Monatsh Chem (2014) 145:1839–1843
DOI 10.1007/s00706-014-1240-7

ORIGINAL PAPER

Iron(II,III) oxide nanoparticle-catalyzed selective synthesis of unknown dihydropyrano[*c*]chromenes under green conditions

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Received: 18 February 2014 / Accepted: 22 April 2014 / Published online: 5 June 2014

Abstract An efficient one-pot, three-component condensation reaction between 4-hydroxycoumarin, aryl glyoxals, and malononitrile for the synthesis of new dihydropyrano[*c*]chromenes is reported. The reactions were efficiently catalyzed by magnetically recyclable Fe_3O_4 nanoparticles to afford the desired products in good to excellent yields. The method is simple and completely in accordance with green chemistry principles.

Keywords Fe_3O_4 NPs · Aryl glyoxal · Dihydropyrano[*c*]chromenes · Malononitrile · Catalyst

Introduction

The continual insistence on the development of facile, convenient, and nonpolluting synthetic procedures drives chemists to update methods and increase their potency with respect to green chemistry factors [1–3]. Multicomponent reactions (MCR) of three or more substrates are one approach to address this challenge, because they offer the greatest possibilities for molecular diversity in one step with minimum synthetic time and effort; many attempts have been made to extend new MCRs in the past decade [4–6]. Other promising areas of green chemistry are the design of organic reactions in nontoxic and inexpensive solvents and using safe and recyclable catalysts [7–9].

The design and synthesis of biologically active compounds is one of the most important aspects of medicinal chemistry. Pyranochromenes represent an interesting template for medicinal chemistry and play an essential role as biologically active molecules, e.g., antifungal, insecticidal, anticancer, anti-HIV, anti-inflammatory, and antibacterial agents [10–12]. Figure 1 shows some drugs with dihydropyran substructures and a 4-hydroxycoumarin moiety. Among pyranochromenes, the dihydropyrano[*c*]chromenes are the most synthetically feasible.

To our knowledge, a number of methods are available for the synthesis of dihydropyrano[*c*]chromenes via domino Knoevenagel condensation and Michael addition reaction of aromatic aldehyde with malononitrile and 4-hydroxycoumarin, but there is no report on using aryl glyoxal instead of aromatic aldehydes [13].

Recently, iron oxide nanoparticles (Fe_3O_4 NPs) were extensively applied as a powerful catalyst for some organic transformations [14, 15]. Fe_3O_4 NPs have special features that make them advantageous over some common catalysts and suitable for many organic reactions. For instance, they are safe, inexpensive, stable, and recyclable materials. In this context, we report the use of Fe_3O_4 NPs as an eco-friendly and efficient catalyst for the synthesis of new dihydropyrano[*c*]chromenes containing an aryloyl group.

Results and discussion

The Fe_3O_4 NPs, homogeneous in size and composition, were prepared according to previous methods [16] by modification and characterized by Fourier transform

Electronic supplementary material The online version of this article (doi:10.1007/s00706-014-1240-7) contains supplementary material, which is available to authorized users.

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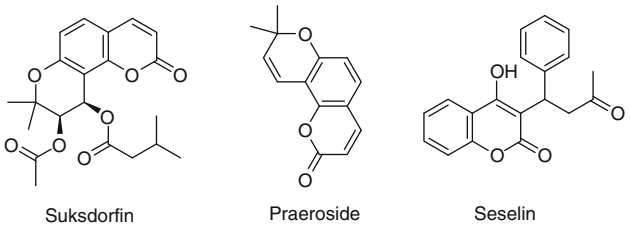


Fig. 1 Representative drugs with dihydropyran substructures and a 4-hydroxycoumarin moiety

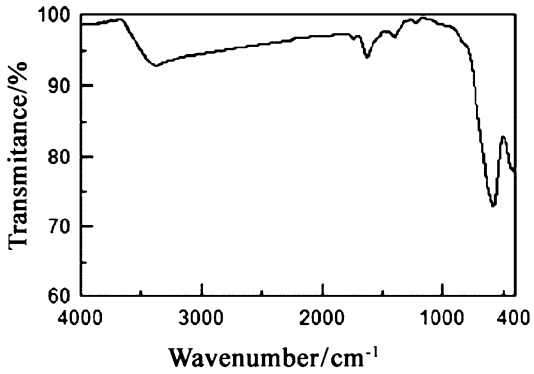


Fig. 2 FT-IR spectra of Fe_3O_4 NPs

infrared (FT-IR) and transmission electron microscopy (TEM). The FT-IR spectrum of Fe_3O_4 NPs is shown in Fig. 2. The absorbance band at 583.3 cm^{-1} can be ascribed to Fe^{2+}–O^{2-}, which is consistent with the reported IR spectra for Fe_3O_4 NPs [17]. The morphology and microstructure of the NPs were further investigated by TEM analysis. The TEM images in Fig. 3 reveal spherical Fe_3O_4 NPs with an average size of 10–15 nm.

In continuation of our interest in developing convenient methodologies for new heterocyclic synthesis using green catalysts [18–22], we found that treatment of 4-hydroxycoumarin (**1**), aryl glyoxals **2**, and malononitrile (**3**) via a one-pot, three-component reaction in the presence of catalytic amounts of Fe_3O_4 NPs provided the dihydropyrano[*c*]chromenes **4**.

The synthesis of pyrano[*c*]chromenes **4** from the aryl glyoxals was unknown. In order to optimize the reaction conditions, we studied the model reaction between 4-hydroxycoumarin, phenyl glyoxal, and malononitrile. The reaction was initially conducted in EtOH/H_2O (1:1) without using catalysts, but no product formation was observed after 6 h at rt or reflux conditions. Addition of a small amount of Fe_3O_4 NPs (2 mol%) to the reaction mixture afforded the desired product after 180 min. Increasing the catalyst to 3 mol% increased the reaction rate. Further increase did not improve the reaction rate or product yield.

A few additional experiments were performed to investigate whether this transformation took place when other common catalysts (*p*-TSA, Na_2CO_3, $FeCl_3$, AcOH, and $ZnCl_2$) were used in place of the Fe_3O_4 NPs, but no reaction was observed in all cases. Next, some experiments were performed to investigate the solvent effect. Solvent-free reactions produced a mixture of undesired and non-isolable products. Among solvents screened, EtOH/H_2O (1:1) was found to be the best in terms of the product yield and completion time in comparison with common organic solvents. The results of these studies are summarized in Table 1. This reaction was efficiently completed using Fe_3O_4 NPs (3 mol%) under controlled temperature (see "Experimental" section) in EtOH/H_2O (1:1) after about 100 min (entry 3).

Having established the optimized reaction conditions, we concentrated on the scope of this reaction with a variety of aryl glyoxals to check the viability of this protocol in obtaining a library of dihydropyrano[*c*]chromenes (Table 2). It is worth noting that the experimental procedure is very efficient, convenient, and rapid and has the ability to tolerate a variety of functional groups, such as methoxy, nitro, and halides under optimized reaction conditions.

We also examined the use of alkyl cyanoactates to get the corresponding products but there was no product formation even after 12 h under the optimized reaction conditions.

The structures of compounds **4** were assigned on the basis of comprehensive spectroscopic analyses such as IR, ^1H and ^{13}C NMR spectroscopy, and CHN analysis. The ^1H NMR spectrum of **4a** exhibited two sharp singlets identified as methine ($\delta = 5.42$ ppm) and NH_2 (7.69 ppm) protons. Also, the signals at 7.53–8.17 ppm corresponded to aromatic protons. The proton-decoupled ^{13}C NMR spectrum of **4a** showed 18 distinct resonances in agreement with the proposed structure. The IR spectrum of **4a** showed characteristic bands at 3402–3292 cm^{-1} (NH_2), 2201 cm^{-1} (CN), and at 1708–1678 cm^{-1} (C=O).

The Fe_3O_4 NPs are magnetically separable and could be recovered easily and reused for subsequent runs. As it is shown in Fig. 4, no obvious decrease was observed on the recovered catalyst. After three reuses, the Fe_3O_4 NPs maintained their high catalytic efficiency in the model reaction.

In summary, an efficient, economical, and environmentally benign multicomponent protocol for the construction of a new class of dihydropyrano[*c*]chromenes under Fe_3O_4 NP-catalyzed conditions has been achieved. The use of controlled heating allows the tuning of reaction conditions to access products in good to excellent yield. The promising points of the presented methodology are the avoidance of toxic and expensive solvents, use of safe and recyclable catalyst, and simplicity of operation. It is also noteworthy that the presence of transformable functionalities in the products makes them potentially valuable for further synthetic manipulations.

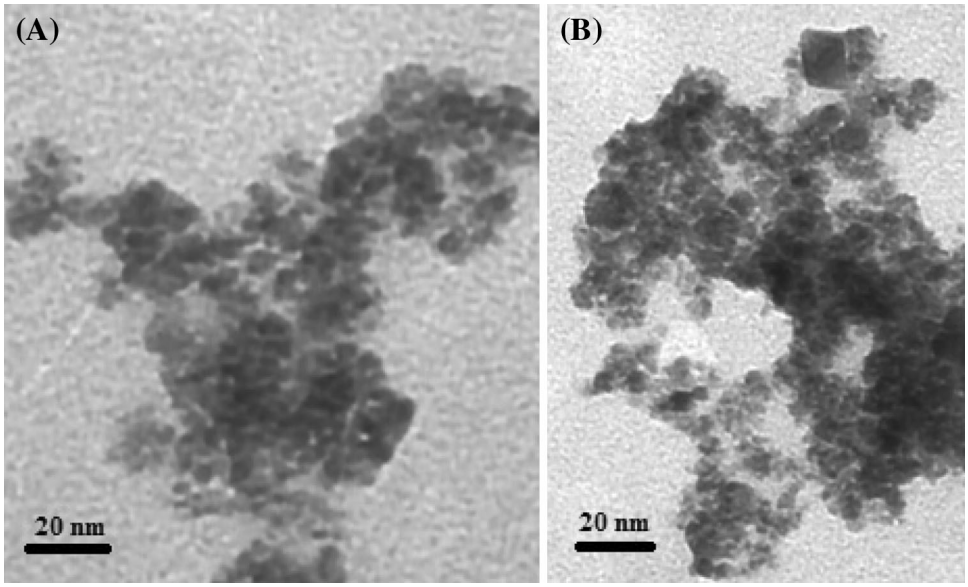


Fig. 3 TEM image shows spherical Fe_3O_4 NPs with 10–15 nm size

Table 1 Effect of several conditions in synthesis of **4a** under controlled temperature (reaction time: 100 min)

Entry	Catalyst	Solvent	Yield/%
1	Catalyst-free	EtOH/H_2O (1:1)	–
2	Fe_3O_4 NPs (2 mol%)	EtOH/H_2O (1:1)	70
3	Fe_3O_4 NPs (3 mol%)	EtOH/H_2O (1:1)	90
4	Fe_3O_4 NPs (5 mol%)	EtOH/H_2O (1:1)	90
5	Fe_3O_4 NPs (3 mol%)	EtOH	75
6	Fe_3O_4 NPs (3 mol%)	MeOH	70
7	Fe_3O_4 NPs (3 mol%)	THF	50
8	Fe_3O_4 NPs (3 mol%)	H_2O	25
9	Fe_3O_4 NPs (3 mol%)	CH_2Cl_2	25
10	Fe_3O_4 NPs (3 mol%)	Solvent-free	Trace
11	*p*-TSA (5 mol%)	EtOH/H_2O (1:1)	Trace
12	Na_2CO_3 (5 mol%)	EtOH/H_2O (1:1)	–
13	$FeCl_3$ (5 mol%)	EtOH/H_2O (1:1)	–
14	AcOH (5 mol%)	EtOH/H_2O (1:1)	–
15	$ZnCl_2$ (5 mol%)	EtOH/H_2O (1:1)	–

Experimental

All chemicals were purchased from Merck and Aldrich. Aryl glyoxals were synthesized according to our previous method [23]. The reactions were monitored by thin-layer chromatography (TLC; silica-gel 60 F_{254}, *n*-hexane/ethyl acetate). IR spectra were recorded on an FT-IR JASCO-680 and the ^1H NMR spectra were obtained on a Bruker-Instrument DPX-400 and 300 MHz Avance 2 model. The varioEl CHNS system at Isfahan Industrial University was used for elemental analysis. Transmission electron microscopy (TEM) images were taken with a Philips CM-10 TEM microscope

Table 2 Synthesis of dihydropyrano[*c*]chromenes using Fe_3O_4 NPs in EtOH/H_2O (Scheme 1)

Product	Ar	Yield[a]/%	Time/min	M.p./°C
4a	C_6H_5	90	100	272–274
4b	4-F-C_6H_4	85	100	253–255
4c	4-Cl-C_6H_4	87	95	263–265
4d	4-Br-C_6H_4	90	110	263–265
4e	3-NO_2-C_6H_4	80	120	248–250
4f	4-NO_2-C_6H_4	93	100	273–275
4g	3-MeO-C_6H_4	80	115	268–270
4h	4-MeO-C_6H_4	85	110	266–268
4i	1-Naphthyl	93	90	271–273
4j	2-Naphthyl	95	90	278–280

[a] Isolated yields

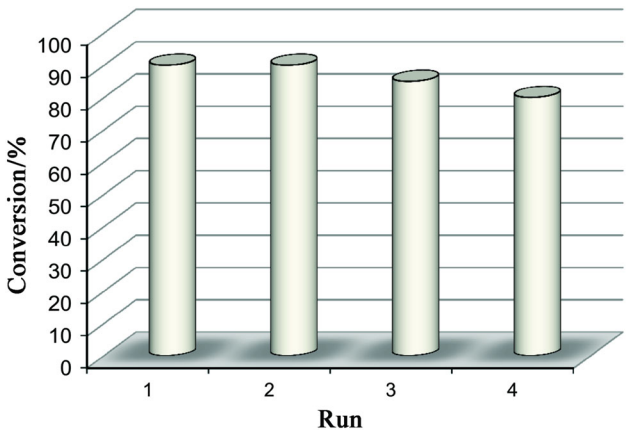
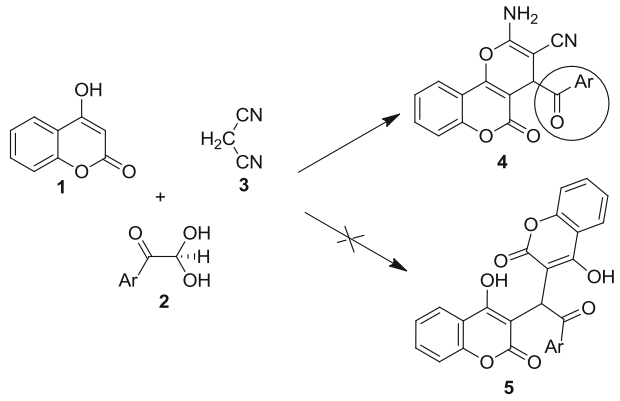


Fig. 4 Recyclability of Fe_3O_4 NPs in synthesis of **4a** (reaction time 100 min)

Scheme 1



Conditions: EtOH/H_2O (1:1), Fe_3O_4 NPs (3 mol%), r.t. to reflux

operated at 100 kV. The structures and purity of the obtained products were deduced from their IR, elemental analysis, and NMR spectral data.

Preparation of Fe_3O_4 NPs

$FeCl_3{\cdot}6H_2O$ (6.1 g, 0.02 mol) and 2.35 g $FeCl_2{\cdot}4H_2O$ (0.01 mol) were dissolved in 100 cm^3 de-ionized H_2O under magnetic stirring for 10 min. The solution was then heated to 90 °C under nitrogen atmosphere. Subsequently, 10 cm^3 ammonium hydroxide solution (25 %) was added dropwise to the reaction mixture which was then stirred for about 1 h. The reaction mixture was cooled to room temperature and the black precipitate was separated from the reaction mixture using a magnetic field and then washed with de-ionized H_2O several times to remove the impurities.

*General procedure for synthesis of **4***

To a stirred solution of 4-hydroxycoumarin (1 mmol), aryl glyoxal (1 mmol), and malononitrile (1.2 mmol) in 10 cm^3 EtOH/H_2O (1:1) was added Fe_3O_4 NPs (0.03 mmol). The mixture was stirred under reflux for 40 min. The reaction progress was monitored by TLC (hexane/AcOEt, 1:1). After completion of the reaction, the precipitate was filtered, dried, and dissolved in hot EtOH/THF (3:1) to separate the catalyst using a magnet. Pure **4** was obtained after recrystallization from EtOH/THF (3:1).

2-Amino-4-benzoyl-5-oxo-4H,5H-pyrano[3,2-c]chromene-3-carbonitrile (**4a**, $C_{20}H_{12}N_2O_4$)
IR (KBr): $\bar{\nu}$ = 3,402, 3,292, 2,201, 1,708, 1,678, 1,606, 1,373, 1,064 cm^{-1}; ^{1}H NMR (DMSO-d_6, 300 MHz): δ = 8.16 (d, 2H, J = 7.2 Hz), 7.90 (dd, 1H, J = 8.2, 1.6 Hz), 7.81–7.73 (m, 2H), 7.69 (s, 2H), 7.62 (t, 2H, J = 8.2 Hz), 7.57–7.53 (m, 2H), 5.42 (s, 1H) ppm; ^{13}C NMR (DMSO-d_6, 75 MHz): δ = 198.12, 160.08, 159.55, 154.72, 152.11, 135.34, 134.13, 133.35, 129.13, 128.89, 125.03, 122.15, 118.54, 116.83, 112.58, 101.91, 51.91, 37.14 ppm.

2-Amino-4-(4-fluorobenzoyl)-5-oxo-4H,5H-pyrano[3,2-c]-chromene-3-carbonitrile (**4b**, $C_{20}H_{11}FN_2O_4$)
IR (KBr): $\bar{\nu}$ = 3,474, 3,404, 2,205, 1,712, 1,677, 1,595, 1,371, 1,218, 1,061 cm^{-1}; ^{1}H NMR (DMSO-d_6, 300 MHz): δ = 8.27 (m, 2H), 7.90 (dd, 1H, J = 8.2, 1.8 Hz), 7.82–7.76 (m, 1H), 7.70 (s, 2H), 7.58–7.53 (m, 2H), 7.46 (t, 2H, J = 8.8 Hz), 5.44 (s, 1H) ppm; ^{13}C NMR (DMSO-d_6, 75 MHz): δ = 196.77, 160.08, 159.54, 154.74, 152.13, 133.37, 132.32, 132.19, 125.04, 122.17, 118.50, 116.84, 116.15, 115.86, 112.58, 101.76, 51.85, 37.19 ppm.

2-Amino-4-(4-chlorobenzoyl)-5-oxo-4H,5H-pyrano[3,2-c]-chromene-3-carbonitrile (**4c**, $C_{20}H_{11}ClN_2O_4$)
IR (KBr): $\bar{\nu}$ = 3,319, 3,186, 3,027, 2,871, 2,205, 1,713, 1,673, 1,587, 1,375, 1,058, 759 cm^{-1}; ^{1}H NMR (DMSO-d_6, 300 MHz): δ = 8.20 (d, 2H, J = 8.4 Hz), 7.89 (dd, 1H, J = 8.2, 1.4 Hz), 7.81–7.69 (m, 5H), 7.57–7.52 (m, 2H), 5.43 (s, 1H) ppm; ^{13}C NMR (DMSO-d_6, 75 MHz): δ = 197.28, 160.08, 159.54, 154.75, 152.13, 139.22, 134.13, 133.39, 130.99, 129.07, 125.04, 122.18, 118.49, 116.83, 112.55, 101.64, 51.70, 37.26 ppm.

2-Amino-4-(4-bromobenzoyl)-5-oxo-4H,5H-pyrano[3,2-c]-chromene-3-carbonitrile (**4d**, $C_{20}H_{11}BrN_2O_4$)
IR (KBr): $\bar{\nu}$ = 3,316, 3,186, 3,027, 2,871, 2,203, 1,715, 1,673, 1,582, 1,374, 1,057, 620 cm^{-1}; ^{1}H NMR (DMSO-d_6, 300 MHz): δ = 8.10 (d, 2H, J = 8.6 Hz), 7.91–7.76 (m, 4H), 7.71 (s, 2H), 7.58–7.53 (m, 2H), 5.42 (s, 1H) ppm; ^{13}C NMR (DMSO-d_6, 75 MHz): δ = 197.51, 159.54, 154.75, 152.14, 134.45, 133.40, 132.04, 131.05, 125.05, 122.19, 116.84, 112.55, 101.64, 51.70, 37.25 ppm.

2-Amino-4-(3-nitrobenzoyl)-5-oxo-4H,5H-pyrano[3,2-c]-chromene-3-carbonitrile (**4e**, $C_{20}H_{11}N_3O_6$)
IR (KBr): $\bar{\nu}$ = 3,415, 3,086, 2,928, 2,197, 1,712, 1,673, 1,609, 1,525, 1,385, 1,352, 1,063 cm^{-1}; ^{1}H NMR (DMSO-d_6, 300 MHz): δ = 8.83 (t, 1H, J = 1.8 Hz), 8.65 (d, 1H, J = 7.8 Hz), 8.61–8.57 (m, 1H), 7.98–7.89 (m, 2H), 7.82–7.77 (m, 3H), 7.59–7.53 (m, 2H), 5.57 (s, 1H) ppm; ^{13}C NMR (DMSO-d_6, 75 MHz): δ = 197.01, 160.15, 159.57, 154.81, 152.16, 148.17, 136.61, 135.29, 133.51, 130.91, 128.39, 125.11, 123.17, 122.22, 118.51, 116.89, 112.50, 101.33, 51.24, 37.65 ppm.

2-Amino-4-(4-nitrobenzoyl)-5-oxo-4H,5H-pyrano[3,2-c]-chromene-3-carbonitrile (**4f**, $C_{20}H_{11}N_3O_6$)
IR (KBr): $\bar{\nu}$ = 3,461, 3,336, 2,192, 1,720, 1,681, 1,613, 1,517, 1,383, 1,326, 1,070, 1,064 cm^{-1}; ^{1}H NMR (DMSO-d_6, 300 MHz): δ = 8.46–8.38 (m, 4H), 7.90 (dd, 1H, J = 8.2, 1.4 Hz), 7.82–7.77 (m, 3H), 7.59–7.54 (m, 2H),

5.51 (s, 1H) ppm; ^{13}C NMR (DMSO-d_6, 75 MHz): $\delta = 197.79$, 160.15, 159.56, 154.81, 152.17, 150.49, 140.22, 133.50, 130.43, 125.10, 124.01, 122.24, 116.88, 112.51, 101.33, 51.24, 37.98 ppm.

2-Amino-4-(3-methoxybenzoyl)-5-oxo-4H,5H-pyrano[3,2-c]-chromene-3-carbonitrile (**4g**, $C_{21}H_{14}N_2O_5$)
IR (KBr): $\bar{\nu} = 3{,}477$, 3,344, 3,072, 2,934, 2,196, 1,721, 1,674, 1,577, 1,386, 1,065 cm^{-1}; ^{1}H NMR (DMSO-d_6, 300 MHz): $\delta = 7.90$ (dd, 1H, $J = 8.2$, 1.8 Hz), 7.81–7.75 (m, 2H), 7.70 (s, 2H), 7.62 (t, 1H, $J = 2.4$ Hz), 7.57–7.52 (m, 3H), 7.32 (dd, 1H, $J = 7.8$, 2.1 Hz), 5.40 (s, 1H), 3.87 (s, 3H) ppm; ^{13}C NMR (DMSO-d_6, 75 MHz): $\delta = 197.77$, 160.04, 159.61, 159.44, 154.73, 152.13, 136.69, 133.34, 130.02, 125.02, 122.16, 121.68, 120.22, 118.57, 116.82, 113.52, 112.60, 101.96, 55.39, 52.02, 37.41 ppm.

2-Amino-4-(4-methoxybenzoyl)-5-oxo-4H,5H-pyrano[3,2-c]-chromene-3-carbonitrile (**4h**, $C_{21}H_{14}N_2O_5$)
IR (KBr): $\bar{\nu} = 3{,}426$, 3,320, 2,926, 2,200, 1,714, 1,673, 1,597, 1,383, 1,062 cm^{-1}; ^{1}H NMR (DMSO-d_6, 300 MHz): $\delta = 8.15$ (d, 2H, $J = 9.0$ Hz), 7.89 (dd, 1H, $J = 8.2$, 1.4 Hz), 7.80–7.74 (m, 1H), 7.65 (s, 2H), 7.57–7.52 (m, 2H), 7.14 (d, 2H, $J = 9.0$ Hz), 5.36 (s, 1H), 3.90 (s, 3H) ppm; ^{13}C NMR (DMSO-d_6, 75 MHz): $\delta = 196.24$, 163.92, 160.04, 159.50, 154.67, 152.08, 133.26, 131.65, 128.10, 124.99, 122.12, 118.62, 116.79, 114.12, 112.62, 102.10, 55.65, 52.28, 36.72 ppm.

2-Amino-4-(1-naphthoyl)-5-oxo-4H,5H-pyrano[3,2-c]-chromene-3-carbonitrile (**4i**, $C_{24}H_{14}N_2O_4$)
IR (KBr): $\bar{\nu} = 3{,}477$, 3,327, 3,050, 2,923, 2,191, 1,728, 1,675, 1,574, 1,382, 1,177 cm^{-1}; ^{1}H NMR (DMSO-d_6, 300 MHz): $\delta = 8.37$ (m, 2H), 8.24 (d, 1H, $J = 8.4$ Hz), 8.09–8.05 (m, 1H), 7.92 (dd, 1H, $J = 8.2$, 1.8 Hz), 7.83–7.70 (m, 4H), 7.66–7.54 (m, 4H), 5.43 (s, 1H) ppm; ^{13}C NMR (DMSO-d_6, 75 MHz): $\delta = 200.33$, 160.26, 159.65, 154.68, 152.21, 134.20, 133.46, 133.39, 133.29, 129.87, 129.08, 128.56, 128.03, 126.59, 125.10, 125.05, 124.76, 122.20, 118.33, 116.86, 112.63, 101.71, 51.45, 38.65 ppm.

2-Amino-4-(2-naphthoyl)-5-oxo-4H,5H-pyrano[3,2-c]-chromene-3-carbonitrile (**4j**, $C_{24}H_{14}N_2O_4$)
IR (KBr): $\bar{\nu} = 3{,}428$, 3,321, 2,938, 2,199, 1,714, 1,674, 1,631, 1,597, 1,569, 1,382, 1,172 cm^{-1}; ^{1}H NMR (DMSO-d_6, 300 MHz): $\delta = 8.44$ (s, 1H), 7.98–7.95 (m, 2H), 7.83 (d, 1H, $J = 8.4$ Hz), 7.76–7.72 (m, 1H), 7.62–7.52 (m, 3H), 7.49–7.42 (m, 2H), 7.39 (s, 2H), 7.33 (d, 1H, $J = 7.2$ Hz), 5.47 (s, 1H) ppm; ^{13}C NMR (DMSO-d_6, 75 MHz): $\delta = 199.66$, 159.56, 157.85, 153.82, 152.05, 133.27, 132.93, 130.93, 128.47, 127.43, 126.18, 126.13, 126.02, 125.85, 125.75, 124.74, 123.43, 122.45, 119.15, 116.61, 112.96, 104.65, 53.62, 37.27 ppm.

Acknowledgments Financial support for this work by the Young Researchers and Elite Club (Iran) and the Iranian Nanotechnology Initiative Council is gratefully acknowledged.

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