

## Synthesis of acetamides via oxidative C–C bond cleavage of ketones catalyzed by Cu-immobilized magnetic nanoparticles

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Akbar Heydari, Chemistry Department, Tarbiat Modares University, P.O. Box 14155-4838. Tehran, Iran. Email: heydar\_a@modares.ac.ir Copper supported on magnetite nanoparticles modified with environmentally friendly ligand tricine was devised for synthesis of acetamides via C–C oxidative cleavage of ketones with amines. The catalyst was characterized using different techniques, including Fourier transform infrared, X-ray diffraction, scannin electron microscopy, vibrating sample magnetometry, thermogravimetric analysis, and energy dispersive x-ray spectroscopy. The protocol showed relatively high yields of acetamide products. Furthermore, the magnetic recovery of the catalyst rendered the overall process fast and efficient. It was used in the reaction for six consecutive cycles with negligible loss of catalytic activity. This research is the first report of application of magnetic nanocatalysts for synthesis of acetamides from ketones of low activity through a C–C bond cleavage strategy.

### K E Y W O R D S

amide, C-C oxidative cleavage, copper-immobilized magnetic nanoparticles, ketone

## **1** | INTRODUCTION

Amides are an important class of organic compounds. They are widespread in natural products, pharmaceuticals, and other industrial materials,<sup>[1]</sup> and are extensively used as synthetic intermediates.<sup>[2,3]</sup> The antitumor, antibacterial, and anti-inflammatory characteristics of the amide functional group make it suitable for medicinal chemistry.<sup>[4–8]</sup> There are diverse methods for the synthesis of the amide functional group, such as amination of activated carboxylic acid derivatives,<sup>[9]</sup> direct acid and amine coupling,<sup>[10]</sup> Beckman rearrangement,<sup>[11]</sup> oxidative amidation,<sup>[12–15]</sup> and cross-coupling reactions.<sup>[16,17]</sup> However, these synthetic procedures suffer from high sensitivity to moisture, side reactions, not being economical, and harsh reaction conditions. To overcome these problems, a new protocol has emerged. Recently, oxidative C–C bond cleavage has been explored, <sup>[18–22]</sup> but this strategy is rarely reported for synthesis of amides. The pioneering work on synthesis of amides via oxidative C–C bond cleavage was the unexpected findings of Ballini *et al.* in 2003.<sup>[23]</sup> The concept explored further in the following years, but there are several drawbacks and limitations in the reported protocols such as narrow scope, high activity of ketone substrate, hazardous and expensive reagents, and use of radical initiators.<sup>[24–29]</sup> Moreover, in spite of the application of many homogeneous and heterogeneous catalysts designed for this process, almost all of them could not overcome the obstacle of inactivity of the simple ketones in the procedure.<sup>[24–27]</sup>

Recently a novel protocol for synthesis of amides from ketones taking advantage of the C–C bond cleavage strategy has been devised which avoids

This article is dedicated to memory of Naser Shabani.

the drawbacks of previous ones.<sup>[30]</sup> However, this protocol suffers from cumbersome separation of catalyst from the reaction mixture, lack of recyclability, and relatively high loadings of copper as the catalyst. To address these issues, using of superparamagnetic nanoparticles was proposed. Superparamagnetic nanoparticles are widely applicable in different areas of science and technology.<sup>[31]</sup> Amongst different types of magnetic nanoparticles, iron oxides and especially magnetite (Fe<sub>3</sub>O<sub>4</sub>) are of considerable interest due to their properties, such as high surface area, ecofriendliness, thermal stability, and very high magnetic remanence.<sup>[32]</sup> Despite these useful properties, bare magnetite nanoparticles are not active enough to catalvze the reactions of interest. Furthermore, unmodified magnetite nanoparticles are not sufficiently stable under different chemical conditions. Modification of the surface of magnetite nanoparticles is therefore necessary in chemical processes. Fortunately, this is straightforward because of there are many hydroxyl groups on the external surface of nanoparticles.<sup>[33]</sup> In industry, there is interest in simplifying the reagents, conditions, and catalysts required for a specific operation. We therefore chose one of the simplest ligands for surface modification with easy linkage to the support as well as high chelating power to reduce leaching of copper ions to the solvent. With this goal in mind, we modified the surface of magnetite nanoparticles with a novel environmentally favorable ligand, tricine. Tricine is an analog of glycine (from which it gets its name) that has applications in buffer solutions and electrophoresis.[34]

In our ongoing attempts to synthesize efficient environmentally benign catalysts, we devised a novel heterogeneous catalytic strategy for C–C bond cleavage synthesis of amides. The use of magnetite nanoparticles as a catalyst bed enables easy separation of catalyst from the reaction mixture with the aid of a permanent magnet. This is the first report of such an application in the C–C bond cleavage synthesis of amides.

## 2 | EXPERIMENTAL

## 2.1 | General remarks

All chemicals and solvents were purchased from commercial suppliers and used without further purification. Fourier transform infrared (FT-IR) spectra were obtained over the region 400–4,000 cm<sup>-1</sup> with a Nicolet IR100 FT-IR (US) with spectroscopic grade KBr (Germany). X-ray diffraction (XRD) patterns were obtained at room temperature with a qPhilips Xpert 1,710 diffractometer (Netherlands) with Co K $\alpha$   $(\lambda = 1.78897 \text{ Å})$ , 40 kV voltage, 40 mA current, and in the range  $10-90^{\circ}$  (2 $\theta$ ) with a scan speed of  $0.020^{\circ} \text{s}^{-1}$ . Scanning electron microscopy (SEM; Philips XL 30 and S-4160, Netherlands) was used to study the catalyst morphology and size. Magnetic saturation of the catalyst was investigated using vibrating а magnetometer/alternating gradient force magnetometer (MDK Co., Iran). Thermogravimetric analysis (TGA) was conducted using a thermal analyzer with a heating rate of 20°C min<sup>-1</sup> over a temperature range of 25-1,100°C under flowing nitrogen. The hydrodynamic diameter of the nanoparticles was measured using a Zetasizer Nano MAL1001767 (Malvern Instruments, Malvern, UK) by dynamic light scattering with the nanoparticles sonicated in water before the measurement. <sup>1</sup>H NMR spectra were recorded with a Bruker Avance (DRX 500 MHz, DRX 250 MHz, US) in pure deuterated dimethylsulfoxide (DMSO) solvent with tetramethylsilane as an internal standard.

## 2.2 | Preparation of copper-immobilized tricine-modified magnetite nanoparticles (MN-Tric-Cu)

# 2.2.1 | Preparation of tricine-modified magnetite nanoparticles (MN-Tric)

Magnetite nanoparticles were prepared according to the previously reported literature.<sup>[35]</sup> To a vigorously stirred solution of 10 mmol FeCl<sub>3</sub>.6H<sub>2</sub>O, 5 mmol of FeCl<sub>2</sub>.4H<sub>2</sub>O, 3 mmol of tricine in 40 ml of deionized water at 80°C, and 10 ml of aqueous ammonia solution (25% w/w) were added in small portions. After 6 hr, the resulting black solid was magnetically filtered and washed several times with water until it reached pH 7. Then the nanoparticles were washed twice with ethanol and dried at ambient temperature.

## 2.2.2 | Immobilization of copper acetate on the surface of tricine-modified magnetic nanoparticles (MN-Tric-Cu)

First, 1 g of as-prepared tricine-modified magnetic nanoparticles was dispersed in 40 ml of methanol and 3 mmol of copper acetate, then 1 mmol of KOH was added. The resulting mixture was stirred under reflux for 6 hr. Finally, the obtained nanoparticles were separated using a magnet and washed several times with water and acetone consecutively.

## 2.3 | General procedure for synthesis of amides

First, 1 mmol of amine and 1 mmol of acetone were charged in a round-bottomed flask and 4 ml of  $DMSO/CHCl_3$  (3/1) mixture was added. The mixture was heated at  $120^{\circ}C$  for 24 hr. Reaction progress was monitored using thin layer chromatography. The catalyst was removed with a permanent magnet and the reaction flask was flushed a few times with ethyl acetate and the product extracted with dilute (10%) HCl solution. The residual amine remained in the aqueous phase as ammonium salt and a solution of amide product in ethyl acetate was obtained. After the removal of the solvent under reduced pressure, the resultant amide product was obtained (details of amide characterization are provided in Supporting Information Data S1).

## 3 | RESULTS AND DISCUSSION

The schematic pathway for the synthesis of the catalyst is shown in Scheme 1.

The synthesized nanoparticles were characterized using different spectroscopic and thermogravimetric techniques, including FT-IR, XRD, VSM, TGA, SEM, and energy dispersive X-ray spectroscopy. The FT-IR spectrum of the catalyst (Figure 1) shows an absorption peak at 3400 cm<sup>-1</sup>, which is attributed to the stretching vibration of surface hydroxyl groups of magnetite



SCHEME 1 Pathway to synthesis of MN-Tric-Cu



FIGURE 1 XRD pattern of MN-Tric-Cu

nanoparticles and also free hydroxyl groups of the tricine ligand. The absorption peak at 1624 cm<sup>-1</sup> is due to the stretching vibration of the carbonyl compound of tricine, which is slightly shifted downfield compared to free tricine ligand (1650 cm<sup>-1</sup>).<sup>[36]</sup> The peak at 2900 cm<sup>-1</sup> can be assigned to C–H of tricine. Finally, the strong band at 590 cm<sup>-1</sup> is due to stretching vibrations of F–O bonds, which are characteristic of magnetite nanoparticles.

To confirm the complete formation of the spinel structure, the XRD pattern of the nanoparticles was obtained (Figure 2). The reported diffraction peaks of Fe<sub>3</sub>O<sub>4</sub> (JCPDS card no. 74–2,403) were seen at  $2\theta = 21.13$ , 34.85, 41.21, 50.30, 62.42, 66.43, and 73.68.

The magnetic properties of the prepared nanoparticles were studied using VSM in the applied field of -1,000 to +1,000 Oe (Figure 3). The magnetic saturation of nanoparticles is 32 emu g<sup>-1</sup>, which confirms the superparamagnetic nature of nanoparticles. Moreover, the superparamagnetic nanoparticles also have reversibility and zero remanence.



FIGURE 2 FT-IR spectra of MN-Tric-Cu



FIGURE 3 VSM of MN-Tric-Cu



FIGURE 4 TGA of MN-Tric-Cu

The thermal stability of nanoparticles and the amount of organic part can be analyzed using thermogravimetric analysis (TGA) (Figure. 4). The absorbed moisture of the nanoparticles is about 21% according to the curve (weight



**FIGURE 5** SEM image of MN-Tric-Cu

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loss under 200°C) and almost 30% of the material consists of organic moiety (weight loss between 200 and  $600^{\circ}$ C).

SEM was applied to survey the morphology of the nanoparticles (Figure 5). The SEM image shows that the nanoparticles are spherical and of uniform size.

Successful immobilization of copper ions on the surface of the nanoparticles was demonstrated by EDS (energy dispersive X-ray spectroscopy) (Figure 6). The EDS spectrum shows emission peaks for the constituent elements of the ligand (N, C, O) and the magnetic core (Fe). The copper content of the nanoparticles was analyzed using ICP(Inductively coupled plasma) analysis, which showed 0.4 mmol copper per gram of nanoparticles.

The prepared catalyst was used in the reaction of amine with acetone. The best reaction conditions were determined using the reaction of aniline as the model reaction (Scheme 2). No product was obtained in the absence of catalyst. When bare  $Fe_3O_4$  was used, the product did not form. The reaction was tested in various conditions (Table 1). It was observed that reaction did not proceed in any solvent except a little in DMSO. We therefore examined mixtures of DMSO with other solvents to find a way to promote the reaction. Using a mixture of DMSO and CHCl<sub>3</sub> resulted in higher yields. The best ratio of DMSO and CHCl<sub>3</sub> was 3/1. Different amounts of catalyst were also used and a suitable amount was chosen. Furthermore, the effect of different temperatures on the reaction was surveyed (Table 2).

The reactivity of different amines was tested under the optimized reaction conditions (Table 3). No noticeable difference between electron-donating and withdrawing substituents was detected.

The reusability of the catalyst was tested using the model reaction. After completion of the reaction, the catalyst was removed using magnetic decantation. It was then washed with ethanol, water, and ether and dried at 40°C then reused for the next run of the reaction. This process was repeated until a noticeable loss in reaction yield occurred (five cycles). The results are shown in Figure 7. The heterogeneity of the catalyst was confirmed using the hot filtration test and ICP analysis of the residual reaction mixture.



**FIGURE 6** EDS analysis of MN-Tric-Cu



**SCHEME 2** Acetylation of aniline and acetone

TABLE 1	Optimization of reaction conditions for aniline and
acetone <sup>a</sup>	

Entry	Catalyst (mg)	Solvent	Yield (%) <sup>b</sup>
1	40	$CH_2Cl_2$	-
2	40	CHCl <sub>3</sub>	-
3	40	CH <sub>3</sub> CN	-
4	40	EtOH	-
5	40	МеОН	-
6	40	H <sub>2</sub> O	-
7	40	DMF	-
8	40	Toluene	-
9	40	THF	-
10	40	DMSO	3
11	40	DMSO/CH <sub>2</sub> Cl <sub>2</sub> (1/1)	3
12	40	DMSO/CHCl <sub>3</sub> (1/1)	70
13	40	DMSO/CH <sub>3</sub> CN	3
14	40	DMSO/EtOH	3
15	40	DMSO/MeOH	3
16	40	DMSO/H <sub>2</sub> O	3
17	40	DMSO/DMF	3
18	40	DMSO/Toluene	3
19	40	DMSO/THF	3
20	40	DMSO/CHCl <sub>3</sub> (2/1)	85
21	40	DMSO/CHCl <sub>3</sub> (3/1)	90
22	40	DMSO/CHCl <sub>3</sub> (4/1)	88
23	40	DMSO/CHCl <sub>3</sub> (5/1)	76
24	-	DMSO/CHCl <sub>3</sub> (3/1)	-
25	40 <sup>c</sup>	DMSO/CHCl <sub>3</sub> (3/1)	-
26	80	DMSO/CHCl <sub>3</sub> (3/1)	92
27	40 <sup>d</sup>	DMSO/CHCl <sub>3</sub> (3/1)	_

<sup>a</sup>*Note.* DMF, dimethylformamide; DMSO, dimethyl sulfoxide; THF, tetrahydrofuran.

<sup>a</sup>Reaction conditions: amine (1 mmol), acetone (1 mmol), catalyst and solvent (each 1 ml), reflux (DMF, DMSO and DMSO mixture,  $120^{\circ}$ C), 24 hr.

<sup>b</sup>Isolated yield.

°Fe<sub>3</sub>O<sub>4</sub> nanoparticles.

<sup>d</sup>Copper powder.

**TABLE 2** Optimization of reaction temperature for aniline

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	-
and acetone <sup>a</sup>	

Entry	Temperature (°C)	Yield (%) <sup>b</sup>
1	25	-
2	40	-
3	60	-
4	80	_
5	100	70
6	120	90
7	130	90
8	140	92
9	150	92
10	160	92

<sup>a</sup>Reaction conditions: amine (1 mmol), acetone (1 mmol), catalyst (40 mg), DMSO/CHCl<sub>3</sub> (each 1 ml), 24 hr. <sup>b</sup>Isolated yield

 $^{\circ}$ Fe<sub>3</sub>O<sub>4</sub> nanoparticles.

TABLE 3	MN-Tric-Cu catalyzed reaction of amines and
acetone	



<sup>a</sup>Reaction conditions: amine (1 mmol), acetone (1 mmol), catalyst (40 mg), DMSO/CHCl<sub>3</sub> (3/1, 4 ml), 120°C, 24 hr.
<sup>b</sup>Isolated yield.

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The recycled catalyst was characterized after the sixth run and showed no significant changes in its structure (Figure 8).

A comparison between the efficiency of the reported catalyst with other reports of amide formation from C–C bond oxidative cleavage of ketones is shown in Table 4. It



**FIGURE 7** Reusability of MN-Tric-Cu in the reaction of aniline and acetone

is evident that the reported catalyst is more efficient than the others for much lower copper catalyst loading. This catalyst can also be recycled for six consecutive cycles, which is a highly important characteristic in industrial point of view.

A plausible mechanism for the reaction is represented in Scheme 3.<sup>[30]</sup> First, amine and acetone react to form enamine (step A). Evidence for the enamine intermediate was confirmed experimentally. Enamine was formed and isolated according to classical procedures (aniline and acetone plus a few drops of concentrated sulfuric acid) and underwent C-C oxidative cleavage reaction under the optimized reaction conditions.<sup>[37]</sup> In addition, chloroform reacts to hydrochloric acid and dichlorocarbine in reaction with DMSO (step B). In the next step, a carbine complex of copper forms which in reaction with enamine (step C) transfers the carbine moiety to enamine to undergo cyclopropanation (step D). The copper atom adds to the C-C bond of cyclopropane to form a copper complex (steps E and F), which leads to a peroxy-copper species after oxidation with dioxygen from air (step G). Next the terminal oxygen of the peroxy complex



FIGURE 8 (a) FT-IR, (b) XRD, (c) VSM, (d) TGA, (e) SEM, and (f) EDS of recycled catalyst after the fifth run

#### TABLE 4 Comparison of catalyst efficiency in the C-C oxidative cleavage synthesis of amides<sup>a</sup>

Entry	Catalyst (mg)	Catalyst loading (mol%)	Yield <sup>b</sup> (%)
1	MN-Tric-Cu	1.6	90
2	Cu (OAc) <sub>2</sub>	10	86

<sup>a</sup>Reaction conditions: amine (1 mmol), acetone (1 mmol), catalyst (40 mg), DMSO/CHCl<sub>3</sub> (3/1, 4 ml), 120°C, 24 hr. <sup>b</sup>Isolated yield.

### SCHEME 3 Proposed

mechanism of the reaction



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attacks the nitrogen of the imine intermediate and dichloroethene forms as the side product (step H). Following attack by the acetate ion on the copper complex, the initial active catalyst is recycled (step I). In the next step (step J), after hydrogen exchange with released hydrogen chloride (step B) and chloride ion attack, the desired product, acetylated amine, is obtained (step K).

## **4** | **CONCLUSION**

We devised a novel heterogeneous copper nanocatalyst for direct and efficient synthesis of acetamides from amine and acetone. This synthetic strategy has the advantage of easy separation of catalyst as well as high reusability of the catalyst. Furthermore, no sensitive reagent and harsh reaction conditions are necessary, and this protocol is environmentally friendly. In addition, no cumbersome or time-consuming procedure is required for purification of the product.

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#### SUPPORTING INFORMATION DATA

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Spectroscopic and characterization data for the catalyst and products are contained in the electronic Supporting Information Data file in the supporting information tab for this article.

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#### SUPPORTING INFORMATION

Additional supporting information may be found online in the Supporting Information section at the end of this article.

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