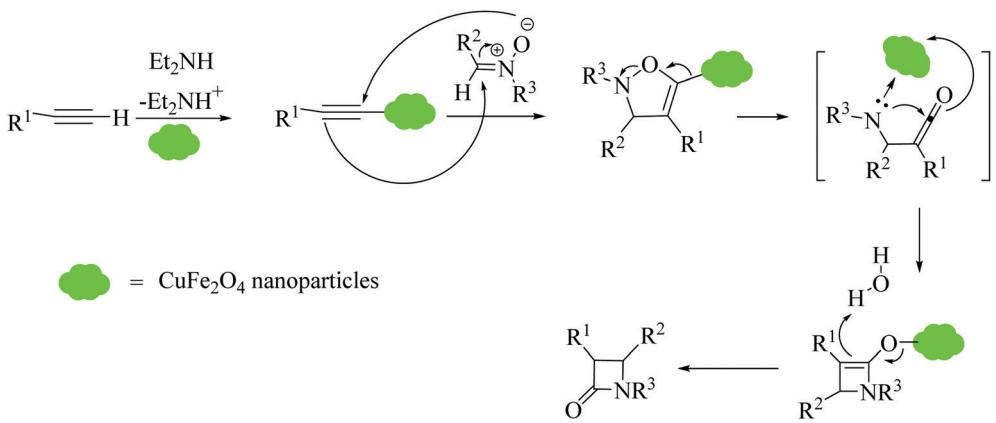
investigated for the synthesis of 2-azetidinone **3a** (Table 3). The catalyst was reusable for at least four cycles.

The scalability of the catalytic procedure for 15 mmol of alkynes for the synthesis of **3a** was examined: product **3a** was obtained at 79% yield.

A probable mechanistic pathway was suggested based on the work by Tang and coworkers[48] for the Kinugasa reaction using Cu(II), and Wang and coworkers[30] for $CuFe_2O_4$ nanoparticle-catalyzed synthesis of benzoxazoles, benzothiazoles and benzimidazoles. This mechanism describes opening of the isoxazoline ring, formation of an imidoketene intermediate in the presence of $CuFe_2O_4$ nanoparticles and, finally, formation of the 2-azetidinone ring in the presence of water (Scheme 2).

Conclusions

A reliable and practical procedure for the synthesis of 1,3,4-trisubstituted 2-azetidinones was developed. It involved application of $CuFe_2O_4$ nanoparticles as a magnetically separable catalyst in the reaction of terminal alkynes with nitrones. This was an active, non-toxic, and heterogeneous catalyst which was recyclable by rapid and easy separation using an external magnetic field. The reaction proceeded efficiently under mild conditions at room



Scheme 2 Probable mechanistic pathway.

temperature, and produced good-to-excellent yields of products after simple crystallization.

Experimental section

General considerations

All required chemicals were purchased from Merck, Fluka and Acros chemical companies. The melting point was determined on an Electrothermal 9200 apparatus and were uncorrected. IR spectra were measured on a Galaxy series FT-IR 5000 spectrometer. NMR spectra were recorded in $CDCl_3$ using a Bruker spectrometer (^{1}H NMR: 300 MHz; ^{13}C NMR: 75 MHz) and coupling constants were given in cycles per second (Hz). Elemental analyses were undertaken on a Vario EL III elemental analyzer. The morphology of nanoparticles was investigated by TEM on a 906e system (HT = 100 kV; Zeiss). The phase composition of nanoparticles was considered by X-ray diffraction using a D8 Advance system (Bruker). Thin-layer chromatography was carried out on silica gel 254 analytical sheets obtained from Fluka.

Catalyst preparation[38]

In a typical procedure, 3.6 g of $FeCl_3{\cdot}6H_2O$ and 3.5 g of $FeSO_4{\cdot}7H_2O$ were dissolved in 30 mL of deionized water. Then, 1 mL of concentrated HCl (37%) was added. The resulting clear solution was added dropwise to 300 mL of NaOH (1.5 M) solution with the reaction mixture stirred vigorously at 75 °C for 3 h. Then, the mixture was cooled to room temperature. The final products were collected and washed with deionized water and ethanol several times and dried in air at 80 °C.

General procedure for the synthesis of β-lactams 3a–p using $CuFe_2O_4$ nanocatalyst

Nitrones **2** (1 mmol) were added to a mixture of alkynes **l** (1 mmol), $CuFe_2O_4$ (15 mol%), and Et_2NH (1.1 mmol) in wet-CH_3CN (10 mL) at room temperature. The resulting mixture was stirred for 18 h. Then, the catalyst was separated from the reaction mixture with an external magnet. The reaction mixture was extracted, poured onto water (15 mL), extracted with chloroform (30 mL), washed with brine (30 mL), and dried over dry Na_2SO_4. The solvent was removed under reduced pressure and pure β-lactams **3a–p** were obtained after purification by crystallization from EtOAc. Characterization data for **3a–c** and **3e–l** have been reported.[16,39–45]

1-(4-Chlorophenyl)-3-(hydroxymethyl)-4-(4-methoxy-phenyl)-azetidin-2-one (3d). Colorless solid. M.p. 114–116 °C. IR (KBr) cm^{-1}: 1756 (CO, β-lactam), 3449 (OH); ^{1}H NMR (300 MHz) δ 1.42 (OH, br, 1H), 3.39–3.55 (H-3 and CH_2O, m, 3H), 3.65 (OMe, s, 3H), 5.13 (H-4, d, 1H, J = 5.9), 6.90–7.07 (ArH, m, 4H), 7.19–7.38 (ArH, m, 3H), 7.42 (ArH, d, 1H, J = 8.6); ^{13}C NMR (75 MHz) δ 55.9 (CH_2O), 56.7 (OMe), 58.7 (C-3), 60.7 (C-4), 115.2, 120.4, 125.8, 127.9, 131.8, 133.1, 134.8, 153.4 (aromatic carbons), 161.3 (CO, β-lactam); elemental anal. calcd for $C_{17}H_{16}ClNO_3$: C, 64.26; H, 5.08; N, 4.41. Found: C, 64.45; H, 5.24; N, 4.59.

1-(4-Chlorophenyl)-3-hydroxymethyl-4-phenyl-2-azetidinone (3m). Colorless solid. M.p: 172–175 °C. IR (KBr) cm^{-1}: 1749 (CO, β-lactam), 3310 (OH); ^{1}H NMR (300 MHz): δ 1.46 (OH, br, 1H), 3.50–3.68 (CH_2, H-3, m, 3H), 4.98 (H-4, d,1H, J = 5.3), 6.91–7.07 (ArH, m, 4H), 7.29–7.41 (ArH, m, 5H); ^{13}C NMR (75 MHz): δ 56.2 (CH_2), 59.5 (C-3), 62.0 (C-4), 116.8, 120.8, 124.1, 126.2, 129.4, 133.6, 134.6, 142.1 (aromatic carbons), 162.3 (CO, β-lactam); elemental anal. calcd for $C_{16}H_{14}ClNO_2$: C, 66.79; H, 4.90; N, 4.87. Found: C, 66.93; H, 5.11; N, 4.99.

3-Butyl-1-(4-chlorophenyl)-4-phenyl-2-azetidinone (3n). Colorless solid. M.p: 167–169 °C. IR (KBr) cm^{-1}: 1742 (CO, β-lactam); ^{1}H NMR (300 MHz): δ 0.77 (Me, t, 3H, J = 6.9), 1.10–1.19 (CH_2, m, 1H), 1.30–1.39 (CH_2, m, 3H), 1.54–1.73 (CH_2, m, 2H), 3.65 (H-3, dt, 1H, J = 5.7, 7.5), 5.22 (H-4, d, 1H, J = 5.7), 6.94–6.97 (ArH, d, 2H, J = 7.1), 7.06–7.11 (ArH, m, 1H), 7.23–7.36 (ArH, m, 6H); ^{13}C NMR (75 MHz): δ 13.7 (Me), 23.7, 26.2, 30.0 (CH_2), 53.3 (C-3), 59.7 (C-4), 118.4, 122.0, 125.9, 128.6, 130.2, 134.1, 135.6, 139.3 (aromatic carbons), 164.6 (CO, β-lactam); elemental anal. calcd for $C_{19}H_{20}ClNO$: C, 72.72; H, 6.42; N, 4.46. Found: C, 72.84; H, 6.59; N, 4.61.

4-(4-Chlorophenyl)-3-hydroxymethyl-1-phenyl-2-azetidinone (3o). Colorless solid. M.p: 151–153 °C. IR (KBr) cm^{-1}: 1743 (CO, β-lactam), 3346 (OH); ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.38 (OH, s, 1H), 3.48–3.70 (CH_2, H-3, m, 3H), 4.95 (H-4, d, 1H, J = 5.7), 6.92–7.07 (ArH, m, 3H), 7.23–7.45 (ArH, m, 4H), 7.52–7.63 (ArH, m, 2H); ^{13}C NMR (75 MHz): δ 54.3 (CH_2), 60.2 (C-3), 61.9 (C-4), 117.0, 121.0, 125.1, 126.6, 130.4, 132.4, 134.6, 140.8 (aromatic carbons), 162.6 (CO, β-lactam); elemental anal. calcd for $C_{16}H_{14}ClNO_2$: C, 66.79; H, 4.90; N, 4.87. Found: C, 66.95; H, 5.08; N, 5.01.

3-Butyl-4-(4-chlorophenyl)-1-phenyl-2-azetidinone (3p). Colorless solid. M.p: 157–159 °C. IR (KBr) cm^{-1}: 1759 (CO, β-lactam); ^{1}H NMR (300 MHz, $CDCl_3$): δ 0.78 (Me, t, 3H, J = 7.1), 1.04–1.08 (CH_2, m, 1H), 1.29–1.41 (CH_2, m, 3H), 1.64–1.74 (CH_2, m, 2H), 3.48 (H-3, dt, 1H, J = 6.0, 7.3), 5.04 (H-4, d,1H, J = 6.0), 7.07–7.17 (ArH, m, 2H), 7.28–7.48 (ArH, m, 7H); ^{13}C NMR (75 MHz): δ 14.1 (Me), 24.0, 27.6, 31.2 (CH_2), 54.0 (C-3), 61.7 (C-4), 118.6, 120.5, 124.5, 126.4, 129.2, 132.6, 137.1, 141.0 (aromatic carbons), 165.3 (CO, β-lactam); elemental anal. calcd for $C_{19}H_{20}ClNO$: C, 72.72; H, 6.42; N, 4.46. Found: C, 72.80; H, 6.62; N, 4.59.

Conflicts of interest

There are no conflicts to declare.

Acknowledgements

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