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Efficient One-Pot Synthesis of Some New Pyrimido[5',4':5,6]pyrido[2,3-*d*]pyrimidines Catalyzed by Magnetically Recyclable Fe_3O_4 Nanoparticles[1]

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Abstract—The study is devoted to one-pot reaction of 1,3-dimethylbarbituric acid with aromatic aldehydes and ammonium acetate using Fe_3O_4 nanoparticles as efficient and magnetically recyclable catalysts. Aromatic aldehydes substituted with electron-withdrawing groups or none, reacted successfully with 1,3-dimethylbarbituric acid and ammonium acetate to give new pyrimido[5',4':5,6]pyrido[2,3-*d*]pyrimidine derivatives (can be also named as pyrido[2,3-*d*:6,5-*d'*]dipyrimidines) in high yields over relatively short reaction time. The Knoevenagel condensation products were isolated using aromatic aldehydes bearing electron-donating substituents. The catalyst could be efficiently used for four times without substantial reduction in its activity. The new products were characterized on the basis of FT-IR, ^{1}H NMR and ^{13}C NMR spectral data.

Keywords: pyrimidopyridopyrimidines, pyridodipyrimidines, Fe_3O_4 nanoparticles, magnetically recyclable

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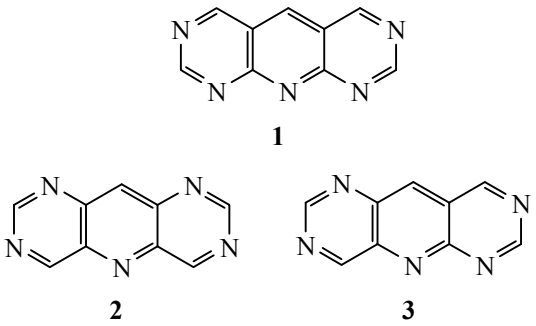
INTRODUCTION

Over recent years pyrimidine-fused pyridines, especially pyrido[2,3-*d*]pyrimidines, have been studied intensively due to their important biological activities including antitumor [1, 2], anticonvulsant and antidepressant [3], antibacterial [4], antiviral [5], antiinflammatory [6], antitubercular [7], antifungal [8], cytotoxic [9, 10], and some others.

There are various tricyclic pyrimidopyridopyrimidines including pyrimido[5',4':5,6]pyrido[2,3-*d*]pyrimidines (**1**), pyrimido[4',5':5,6]pyrido[3,2-*d*]pyrimidines (**2**), and pyrimido[4',5':5,6]pyrido[2,3-*d*]pyrimidines (**3**) (see figure), that are constructed from two fused pyridopyrimidine and pyrimidine rings. Among them, pyrimido[5',4':5,6]pyrido[2,3-*d*]pyrimidines **1**, which can be named as pyrido[2,3-*d*:6,5-*d'*]dipyrimidines, are of most interest. Derivatives of **1** demonstrated antibacterial [11], antiviral [5] and NAD-type redox catalytic [12] activities. Some of them act as inhibitors of α-glucosidase and α-amylase [13]. Solid-state ribbons have also been described from the self-assembly of an amine derivative of **1**, which demonstrates the potential of these heterocyclic compounds in supramolecular synthesis applications [14].

Several methods have been reported for the synthesis of pyrimido[5',4':5,6]pyrido[2,3-*d*]pyrimidines such as reaction of 1,3-diarylbarbituric acid with pyrrole-2-carbaldehyde and liquor ammonia [11], reaction of 6-amino-1,3-dimethyluracil with aromatic aldehydes followed by oxidation by the air and catalyzed by *p*-TSA in [bmim]Br media [15], SBA-15-SO_3H catalyzed reaction of 6-aminouracil with different aldehydes under solvent-free conditions [16], microwave irradiation of 6-amino-1-methyluracil and aromatic aldehyde in the presence of acetic acid [17], reaction of 2,6-diaminopyrimidin-4(3*H*)-one with isatins in the presence of *p*-TSA [18], reaction of 6-amino-2-butylthiopyrimidin-4(3*H*)-one with aryl al-

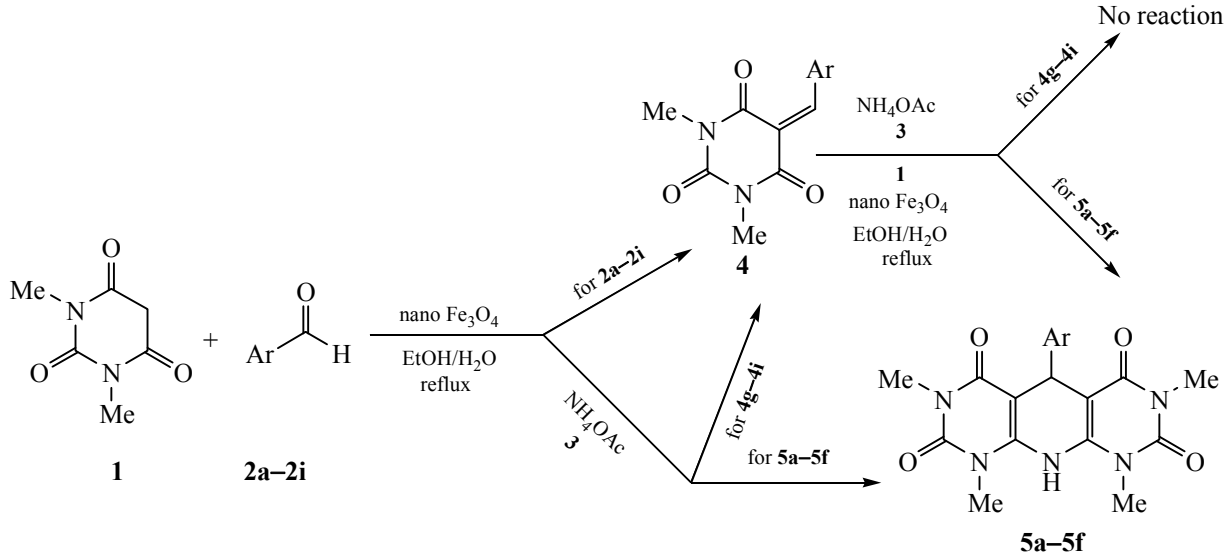


1 **2** **3**

Structures of various pyrimidopyridopyrimidines.

[1] The text was submitted by the authors in English.

Scheme 1.



Ar = C_6H_5 (**a**), Ar = 4-ClC_6H_4 (**b**), Ar = 2-ClC_6H_4 (**c**), Ar = 4-FC_6H_4 (**d**), Ar = 4-$O_2NC_6H_4$ (**e**), Ar = 4-pyridyl (**f**), Ar = 4-MeC_6H_4 (**g**), Ar = 4-$MeOC_6H_4$ (**h**), Ar = 4-HOC_6H_4 (**i**).

dehydes using sulfonic acid supported on hydroxyapatite-encapsulated-γ-Fe_2O_3 [19], and by multistep syntheses [20].

Considering the above facts and due to our interest in the synthesis of new heterocyclic compounds with potential biological activities [21–28], we report here efficient synthetic approach to some new pyrimido[5',4':5,6]pyrido[2,3-*d*]pyrimidine derivatives by one-pot cyclocondensation reaction of 1,3-dimethylbarbituric acid with aromatic aldehydes and ammonium acetate using nano Fe_3O_4 as a catalyst (Scheme 1).

RESULTS AND DISCUSSIONS

Treatment of 1,3-dimethylbarbituric acid **1** (2 mmol) and 4-chlorobenzaldehyde **2b** (1 mmol) with ammonium acetate **3** (1.2 mmol) in the presence of Fe_3O_4 nanoparticles in aqueous ethanol, followed by refluxing of the mixture gave the corresponding pyrimido[5',4':5,6]pyrido[2,3-*d*]pyrimidine **5b** in high yield (Table 1). A mixture EtOH–H_2O (50 : 50) proved to be much better media than the others tested including EtOH, H_2O, $CHCl_3$, and solvent-free conditions. The latter solvents supported formation of the target product in moderate yield within long reaction time. The optimal amount of the catalyst was determined to be 0.75 g (Table 1, entry 11). The higher amount of the catalyst had no significant effect on the yield and reaction time. To demonstrate the efficiency of the catalyst of choice, a blank reaction was carried out in the absence of Fe_3O_4 nanoparticles. Refluxing in EtOH, H_2O, or EtOH–H_2O lasted for 180 min and led to moderate yields of **5b** (Table 1).

The scope of the reaction was further studied with different aromatic aldehydes under the optimized reaction conditions (Table 2). Aromatic aldehydes substituted with electron-withdrawing group or none **2a–2f** gave pyrimido[5',4':5,6]pyrido[2,3-*d*]-pyrimidines **5a–5f** in high yields within short reaction time. The reaction with electron-rich aldehydes did not give the expected tricyclic products even upon prolonged reaction time, instead, the Knoevenagel condensation reaction occurred and the products **4g–4i** were isolated in high yields (Table 2). Formation of the products **5a–5f** probably proceed via Knoevenagel intermediates **4a–4f** that reacted with second equivalent of 1,3-dimethylbarbituric acid **1** and ammonium acetate **3**. Under these conditions, attempts to isolate the intermediates **4a–4f** failed. To prove this hypothesis, 1,3-dimethylbarbituric acid **1** (1 mmol) was allowed to interact with aromatic aldehydes **2a–2i** in the absence of ammonium acetate **3** under the same reaction conditions. In that case Knoevenagel products **4a–4i** were isolated. Finally, the reaction of **4a–4f** with 1,3-dimethylbarbituric acid **1** and ammonium acetate **3**, conducted under similar conditions, gave **5a–5f** in high yields.

Table 1. Optimization of conditions for synthesis of **5b** catalyzed by Fe_3O_4 nanoparticles[a]

Entry no.	Catalyst, g	Solvent	*T*, °C	Time, min	Yield[b], %
1	–	EtOH	Reflux	180	45
2	–	H_2O	Reflux	180	47
3	–	EtOH–H_2O	Reflux	180	52
4	0.050	EtOH	Reflux	180	70
5	0.075	EtOH	Reflux	120	76
6	0.050	H_2O	Reflux	120	73
7	0.075	H_2O	Reflux	90	78
8	0.075	$CHCl_3$	Reflux	240	68
9	0.025	EtOH–H_2O	Reflux	105	78
10	0.050	EtOH–H_2O	Reflux	40	80
11	0.075	EtOH–H_2O	Reflux	30	89
12	0.075	EtOH–H_2O	50	70	72
13	0.075	EtOH–H_2O	Room temperature	120	68
14	0.100	EtOH–H_2O	Reflux	30	87
15	0.075	–	100	50	78
16	0.075	–	120	40	80

[a] Reaction conditions: 1,3-dimethylbarbituric acid **1** (2 mmol), 4-chlorobenzaldehyde **2b** (1 mmol) and ammonium acetate **3** (1.2 mmol).
[b] Isolated yields.

Table 2. Synthesis of pyrimido[5',4':5,6]pyrido[2,3-*d*]pyrimidines **5a–5f** and Knoevenagel products **4g–4i** using Fe_3O_4 nanoparticles as the catalyst[a]

Comp. no.	Ar	Time, min	Yield[b], %	mp, ºC	
				found	calculated
4g	4-MeC_6H_4	30	93	144–146	142–142.5 [29]
4h	4-$MeOC_6H_4$	25	95	151–153	149–151 [30]
4i	4-HOC_6H_4	20	91	291–293	293–295 [30]
5a	C_6H_5	35	80	186–188	New
5b	4-ClC_6H_4	30	89	187–189	New
5c	2-ClC_6H_4	35	80	226–228	New
5d	4-FC_6H_4	20	85	159–161	New
5e	4-$O_2NC_6H_4$	20	91	210–212	New
5f	4-Pyridyl	25	82	245–246	New

[a] Reaction conditions: 1,3-dimethylbarbituric acid **1** (2 mmol), an aromatic aldehyde **2a–2i** (1 mmol), ammonium acetate **3** (1.2 mmol), Fe_3O_4 nanoparticles (0.075 g), EtOH/H_2O (5 mL, 50 : 50), reflux.
[b] Isolated yields.

Structures of new products **5a–5f** were elucidated on the basis of spectral data. For example, ^{1}H NMR spectrum of **5e** in DMSO-d_6 and D_2O demonstrated a singlet at 3.15 ppm assigned to four methyl groups. A singlet at 6.25 ppm (methine group), as well as two doublets in the aromatic region assigned to *para*-substituted aromatic ring indicated formation of the compound **5e**. IR spectrum of **5e** demonstrated a band at 3425 cm^{-1} (NH) and a strong band at 1693 cm^{-1} (C=O). The ^{13}C NMR spectrum was consistent with the assigned structure.

Recyclability and reusability of Fe_3O_4 nanoparticles were studied in the synthesis of compound **5b** under optimized conditions. Fe_3O_4 Nanoparticles were readily recovered from the reaction mixture using the procedure outlined in the experimental section. The catalyst could be used at least four times with only a slight reduction in activity. Retention of the structure of the catalyst was confirmed by comparing FT-IR spectra of the recovered catalyst after the fourth run with that of the fresh catalyst used in the model reaction. The spectra were almost identical.

EXPERIMENTAL

Nanosize Fe_3O_4 was purchased from Tecnan Spanish company. All other chemicals were purchased from Merck and Aldrich and used without purification. Melting points were measured on a Stuart SMP3 melting point apparatus. IR spectra were recorded on a Tensor 27 Bruker spectrophotometer in KBr disks. ^{1}H and ^{13}C NMR spectra were measured on a Bruker 300 spectrometer at 300 and 75 MHz.

General procedure for the synthesis of pyrimido-[5',4':5,6]pyrido[2,3-*d*]pyrimidine 5a–5f and Knoevenagel products 4g–4i catalyzed by Fe_3O_4 nanoparticles. A mixture of 1,3-dimethylbarbituric

acid **1** (2 mmol) with an aromatic aldehyde **2a–2i** (1 mmol), ammonium acetate **3** (1.2 mmol) and Fe_3O_4 nanoparticles (0.075 g) in EtOH–H_2O (2.5 : 2.5 mL) was refluxed for 20–35 min. The reaction was monitored by TLC. Upon completion of the process, the catalyst was separated using an external magnet. The reaction mixture was cooled down to room temperature. The crude product was filtered off and crystallized from ethanol to give compounds **5a–5f** and **4g–4i** in high yields. The isolated catalyst was washed with hot ethanol, dried at 60°C under vacuum for 1 h and reused in the following similar experiments. The synthesized earlier products **4g–4i** were characterized by IR and ^{1}H NMR spectra and their melting points were close to those of authentic samples [29, 30].

1,3-Dimethyl-5-(4-methylbenzylidene)pyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione (4g). IR spectrum, ν, cm^{-1}: 1664 (C=O). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 2.36 s (3H, CH_3), 3.30 s (3H, NCH_3), 3.34 s (3H, NCH_3), 7.20 d (J = 7.8 Hz, 2H, H_{arom}), 7.98 d (J = 7.8 Hz, 2H, H_{arom}), 8.47 s (1H, =CH).

5-(4-Methoxybenzylidene)-1,3-dimethylpyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione (4h). IR spectrum, ν_{max}, cm^{-1}: 1665 (C=O). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 3.39 s (3H, NCH_3), 3.41 s (3H, NCH_3), 3.91 s (3H, OCH_3), 6.98 d (J = 8.7 Hz, 2H, H_{arom}), 8.33 d (J = 8.7 Hz, 2H, H_{arom}), 8.50 s (1H, =CH).

1,3-Dimethyl-5-(4-hydroxybenzylidene)pyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione (4i). IR spectrum, ν, cm^{-1}: 3199 (OH), 1642 (C=O). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 3.20 s (3H, NCH_3), 3.22 s (3H, NCH_3), 6.89 d (J = 8.7 Hz, 2H, H_{arom}), 8.28 d (J = 8.7 Hz, 2H, H_{arom}), 8.32 s (1H, =CH), 10.85 s (1H, OH).

1,3,7,9-Tetramethyl-5-phenyl-5,10-dihydropyrimido[5',4':5,6]pyrido[2,3-*d*]pyrimidine-2,4,6,8-(1*H*,3*H*,7*H*,9*H*)-tetraone (5a). IR spectrum, ν, cm^{-1}: 3545 (NH), 1663 (C=O). ^{1}H NMR spectrum (DMSO-d_6–D_2O), δ, ppm: 3.15 s (12H, $4NCH_3$), 6.14 s (1H, CH), 7.01–7.17 m (5H, H_{arom}). ^{13}C NMR spectrum (DMSO-d_6–D_2O), δ, ppm: 28.4, 33.8, 91.5, 124.9, 127.2, 128.0, 144.6, 151.9, 163.2.

5-(4-Chlorophenyl)-1,3,7,9-tetramethyl-5,10-dihydropyrimido[5',4':5,6]pyrido[2,3-*d*]pyrimidine-2,4,6,8(1*H*,3*H*,7*H*,9*H*)-tetraone (5b). IR spectrum, ν, cm^{-1}: 3541 (NH), 1630 (C=O). ^{1}H NMR spectrum (DMSO-d_6–D_2O), δ, ppm: 3.16 s (12H, $4NCH_3$), 6.18 s (1H, CH), 7.05 d (J = 7.2 Hz, 2H, H_{arom}), 7.14 d (J = 7.2 Hz, 2H, H_{arom}). ^{13}C NMR spectrum (DMSO-d_6–D_2O), δ, ppm: 28.5, 34.4, 91.5, 125.1, 127.1, 128.0, 144.2, 152.0, 163.5.

5-(2-Chlorophenyl)-1,3,7,9-tetramethyl-5,10-dihydropyrimido[5',4':5,6]pyrido[2,3-*d*]pyrimidine-2,4,6,8(1*H*,3*H*,7*H*,9*H*)-tetraone (5c). IR spectrum, ν, cm^{-1}: 3434 (NH), 1680 (C=O). ^{1}H NMR spectrum (DMSO-d_6–D_2O), δ, ppm: 3.12 s (12H, $4NCH_3$), 5.96 s (1H, CH), 7.06–7.35 m (4H, H_{arom}). ^{13}C NMR spectrum (DMSO-d_6–D_2O), δ, ppm: 28.3, 34.2, 89.6, 126.2, 127.1, 129.6, 130.9, 133.1, 142.5, 151.9, 162.8.

5-(4-Fluorophenyl)-1,3,7,9-tetramethyl-5,10-dihydropyrimido[5',4':5,6]pyrido[2,3-*d*]pyrimidine-2,4,6,8(1*H*,3*H*,7*H*,9*H*)-tetraone (5d). IR spectrum, ν, cm^{-1}: 3489 (NH), 1670 (C=O). ^{1}H NMR spectrum (DMSO-d_6–D_2O), δ, ppm: 3.14 s (12H, $4NCH_3$), 6.07 s (1H, CH), 6.89–7.08 m (4H, H_{arom}). ^{13}C NMR spectrum (DMSO-d_6–D_2O), δ, ppm: 28.4, 33.4, 91.4, 114.6, 128.8, 140.4, 151.8, 158.9, 162.1, 166.4.

1,3,7,9-Tetramethyl-5-(4-nitrophenyl)-5,10-dihydropyrimido[5',4':5,6]pyrido[2,3-*d*]pyrimidine-2,4,6,8(1*H*,3*H*,7*H*,9*H*)-tetraone (5e). IR spectrum, ν, cm^{-1}: 3425 (NH), 1693 (C=O). ^{1}H NMR spectrum (DMSO-d_6–D_2O), δ, ppm: 3.15 s (12H, $4NCH_3$), 6.25 s (1H, CH), 7.30 d (J = 7.8 Hz, 2H, H_{arom}), 8.03 d (J = 7.8 Hz, 2H, H_{arom}). ^{13}C NMR spectrum (DMSO-d_6–D_2O), δ, ppm: 28.5, 34.6, 91.0, 123.4, 128.4, 145.5, 151.8, 154.2, 163.1.

1,3,7,9-Tetramethyl-5-(4-pyridyl)-5,10-dihydropyrimido[5',4':5,6]pyrido[2,3-*d*]pyrimidine-2,4,6,8-(1*H*,3*H*,7*H*,9*H*)-tetraone (5f). IR spectrum, ν, cm^{-1}: 3481 (NH), 1690 (C=O). ^{1}H NMR spectrum (DMSO-d_6–D_2O), δ, ppm: 3.15 s (12H, $4NCH_3$), 6.38 s (1H, CH), 7.77 d (J = 6.6 Hz, 2H, H_{arom}), 8.63 d (J = 6.6 Hz, 2H, H_{arom}). ^{13}C NMR spectrum (DMSO-d_6–D_2O), δ, ppm: 28.5, 35.9, 89.6, 125.7, 141.0, 151.8, 163.0, 167.9.

CONCLUSIONS

Fe_3O_4 nanoparticles were used as the efficient catalyst in the synthesis of new pyrimido[5',4':5,6]-pyrido[2,3-*d*]pyrimidine derivatives by one-pot reaction of 1,3-dimethylbarbituric acid, aromatic aldehydes substituted with electron-withdrawing group or none, and ammonium acetate under refluxing in EtOH–H_2O leading to high yields within short reaction time. Under the same conditions the use of an aromatic aldehyde substituted with electron-donating group gave the corresponding Knoevenagel products. Fe_3O_4 Nanoparticles can be magnetically separated from the

reaction mixture and recycled without significant loss of catalytic activity.

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