Res Chem Intermed
DOI 10.1007/s11164-016-2555-2

Three-component synthesis of pyrano[2,3-*d*] pyrimidinone derivatives catalyzed by Ni^{2+} supported on hydroxyapatite-core–shell-γ-Fe_2O_3 nanoparticles in aqueous medium

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Received: 24 February 2016 / Accepted: 19 April 2016

Abstract Pyrano[2,3-*d*]pyrimidine derivatives were synthesized via one-pot, three-component condensation reaction of aromatic aldehydes, malononitrile, and barbituric acid in aqueous ethanol catalyzed by Ni^{2+} supported on hydroxyapatite-core–shell-γ-Fe_2O_3 nanoparticles (γ-Fe_2O_3@HAp-Ni^{2+} NPs) as Lewis acid. The nontoxic nature and easy handling of the catalyst, environmentally friendly and facile work-up procedure, short reaction time, low catalyst loading, and high effectiveness to give products in good to excellent yield are advantages of this approach. Also, γ-Fe_2O_3@HAp-Ni^{2+} NPs can be easily recovered and reused for at least six runs.

Keywords Nanoparticles · Lewis acid catalyst · Supported Ni^{2+} · Multicomponent reaction · Pyrano[2,3-*d*]pyrimidinone · Knoevenagel reaction

Introduction

Multicomponent reactions (MCRs) have attracted considerable interest due to their ease of performance, high yield, and especially synthetic feasibility and efficiency [1–3]. MCRs include three or more starting materials reacting in a single flask to

Electronic supplementary material The online version of this article (doi:10.1007/s11164-016-2555-2) contains supplementary material, which is available to authorized users.

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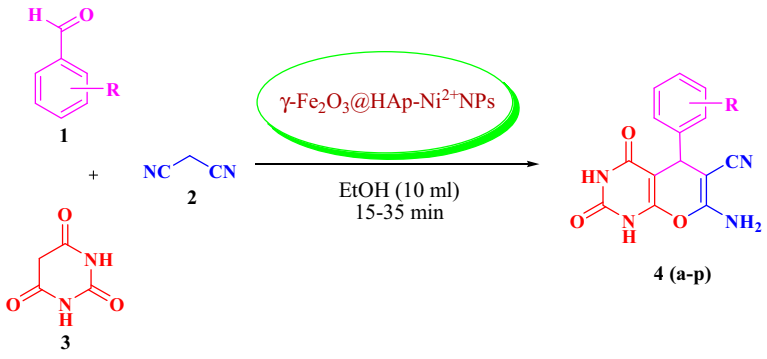
Published online: 28 April 2016

form a new product, where basically all the atoms contribute to the newly formed product. Pyrano[2,3-*d*]pyrimidine derivatives are examples where multicomponent synthesis can be used. Due to the diverse biological activity of compounds in this class, there is great interest in their synthesis.

Pyrano[2,3-*d*]pyrimidine structures have a wide range of biological activities such as spasmolytic, diuretic, anticoagulant, antitumor, antiallergic, anticoagulant, anticancer, and antianaphylactic activity, as well as acting as potassium channel activators [4–6]. Synthesis of pyrano[2,3-*d*]pyrimidines has been achieved by many methods, including microwave condition [7, 8], ultrasonic irradiation [9], in absence of catalysts [10], $Zn[(L)proline]_2$ [11], 1,4-diazabicyclo[2.2.2]octane (DABCO) [12], alum [13], and ionic liquids [11]. Reported methods appearing in literature usually involve forcing conditions, long reaction time, waste creation, need for complex synthetic procedures, and involvement of organic solvents as well as high energy to proceed. So, due to environmental concerns associated with aspects of organic solvents, development of aqueous-phase synthesis of pyrano[2,3-*d*]pyrimidines is of considerable interest in such research to achieve short reaction time, environmentally friendly procedures, and excellent yield by such new methods. Recently, application of nanoparticles (NPs) has attracted considerable attention in both industrial and academic research due to their unique properties based on their high surface area and unique magnetic properties. Also, they have a wide range of uses in various fields such as biology and medical applications [14], magnetic resonance imaging [15], environmental remediation [16], data storage [17], and magnetic fluids [18], while recent studies have also shown that magnetic nanoparticles are excellent supports for various catalysts [19, 20].

As part of our current studies aimed at identifying ecofriendly methods for synthesis of pyrano[2,3-*d*]pyrimidinones (**4a**–**p**), we report herein a one-pot, three-component reaction between aromatic aldehydes, malononitrile, and barbituric acid catalyzed by Ni^{2+} supported on hydroxyapatite-core–shell-γ-Fe_2O_3 NPs in aqueous ethanol at room temperature, achieving good to excellent isolated yields (85–95 %) (Scheme 1).



Scheme 1 Synthesis of pyrano[2,3-*d*]pyrimidine derivatives

Experimental

Materials and methods

All chemicals were purchased from Merck or Fluka chemical companies. All products are known compounds and were characterized by comparing ^{1}H and ^{13}C nuclear magnetic resonance (NMR) spectroscopic data and melting points with literature values. Uncorrected melting points of all compounds were measured in an open capillary in a paraffin bath. ^{1}H and ^{13}C NMR spectra were recorded using a Bruker instrument (^{1}H at 400 MHz and ^{13}C at 100 MHz) in dimethyl sulfoxide (DMSO)-d_6 solvent with tetramethylsilane (TMS) as internal standard. Infrared (IR) spectra were recorded on a Frontier FT-IR (PerkinElmer) spectrometer using a KBr disk. Thin-layer chromatography (TLC) was performed with silica gel 60 F254 plates with ultraviolet (UV) light for visualization. The phases present in the magnetic materials were analyzed by powder XRD (model X0 Pert with X'Pert; Philips, The Netherlands) with Cu $K_{\alpha 1}$ radiation ($\lambda = 1.5401$ Å), operating the X-ray generator at 40 kV and 30 mA. Diffraction patterns were collected in the 2 h range of 20–80°.

Preparation of γ-Fe_2O_3@HAp-Ni^{2+} NPs

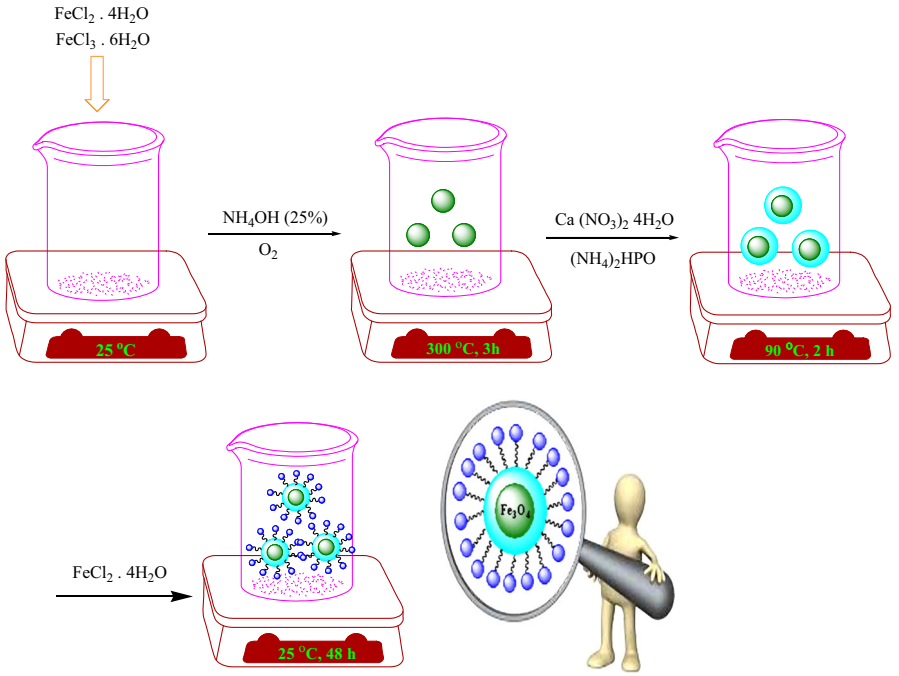
In this study, γ-Fe_2O_3@HAp-Ni^{2+} NPs were prepared according to literature procedure. Then, the mean size and surface morphology of the γ-Fe_2O_3@HAp-Ni^{2+} NPs were characterized by transmission electron microscopy (TEM), scanning electron microscopy (SEM), vibrating-sample magnetometry (VSM), X-ray diffraction (XRD) analysis, and Fourier-transform infrared (FTIR) spectroscopy techniques [21–23].

General procedure for synthesis of pyrano[2,3-*d*]pyrimidinone derivatives 4a–p

Substituted aromatic aldehydes **1** (1 mmol), malononitrile **2** (1 mmol), barbituric acid **3** (1 mmol), and γ-Fe_2O_3@HAp-Ni^{2+} NPs (10 mg) in EtOH (10 mL) was stirred at room temperature for appropriate time (see Table 4). After reaction completion as monitored by TLC (ethyl acetate:*n*-hexane 7:3), the filtrate mixture was recrystallized to provide pure crystals of pyrano[2,3-*d*]pyrimidinone derivatives with excellent yields (85–95 %). The products were known compounds and were characterized based on IR and NMR spectroscopic data. Their melting points (m.p.) were compared with reported values.

Results and discussion

In continuation of our studies toward development of new and cleaner methods for organic transformations [23–32], in this study, we prepared Ni^{2+} supported on hydroxyapatite-core–shell-γ-Fe_2O_3 nanoparticles, according to reported procedures (Scheme 2), and then used them for one-pot, three-component condensation



Scheme 2 Synthesis of catalyst

Table 1 Effect of various loadings of γ-Fe_2O_3@HAp-Ni^{2+} NPs for synthesis of **4a** in solvent-free conditions at room temperature

Entry	Amount of catalyst (mg)	Time (min)	Yield (%)[b]
1	No catalyst	2 h[a]	39
2	1	50	65
3	5	50	72
4	10	40	88
5	15	35	79
6	20	45	68
7	30	45	55

[a] In hours

[b] Isolated yield

reaction of aromatic aldehydes with malononitrile and barbituric acid in EtOH as green solvent at room temperature (Scheme 1). The structure of the γ-Fe_2O_3@HAp-Ni^{2+} NPs was characterized by FTIR spectroscopy, XRD analysis, VSM, SEM, and TEM (see supporting information for experimental procedures for synthesis of γ-Fe_2O_3@HAp-Ni^{2+} NPs) [22, 23].

To optimize the catalyst loading, the reaction of benzaldehyde **1** (1 mmol) with malononitrile **2** (1 mmol) and barbituric acid **3** (1 mmol) was investigated as a model, and its behavior studied under solvent-free conditions at room temperature (Table 1). As shown in Table 1, in absence of catalyst, the reaction was slow and resulted in poor yield (39 %), even after long reaction time (2 h) (Table 1, entry 1). The best result was obtained when the reaction was carried out in presence of 10 mg γ-Fe_2O_3@HAp-Ni^{2+} NPs under solvent-free conditions at room temperature (Table 1, entry 4). Higher

Table 2 Synthesis of compound **4a** in presence of γ-Fe_2O_3@HAp-Ni^{2+} NPs (10 mg) in various solvents

Entry	Solvent	Time (min)	Yield (%)[a]
1	MeCN	45	50
2	EtOH	25	95
3	MeOH	60	71
4	$CHCl_3$	60	Trace
5	Ethyl acetate	60	Trace
6	CH_2Cl_2	60	65
7	$PhCH_3$	60	Trace

[a] Isolated yield

catalyst loading (20–30 mg) neither increased the yield nor shortened the conversion time (Table 1, entries 6, 7). Hence, the optimum concentration of γ-Fe_2O_3@HAp-Ni^{2+} NPs was chosen as 10 mg in the model reaction.

In the next step, to improve the yield of **4a**, we carried out the test reaction in presence of polar and nonpolar solvents such as acetonitrile, ethanol, methanol, chloroform, ethyl acetate, dichloromethane, and acetone; the results are presented in Table 2, indicating that the solvent had a significant effect on product yield and reaction time. As shown in Table 2, it was found that ethanol was a solvent of choice for the reaction, with product **4a** obtained after 25 min in excellent yield (Table 2, entry 2). When the reaction proceeded in other solvents, the desired products were obtained in low yield even after 60 min (Table 2, entries 1, 3, and 6). No reaction occurred when ethyl acetate or chloroform was used as solvent (Table 2, entries 5, 6). Therefore, this reaction was developed with other aldehydes; the results are summarized in Table 4. Use of aqueous ethanol as solvent in the reaction medium showed remarkable benefits, such as environmental safety, lack of carcinogenic effects, comparatively low cost to operate, and simple work-up.

Then, we examined the catalytic activity of various acids such as citric acid, tartaric acid, cyanuric chloride, *N*,*N*,*N*′,*N*′-tetrabromobenzene-1,3-disulfonamide (TBBDA), and poly(*N*,*N*′-dibromo-*N*-ethylbenzene-1,3-disulfonamide) (PBBS) in the model reaction of benzaldehyde **1** (1 mmol) with malononitrile **2** (1 mmol) and

Table 3 Synthesis of compound **4a** using various catalysts

Entry	Catalyst	Time (min)	Yield (%)[c]
1	Citric acid	120	51
2	Tartaric acid	120	45
3	Cyanuric chloride	120	55
4	TBBDA[a]	180	63
5	PBBS[b]	180	65
6	Silica gel	120	NR
7	γ-Fe_2O_3@HAp-Ni^{2+} NPs	25	95

[a] *N*,*N*,*N*′,*N*′-tetrabromobenzene-1,3-disulfonamide

[b] Poly(*N*,*N*′-dibromo-*N*-ethylbenzene-1,3-disulfonamide)

[c] Isolated yield

Table 4 Synthesis of pyrano[2,3-*d*]pyrimidinones catalyzed by γ-Fe_2O_3@HAp-Ni^{2+} NPs

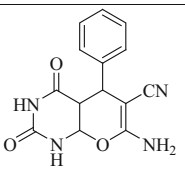
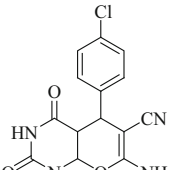
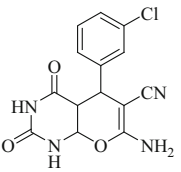
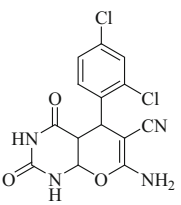
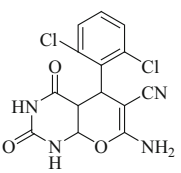
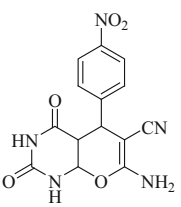
Entry	Product	Time (min)	Yield (%)[a]	m.p. (°C)	
				Found	Reported
1	 **4a**	25	95	205–207	206–208 [34]
2	 **4b**	15	90	235–237	234–235 [34]
3	 **4c**	25	92	241–243	242–244 [34]
4	 **4d**	17	93	238–240	239–241 [34]
5	 **4e**	25	85	225–226	227–228 [35]
6	 **4f**	15	95	237–240	239–241 [34]

Table 4 continued

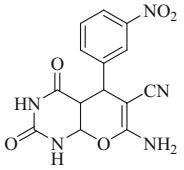
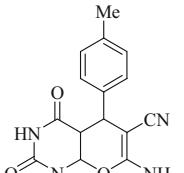
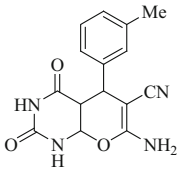
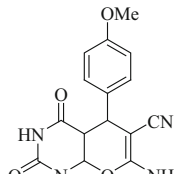
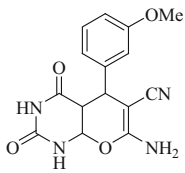
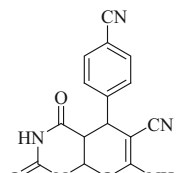
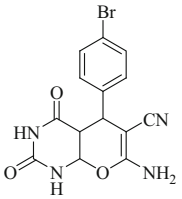
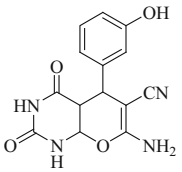
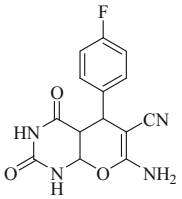
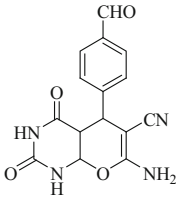
Entry	Product	Time (min)	Yield (%)[a]	m.p. (°C)	
				Found	Reported
7	 **4g**	20	93	272–274	271–272 [34]
8	 **4h**	17	91	226–228	226–227 [34]
9	 **4i**	20	90	225–227	224–225 [34]
10	 **4j**	25	91	280–282	280–284 [34]
11	 **4k**	20	88	223–225	200–202 [34]
12	 **4l**	15	93	252–254	252–253 [34]

Table 4 continued

Entry	Product	Time (min)	Yield (%)[a]	m.p. (°C)	
				Found	Reported
13	 **4m**	15	92	235–236	235–236 [34]
14	 **4n**	30	90	161–163	160–162 [34]
15	 **4o**	20	90	269–271	268–270 [34]
16	 **4p**	35	88	332–334	335–337 [34]

[a] Isolated yield

barbituric acid **3** (1 mmol) in EtOH as green solvent at room temperature (Table 3). As can be seen in Table 3, various catalysts such as citric acid (entry 1), tartaric acid (entry 2), and cyanuric chloride (entry 3) were less efficient. Also, it is also clearly seen from these data that no yield of the corresponding **4a** was obtained when amorphous silica gel was employed as catalyst (Table 3, entry 6). The reaction was very slow when using TBBDA (Table 3, entry 4) or PBBS (Table 3, entry 5), with the corresponding product isolated in only 63 or 65 % yield, respectively, after 180 min (Table 1, entry 8). Excellent product yield (95 %) was obtained in short reaction time (25 min) when γ-Fe_2O_3@HAp-Ni^{2+} NPs was utilized as catalyst (Table 3, entry 7). This study clearly showed that γ-Fe_2O_3@HAp-Ni^{2+} NPs exhibit higher activity under the same reaction conditions and

catalyst loading. γ-Fe_2O_3@HAp-Ni^{2+} NPs are a good catalyst for synthesis of pyrano[2,3-*d*]pyrimidinone derivatives. Therefore, this reaction was developed with other aldehydes; the results are summarized in Table 4.

As shown in Table 4, various pyrano[2,3-*d*]pyrimidinone derivatives (**4a**–**p**) were prepared from aromatic aldehydes (with electron-withdrawing as well as electron-donating groups such as Cl, Br, F, CH_3, OCH_3, CN, OH, and NO_2) with malononitrile and barbituric acid in EtOH as green solvent at room temperature in good to excellent yields (85–95 %) in short reaction times (15–35 min). In general, the results given in Table 4 reveal that excellent yield and short reaction time of pyrano[2,3-*d*]pyrimidinone derivatives were obtained under mild conditions. The data obtained from IR, ^{1}H NMR, and ^{13}C NMR spectra confirmed all of the proposed products. The structures of compounds **4a**–**p** were deduced from IR, ^{1}H and ^{13}C NMR spectra, confirming all of the proposed products.

Catalyst recycling is one of the most important advantages of our method. For this purpose, the reaction of benzaldehyde **1** (1 mmol) with malononitrile **2** (1 mmol) and barbituric acid **3** (1 mmol) was performed in presence of γ-Fe_2O_3@HAp-Ni^{2+} NPs (10 mg) at room temperature. After reaction completion as monitored by TLC, the mixture was filtered, and complete separation of the product from the surface of the catalyst was achieved by washing the catalyst with hot ethanol (2 × 5 mL). Finally, the recovered catalyst was washed with acetone and dried before use for six consecutive runs (Fig. 1).

Although we have not established the reaction mechanism, a possible explanation is given in Scheme 3. The reaction may proceed first via Knoevenagel condensation of aryl aldehyde **5** with malononitrile **6** to afford Michael acceptor **8**. Intramolecular cyclization condensation of **10** gives **11**. Finally, intermediate **11** converts to product **12** after proton transfer and tautomerization. The reaction mechanism is assumed to be the same as that proposed by Rezaee Nezhad et al. in 2009 for synthesis of dihydropyrimidinone derivatives [33].

To show the efficiency of the present method, we compare the results of pyrano[2,3-*d*]pyrimidinone derivative synthesis from benzaldehyde **1** (1 mmol), malononitrile **2** (1 mmol), and barbituric acid **3** (1 mmol) in presence of

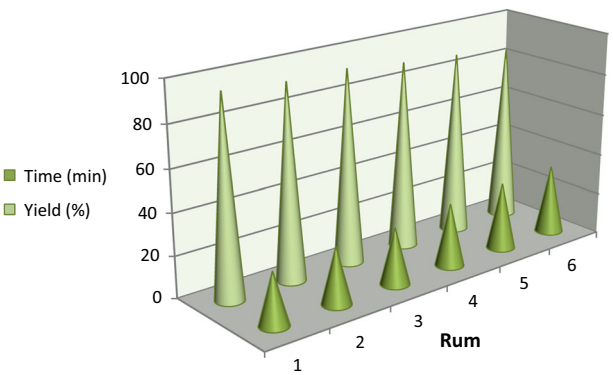
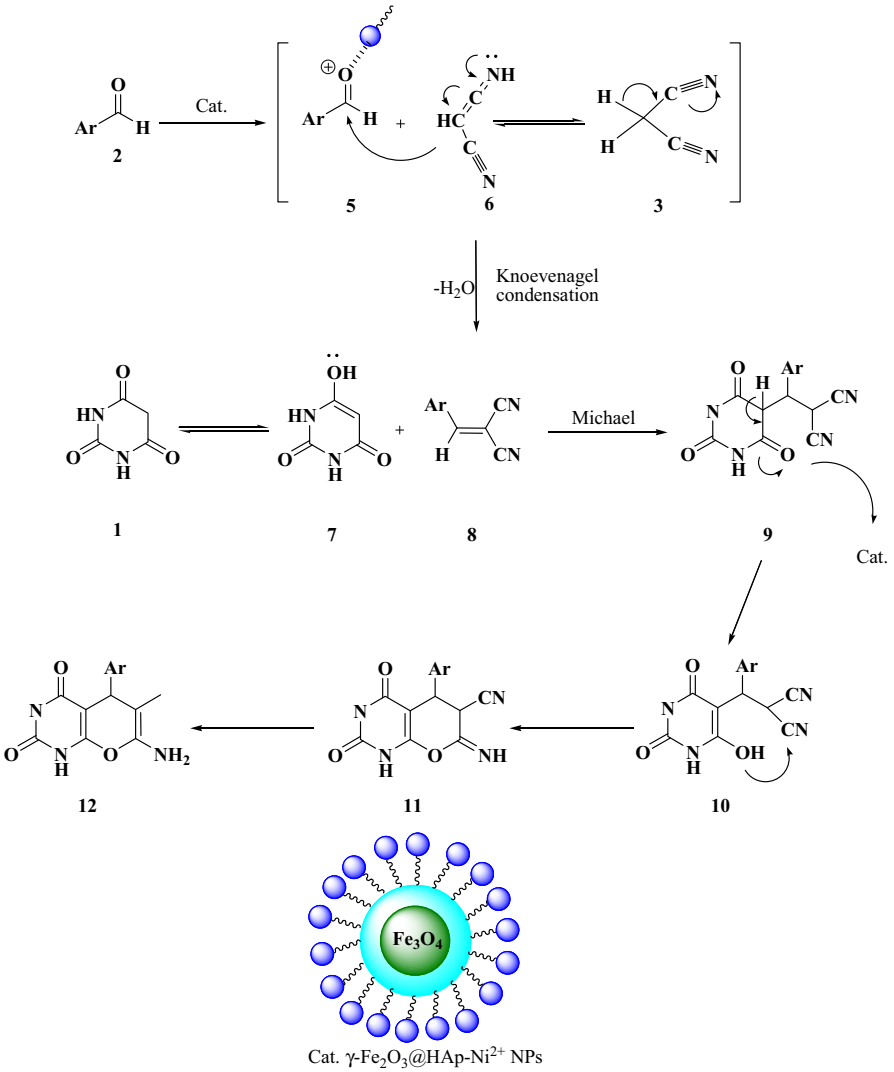


Fig. 1 Recyclability of γ-Fe_2O_3@HAp-Ni^{2+} NPs



Scheme 3 Proposed mechanism for one-pot synthesis of pyrano[2,3-*d*]pyrimidinones catalyzed by γ-Fe_2O_3@HAp-Ni^{2+} NPs

$[KAl(SO_4)_2]$, $[BMIm]BF_4$, Zn[(l)proline]$_2$, diammonium hydrogen phosphate (DAHP), dibutylamine, L-proline, 1,4-dioxane, $H_{14}[NaP_5W_{30}O_{110}$, and SBA-Pr-$SO_3H$ with respect to catalyst reusability, reaction time, product yield, and reaction condition in Table 5. Recently, use of $[BMIm]BF_4$ (Table 5, entry 2), Zn[(l)proline]$_2$ (Table 5, entry 3), and DAHP (Table 5, entry 4) was reported for preparation of pyrano[2,3-*d*]pyrimidine derivatives. These methods have longer reaction times and lower product yields. Also, slightly longer reaction times (30–120 min) are found when using L-proline (Table 5, entry 6) as catalyst, with the corresponding

Table 5 Comparison of results for synthesis of pyrano[2,3-*d*]pyrimidinone derivatives catalyzed by our new catalyst with those obtained by recently reported catalysts

Entry	Catalyst	Condition	Time (min)	Yield (%)[a]	Ref.
1	$[KAl(SO_4)_2]$	H_2O/80 °C	30–45	81–88	[13]
2	[BMIm]BF_4	[BMIm] BF_4/90 °C	3–5 h	82–95	[11]
3	$Zn[(l)proline]_2$	EtOH/reflux	0.5–12 h	80–92	[35]
4	DAHP	EtOH/r.t.	2 h	71–81	[36]
5	Dibutylamine	Aq. EtOH/reflux	43–129	83–94	[37]
6	L-Proline	Aq. EtOH/reflux	30–120	68–85	[38]
7	1,4-Dioxane	H_2O/reflux	1–2	60–70	[39]
8	$H_{14}[NaP_5W_{30}O_{110}$	EtOH/reflux	30–60	85–90	[40]
9	SBA-Pr-SO_3H	Solvent-free/140 °C	10–45	30–91	[34]
10	γ-Fe_2O_3@HAp-Ni^{2+} NPs	EtOH/r.t.	15–35	85–95	This work

[a] Isolated yield

Table 6 Scalability of synthesis of pyrano[2,3-*d*]pyrimidinone derivatives by reaction of aromatic aldehydes (15 mmol), malononitrile (15 mmol), and barbituric acid (15 mmol) using γ-Fe_2O_3@HAp-Ni^{2+} NPs (10 mg) in EtOH as green solvent at room temperature

Entry	Product	Time (min)	Yield (%)[a]
1	**4a**	40	91
2	**4f**	15	92
3	**4h**	25	87
4	**4m**	19	91

[a] Isolated yield

product isolated in only 68–85 % yield. Use of 1,4-dioxane (Table 5, entry 7) as catalyst for synthesis of pyrano[2,3-*d*]pyrimidinone derivatives has been reported, requiring reaction time of almost 1–2 h with low product yields (60–70 %). Use of SBA-Pr-SO_3H (Table 5, entry 9) as catalyst for synthesis of pyrano[2,3-*d*]pyrimidinone derivatives has also been reported. This method relies on use of high temperature (140 °C) and also has low product yields (30–91 %). As indicated by Table 5, γ-Fe_2O_3@HAp-Ni^{2+} NPs show remarkably improved synthesis of pyrano[2,3-*d*]pyrimidinone, with shorter reaction times (15–35 min) and higher yields (85–95 %).

In subsequent work, to determine the scalability of our method, we examined some reactions at larger scale (15 mmol of each reactant); the results are summarized in Table 6, revealing that the reactions were successfully performed at larger scale without significant loss of yield.

Conclusions

We report an efficient and green method for one-pot, three-component synthesis of pyrano[2,3-*d*]pyrimidinones (**4a**–**p**) catalyzed by an efficient and heterogeneous nanocatalyst of Ni^{2+} supported on hydroxyapatite-core–shell-γ-Fe_2O_3 as reusable

Lewis acid catalyst in EtOH at room temperature. The catalyst is stable and can promote yields and reaction times over six runs without significant loss of activity. Furthermore, short reaction time, solvent-free reaction condition, high product yield, nonchromatographic product purification, i.e., simple recrystallization from ethanol, easy work-up, and clean procedure make this approach a useful addition to available methods. The new catalyst was fully characterized by several techniques including FTIR spectroscopy, XRD analysis, VSM, TEM, and SEM.

Acknowledgments We gratefully acknowledge funding from the National Elites Foundation of Iran for this research.

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