

Nano Fe₃O₄ as a magnetically recyclable, powerful, and stable catalyst for the multi-component synthesis of highly functionalized dihydro-2-oxopyrroles

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Abstract A mild and efficient method for the one-pot synthesis of functionalized dihydro-2-oxopyrroles is described via four-component domino reaction of amines, dialkyl acetylenedicarboxylates, and formaldehyde in the presence of super magnetic nano iron oxide (nano Fe₃O₄) in EtOH at room temperature. The major advantages of the present method are mild and green reaction conditions, high yields, short reaction times, clean reaction, magnetically recyclable catalyst, atom economy (63–76 %), operational simplicity, and no need for column chromatography. It is found that the catalyst is recyclable and can be used up to six times without significant loss of its activity.

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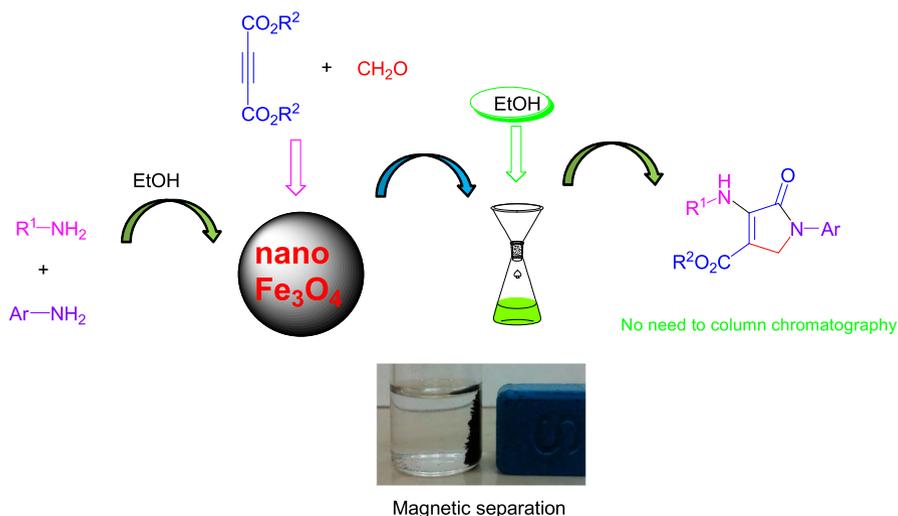
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Graphical Abstract



Keywords Nano Fe_3O_4 · Heterocycle · Dihydro-2-oxopyrrole · Dialkyl acetylenedicarboxylate · Green · Magnetically recyclable · Multi-component

Introduction

Nitrogen heterocycles have been the subject of intense research because of their outstanding biological properties and wide range of applications to pharmaceutical compounds, agrochemicals, and synthetic intermediates. For example, pyrrole and its derivatives such as dihydro-2-oxopyrroles (dihydropyrrol-2-ones) and pyrrolidines are the important structural motifs that are found in natural and synthetic compounds [1]. Especially, functionalized dihydro-2-oxopyrroles are an important class of nitrogen-containing five-membered heterocycles. They are the building blocks of many natural products such as bilirubins [2, 3] and indolocarbazole alkaloids [4]. They have also been used as inhibitors of HIV integrase [5], cardiac cyclic AMP phosphodiesterase [6], vascular endothelial growth factor receptors [7], pesticides [8], herbicides [9] and also as antitumor agents [10]. They are also considered as a precursor to pyrrolizidine alkaloids [11]. Structures of some biologically important dihydro-2-oxopyrrole are shown in Fig. 1. As a result, considerable efforts have been made for the synthesis of this important class of compounds [12–17].

Green chemistry and the development of new processes that reduce pollution in chemical synthesis have received considerable attention due to increasing environmental concerns. Among other factors, two major adverse effects on the environment are consumption of energy for heating and cooling of reactions and using volatile organic solvents. The performance of reactions at ambient temperature in green solvents such as water and ethanol may be a viable option [18, 19].

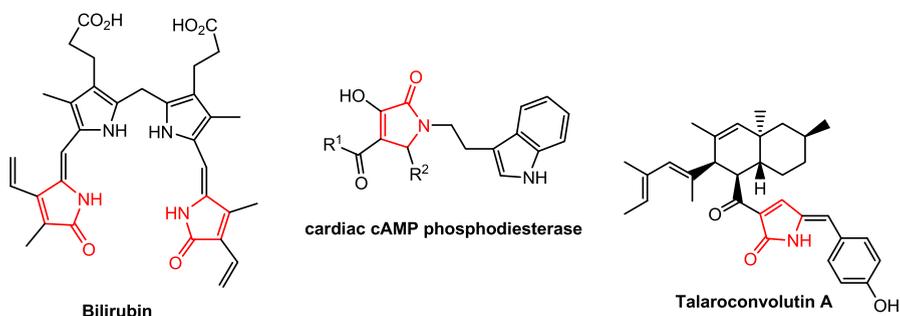


Fig. 1 Bioactive compounds containing dihydro-2-oxopyrrole unit

On the other hand, one active area to access green chemistry is multi-component reactions (MCRs).

Recently, multi-component reactions (MCRs) have received great attention from research groups in medicinal chemistry, drug discovery, and materials science due to their significant advantages over conventional linear-type syntheses, including simple procedures, eco-friendliness, atom economy, and the ability to generate architecturally complex molecules in one synthetic step [20]. Nevertheless, few methods have been reported for the preparation of dihydro-2-oxopyrroles by one-pot MCR using catalysts, such as acetic acid [21], molecular iodine [22], Al(H₂PO₄)₃ [23], Cu(OAc)₂·H₂O [24], 1-methyl-2-oxopyrrolidinium hydrogen sulfate ([Hpyro][HSO₄]) [25], TiO₂ nanopowder [26], tetrabutylammonium hydrogen sulfate ([n-Bu₄N][HSO₄]) [27], (*S*)-camphorsulfonic acid [28], sucrose [29], InCl₃ [30], maltose [31], xylose [32], and Cl₃CCO₂H [33]. However, many of these catalysts are expensive, highly corrosive, and involve tedious separation procedures and also require prolonged reaction times. Therefore, the development of a simple, convenient, and an efficient protocol using inexpensive and readily available reagents would extend the scope of the synthesis of highly functionalized dihydro-2-oxopyrroles. Recently, transition-metal-catalyzed organic reactions are often considered to follow the principles of “green chemistry” due to the use of minimum energy, cleaner reagents or auxiliaries and minimization of waste. Nanocatalysts are considered to be a bridge between heterogeneous and homogeneous catalysis [34, 35]. One of the attractive properties of the nanomaterials is that the active component has a high specific surface area, leading to an increase of the contact with the reactants [36]. Also, a higher surface area gives nanomaterials a more active surface; they are hard to be separated. Therefore, it is important to design a recoverable and well-dispersed catalyst. Magnetite nanoparticles (MNPs) as catalysts are very promising due to their large specific surface area and their magnetic properties [37, 38]. They can be collected very easily using a magnet for reusing it to prevent any loss of the catalyst. Recently, chemists have focused on the catalytic aspects of nano-Fe₃O₄ to improve the protocols of catalytic activity [39–42].

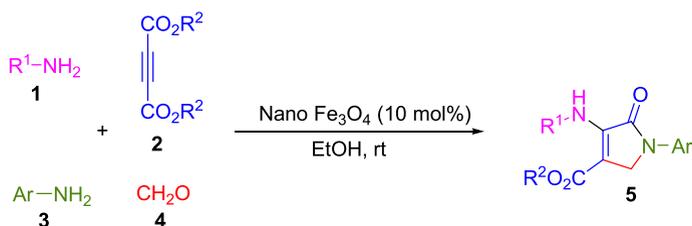
As a part of our current studies on the development of efficient multi-component reactions for the preparation of interesting bioactive molecules [43–46], in this article we report that nano Fe₃O₄ (10 mol %) is a highly efficient and economically

viable catalyst for the one-pot synthesis of highly functionalized dihydro-2-oxopyrroles from amines, dialkyl acetylenedicarboxylates, and formaldehyde in EtOH at room temperature (Scheme 1). Interestingly, this method has none of the above-mentioned drawbacks for the dihydro-2-oxopyrroles. The catalyst can be easily collected by a magnet and recovered without significant loss in the catalytic activity.

Results and discussion

It is well known that nano Fe_3O_4 as a Lewis acid catalyst have gained interesting attraction in recent years due to operational simplicity, efficient, low cost, magnetically recyclable catalyst, being more stable, and economic considerations [47, 48].

Initially, a test reaction using aniline, dimethyl acetylenedicarboxylate (DMAD), and formaldehyde was performed in the absence of a catalyst in EtOH at room temperature. The corresponding product, 5a, was obtained in trace amounts after 24 h (Table 1, entry 15). To optimize the reaction conditions, the above reaction



Scheme 1 Synthesis of highly functionalized dihydro-2-oxopyrroles 5

Table 1 Optimization of the reaction conditions for the synthesis 5a

Entry	Catalyst (mol%)	Solvent	Time (h)	Yield (%) ^a
1	5	EtOH	4	70
2	5	MeOH	6	62
3	5	MeCN	7	56
4	5	CHCl_3	8	45
5	5	CH_2Cl_2	8	48
6	5	EtOAc	8	41
7	5	THF	10	35
8	5	Acetone	7	50
9	5	H_2O	12	53
10	5	–	12	40
11	7.5	EtOH	3	82
12	10	EtOH	2.5	89
13	12.5	EtOH	2.5	89
14	15	EtOH	2.5	86
15	–	EtOH	24	Trace

^a Yield of isolated product

was examined under different conditions and the results are presented in Table 1. The best result was obtained in the presence of 10 mol % Fe₃O₄ in EtOH (Table 1, entry 12). As shown in Table 1, a higher percentage loading of the catalyst neither increased the yield of product nor decreased the reaction time. Additionally, the effect of different solvents including MeOH, MeCN, CHCl₃, CH₂Cl₂, EtOAc, THF, acetone, and H₂O was also investigated on the yield and time of reaction, which was found to be ineffective. When the reaction was carried out under solvent-free conditions, the product was obtained in a moderate yield (40 %), which may be due to the lack of effective interaction of reactants with the catalyst in the absence of a solvent (Table 1, entry 10). Next, the scope of this reaction has been extended by means of reaction between various aromatic amines, dialkyl acetylenedicarboxylates, and formaldehyde under optimized conditions. The results are summarized in Table 2.

In general, all the reactions are clean, and the dihydro-2-oxopyrrole derivatives were obtained in quantitative yields (Table 2, entries 1–11). Encouraged by these results, substituted dihydro-2-oxopyrroles with different

Table 2 Synthesis of highly substituted dihydro-2-oxopyrroles **5**

Entry	R ¹	R ²	Ar	Compound	Time (h)	Yield (%) ^a	Mp (°C)	Lit. mp (°C) (Refs.) ^b
1	Ph	Me	Ph	5a	2.5	89	153–155	155–156 [22]
2	Ph	Et	Ph	5b	3	84	137–139	138–140 [21]
3	4-Me-C ₆ H ₄	Me	4-Me-C ₆ H ₄	5c	2.5	86	177–178	177–178 [22]
4	4-Me-C ₆ H ₄	Et	4-Me-C ₆ H ₄	5d	2.5	85	129–131	131–132 [21]
5	4-Cl-C ₆ H ₄	Me	4-Cl-C ₆ H ₄	5e	2.5	85	170–172	173–174 [22]
6	4-Cl-C ₆ H ₄	Et	4-Cl-C ₆ H ₄	5f	3	83	169–171	168–170 [27]
7	4-F-C ₆ H ₄	Et	4-F-C ₆ H ₄	5g	3.5	80	173–174	172–173 [21]
8	4-OMe-C ₆ H ₄	Me	4-OMe-C ₆ H ₄	5h	2.5	89	174–176	176–177 [22]
9	4-OMe-C ₆ H ₄	Et	4-OMe-C ₆ H ₄	5i	3	80	152–153	152–154 [23]
10	4-Br-C ₆ H ₄	Me	4-Br-C ₆ H ₄	5j	2.5	85	178–180	179–180 [22]
11	4-Br-C ₆ H ₄	Et	4-Br-C ₆ H ₄	5k	3	85	170–171	169–171 [21]
12	PhCH ₂	Me	Ph	5l	2.5	85	139–141	140–141 [21]
13	PhCH ₂	Et	Ph	5m	4.5	82	127–129	130–132 [21]
14	PhCH ₂	Me	4-OMe-C ₆ H ₄	5n	3	81	125–127	129–130 [22]
15	PhCH ₂	Me	4-Cl-C ₆ H ₄	5o	3	83	145–147	147–148 [22]
16	PhCH ₂	Me	4-Br-C ₆ H ₄	5p	3	85	122–123	120–121 [22]
17	<i>n</i> -C ₄ H ₉	Me	Ph	5q	4	79	58–60	60 [22]
18	<i>n</i> -C ₄ H ₉	Me	4-Br-C ₆ H ₅	5r	4	77	108–110	108–109 [22]
19	C ₆ H ₁₁	Me	Ph	5s	3.5	79	92–93	94–95 [21]
20	C ₆ H ₁₁	Me	4-OMe-C ₆ H ₄	5t	4	75	127–129	128–129 [22]
21	C ₆ H ₁₁	Me	4-Cl-C ₆ H ₄	5u	4	77	123–125	124–125 [22]
22	C ₆ H ₁₁	Me	4-Br-C ₆ H ₅	5v	3.5	80	121–122	123–124 [22]

^a Isolated yields

^b The references of known products in the literature

Table 3 Comparison of nano Fe₃O₄ with previously reported catalyst for the synthesis of dihydro-2-oxopyrrole **4b**

Entry	Catalyst	Conditions	Time (h)	Yield (%)	(Refs.)
1	[Hpyro][HSO ₄]	MeOH, r.t.	6	80	[25]
2	AcOH	EtOH, 70 °C	4	85	[21]
3	Al(H ₂ PO ₄) ₃	MeOH, r.t.	5	80	[23]
4	I ₂	MeOH, r.t.	1	81	[22]
5	[n-Bu ₄ N][HSO ₄]	MeOH, r.t.	4	86	[27]
6	Sucrose	MeOH, 60 °C	4	84	[29]
7	InCl ₃	MeOH, r.t.	3	85	[30]
8	Nano Fe ₃ O ₄	EtOH, r.t.	3	84	This work

Table 4 Recycling of the catalyst for the four-component coupling reaction of diethyl acetylenedicarboxylate, 4-methyl aniline, and formaldehyde (product **5d**)

Run	Time (h)	Yield (%) (isolated)
1	2.5	85
2	2.5	85
3	2.5	84
4	2.5	83
5	2.5	83
6	2.5	81

substitutions were synthesized via one-pot four-component domino reaction of aliphatic amines **1**, dialkyl acetylenedicarboxylates **2**, aromatic amine **3**, and formaldehyde **4** (Table 2, entries 12–22). Aliphatic amines include benzyl amine, *n*-butyl amine and cyclo hexyl amine were used and the corresponding products were achieved in good to high yields. As all products were precipitated in EtOH, separated from reaction mixture by simple filtration and washed with EtOH to give pure products.

To show the merit of the present work in comparison with reported results in the literature, we compared results of nano Fe₃O₄ with the other catalysts for the synthesis of compounds **4b**. As shown in Table 3, nano Fe₃O₄ can act as more effective catalyst with respect to reaction times and yields of products.

In addition, a catalytic recycle experiment (Table 4) was done with the standard reaction. After the completion of the reaction, as indicated by TLC, the solid material was separated, dried, and then dissolved in acetone (10 ml). The nanoparticles were separated with an external magnet (Fig. 2) and then washed four times with acetone, dried, and reused for the standard reaction for up to six cycles without significant loss of its activity.

On the basis of above experimental results together with the related reports [24, 25], a plausible mechanism for this four-component domino reaction is proposed as shown in Scheme 2. The first two steps involve the reaction of amine **1** with dialkyl acetylenedicarboxylate **2** to form dialkyl 2-(aryl or alkylamino) fumarate **7**, and the

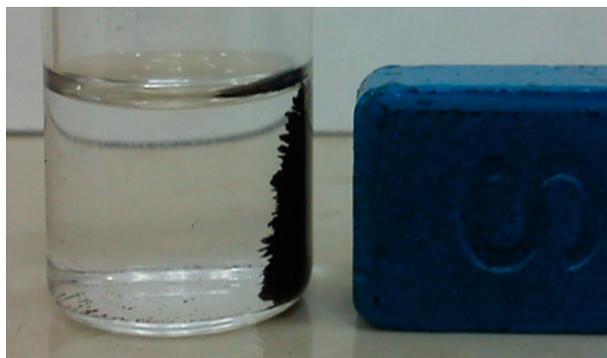


Fig. 2 Schematic representation of nano Fe₃O₄ and its magnetic recyclable capability

condensation of aromatic amine **3** with formaldehyde **4** to generate imine **8**. Next, intermediate **7** undergoes a Mannich-type reaction with imine **8** to furnish reactive intermediate **9**, followed by intramolecular cyclization to afford intermediate **10**. In the final step, cyclic intermediate **10** tautomerizes to obtain the functionalized dihydro-2-oxopyrroles **5**.

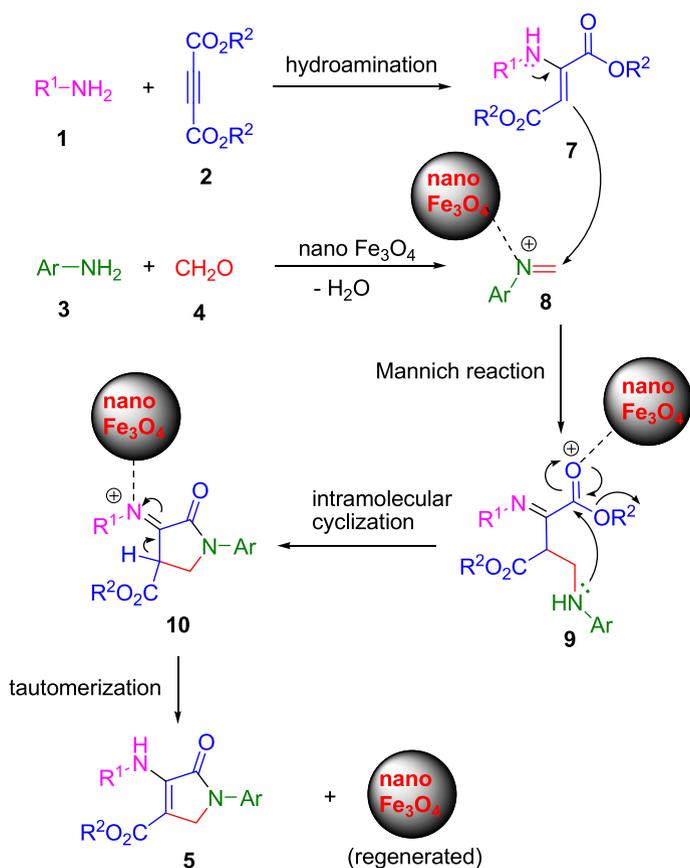
Experimental

General

Melting points were taken on an Electrothermal 9100 apparatus. IR spectra were obtained on a JASCO FT/IR-460 plus spectrometer. The ¹H and ¹³C NMR spectra were recorded on a Bruker DPX-250 Avance instrument with CDCl₃ as solvent and using TMS as internal reference at 250 and 62.9 MHz, respectively. Chemicals were purchased from Merck (Darmstadt, Germany), Acros (Geel, Belgium), and Fluka (Buchs, Switzerland), and used without further purification.

General procedure for the synthesis of dihydro-2-oxopyrroles **5**

A mixture of amine **1** (1 mmol) and dialkyl acetylenedicarboxylate **2** (1 mmol) in ethanol (3 ml) was stirred for 20 min. Next, aromatic amine **3** (1 mmol), formaldehyde **4** (37 % solution, 1.5 mmol), and nano Fe₃O₄ (10 mol %) were added successively. The reaction mixture was allowed to stir at room temperature for the appropriate time (see Table 2). The progress of the reaction was monitored by TLC. After completion, the solid material was separated, dried, and then dissolved in acetone (10 ml). The nanoparticles were separated with an external magnet and then washed five times with acetone. The solvent was evaporated under vacuum. The obtained solid was washed with EtOH (3 × 2 ml) to give the pure product **5**. It was found that the catalyst is recyclable and can be used up to six cycles without significant loss of its activity.



Scheme 2 Proposed mechanism for the synthesis dihydro-2-oxopyroles 5

Spectral data of selected products are represented below

Compound 5a 1H NMR (250 MHz, $CDCl_3$) δ : 3.73 (3H, s, OCH_3), 4.54 (2H, s, CH_2), 7.12–7.42 (8H, m, ArH), 7.78 (2H, d, $J = 8.75$ Hz, ArH), 8.02 (1H, br s, NH).

Compound 5c 1H NMR (250 MHz, $CDCl_3$) δ : 2.34 (6H, s, 2Me), 3.74 (3H, s, OCH_3), 4.48 (2H, s, CH_2), 7.04 (2H, d, $J = 8.25$), 7.12 (2H, d, $J = 8.5$ Hz), 7.18 (2H, d, $J = 8.5$ Hz), 7.66 (2H, d, $J = 8.3$), 8.02 (1H, br s, NH).

Compound 5d 1H NMR (250 MHz, $CDCl_3$) δ : 1.22 (3H, t, $J = 7.1$ Hz, OCH_2CH_3), 2.34 (6H, s, 2 CH_3), 4.21 (2H, q, $J = 7.2$ Hz, OCH_2CH_3), 4.50 (2H, s, CH_2), 7.03 (2H, d, $J = 8.5$ Hz, ArH), 7.11 (2H, d, $J = 8.2$ Hz, ArH), 7.18 (2H, d, $J = 8.25$ Hz, ArH), 7.66 (2H, d, $J = 8.5$ Hz, ArH), 7.98 (1H, br s, NH).

Compound 5g ¹H NMR (250 MHz, CDCl₃) δ: 1.54 (3H, t, *J* = 7.1 Hz, OCH₂CH₃), 4.51 (2H, m, OCH₂CH₃), 4.77 (2H, s, CH₂), 7.25–7.42 (6H, m, ArH), 8.0 (2H, m, ArH), 8.32 (1H, br s, NH).

Conclusions

In summary, the magnetically recoverable iron oxide nanoparticles are found to be more efficient for the synthesis of highly functionalized dihydro-2-oxopyrroles via one-pot, four-component reaction of amines, dialkyl acetylenedicarboxylates and formaldehyde in EtOH at room temperature. The salient features are mild and green reaction conditions, high yields, short reaction times, simple experimental procedure, atom economy (63–76 %), low cost, no need for column chromatography, and the use of easily recyclable catalyst without loss of considerable catalytic activity.

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