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A convenient green protocol for one-pot three-component synthesis of 2-amino-4*H*-chromene derivatives catalyzed by ilmenite ($FeTiO_3$) as an efficient and reusable catalyst in water

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A highly efficient and simple procedure for the synthesis of 2-amino-*4H*-chromene derivatives *via* one-pot, three-component reaction from various aldehydes, malononitrile, and *α*- or *β*-naphthol in the presence of a catalytic amount of ilmenite ($FeTiO_3$) is described. The advantages of this novel protocol include good yield, short reaction time, simple work-up, low toxicity and environmentally friendly catalyst. According to our results, $FeTiO_3$ (10 mol %) can be used as a catalyst for the synthesis of 2-amino-4*H*-chromene derivatives in water solvent under reflux conditions.

Key words: $FeTiO_3$, ilmenite, three-component reaction, 2-amino-*4H*-chromene derivatives, malononitrile, aldehydes.

INTRODUCTION

Multicomponent coupling reactions (MCRs) are emerging as useful tools for the carbon–carbon and carbon–heteroatom bond-forming reactions and for the synthesis of small drug-like molecules with several degrees of structural diversity [1]. One-pot multicomponent reactions provide the possibility of directly synthesizing a complex molecule without needing to isolate the intermediates, which has aroused researchers to design such reactions using different catalysts [2,3].

Ilmenite ($FeTiO_3$) is one of the most common minerals in the earth crust. $FeTiO_3$ is an interesting wide bandgap (2.58–2.9 eV) antiferromagnetic semiconductor with potential applications in spintronics, optoelectronics, high temperature integrated circuits, chemical catalysts, photo catalysts, etc. [4].

Aminochromenes and their derivatives are of considerable interest as they possess a wide range of biological properties [5], such as spasmolytic, diuretic, anticoagulant, anticancer and antianaphylactic activities [6]. In addition, they can be used as cognitive enhancers for the treatment of neurodegenerative diseases, including Alzheimer’s disease, amyotrophic lateral sclerosis, Huntington’s disease, Parkinson’s disease, AIDS associated dementia and Down’s syndrome, as well as for the treatment of schizophrenia and myoclonus [7].

Aminochromenes have been prepared by heating a mixture of malononitrile, aldehydes and activated phenols or naphthols in refluxing DMF or acetonitrile in the presence of hazardous organic bases such as piperidine and triethylamine [8, 9]. In the literature, a lot of synthetic methods for the synthesis of 2 amino-*4H*-chromene derivatives have been reported so far [10-26], Most of these methods are associated with shortcomings like the use of costly, harmful and non-reusable catalysts, tedious separation procedures, long reaction time, tedious work up and use of toxic organic solvents.

To the best of our knowledge, the use of water as a clean solvent in the presence of ilmenite as a reusable catalyst for the synthesis of 2-amino-*4H*-chromene derivatives has not been reported.

In light of the above reports, and in continuation of our ongoing efforts to develop new methods in organic synthesis [27-38], herein we report our results for the synthesis of 2-amino-*4H*-chromene derivatives *via* a one-pot three-component reaction of various aldehydes **(2)**, malononitrile **(3)** and *α*-, or *β*-naphthol **(1)** in the presence of $FeTiO_3$ as an efficient and reusable catalyst in water under reflux conditions (Scheme 1).

RESULTS AND DISCUSSION

As a part of a continuing effort in our laboratory towards the development of new methods in organic synthesis [27-38], in the present paper, a convenient and green protocol for the one-pot three-component synthesis of 2-amino-4*H*-

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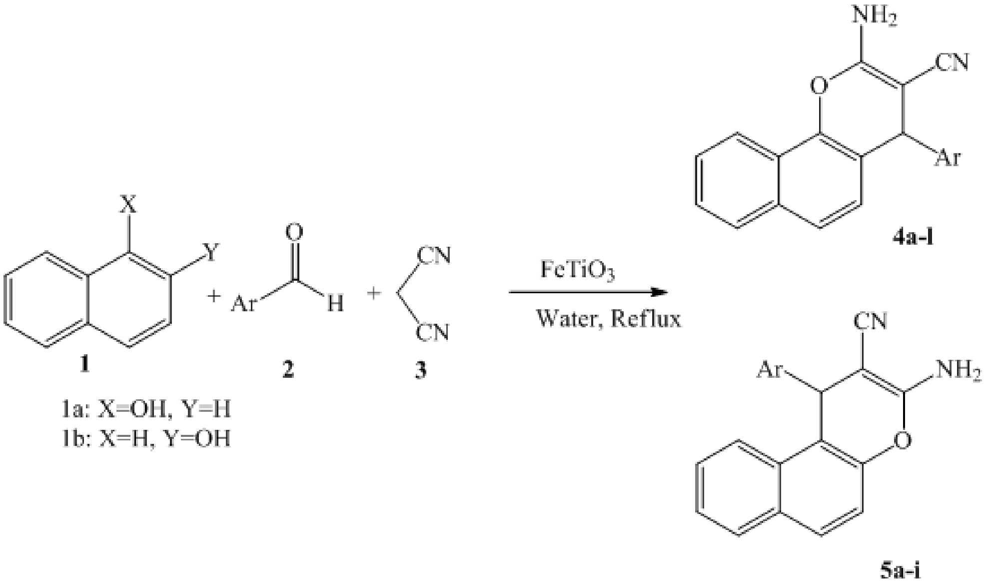
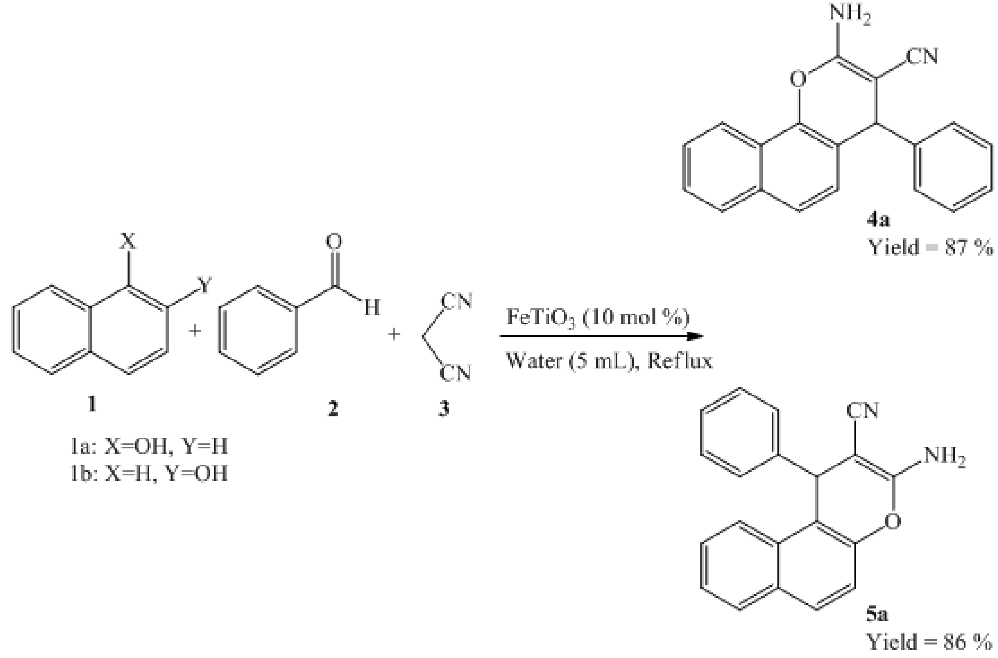
chromene derivatives catalyzed by ilmenite as a reusable catalyst in water is described.

To optimize the reaction conditions, the condensation reaction of *α*- or *β*-naphtol **(1)**, benzaldehyde **(2)**, and malononitrile **(3)** was tested as the model reaction using different amounts of $FeTiO_3$ at 40 or 100 ˚C under reflux conditions in the presence of various solvents. As shown in Table 1, $FeTiO_3$ (10 mol %) as a catalyst under reflux conditions afforded 2-amino-*4H*-chromene derivatives in 30 min with (**4a = 87 %,** and **5a = 86 %**) of yield in water (Table 1, Scheme 2).

In the next section, in order to assess the efficiency and the scope of the catalyst for the preparation of 2-amino-4*H*-chromene derivatives, the reaction of *α*- or *β*-naphthol with various aldehydes and malononitrile was examined in the presence of 10 mol% of $FeTiO_3$ as a catalyst. The corresponding results are displayed in Table 2.



Scheme 1. Synthesis of chromene derivatives from various aromatic aldehydes, malononitrile and naphthols in water.

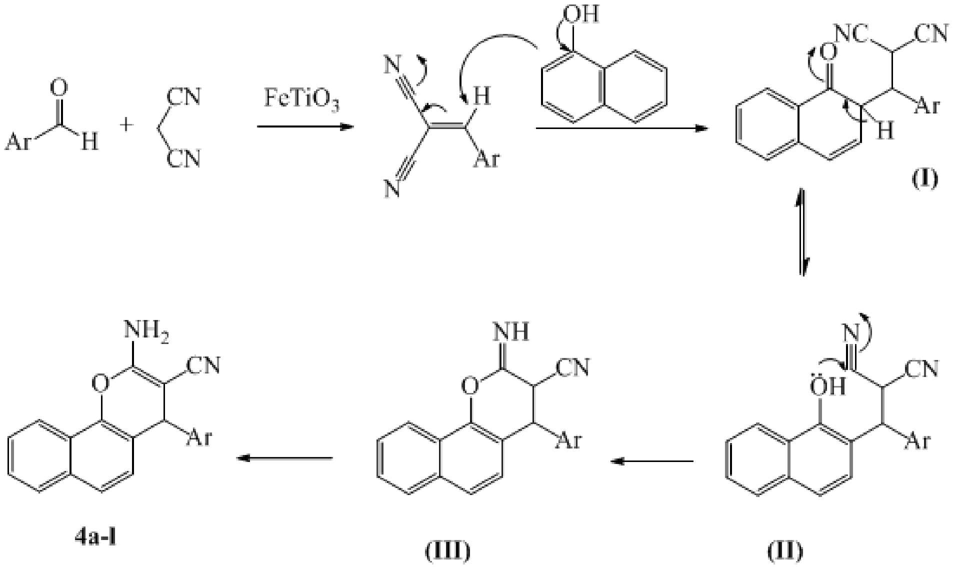


Scheme 2. Preparation of chromene derivatives from benzaldehyde, malononitrile and naphthols in the presence of $FeTiO_3$ as a catalyst.

Table 1. Optimization of conditions for the synthesis of 2-amino-4*H*-chromene derivatives from 4-chlorobenzaldehyde, malononitrile, and α- or β-naphthol in the presence of different amounts of $FeTiO_3$ as a catalyst at different temperatures.

Entry	Solvent	Catalyst (mol %)	T (˚C)	Time (min)	Yield (%) [a,b]	
					4a	**5a**
1	Ethanol	10	60	35	75	73
2	Methanol	10	60	30	82	81
3	Dichloromethane	10	40	60	52	50
4	Acetonitrile	10	60	40	82	81
5	Water	15	Reflux	30	85	84
			90		79	78
			80		67	63
6	Water	10	Reflux	30	87	86
			90		81	80
			80		70	67
7	Water	5	Reflux	30	72	67
			90		64	62
			80		52	45
8	Water	Catalyst-free	Reflux	6 h	40	36
9	Solvent-free	10	100	60	72	70

[a]Reaction conditions: α-naphthol (1 mmol), or β-naphthol (1 mmol), benzaldehyde (1 mmol), malononitrile (1 mmol) and solvent (5 mL) in the presence of $FeTiO_3$ as a catalyst. [b] Isolated yield



Scheme 3 A proposed mechanism for the preparation of 2-amino-4*H*-chromene derivatives from various aldehydes, malononitrile and α-naphthol.

Table 2. Synthesis of 2-amino-4*H*-chromene derivatives using ilmenite as a catalyst [a]

Entry	Ar	Phenol	Product	Time/min	Yield /%[b]	Mp/°C	Lit [c] Mp/°C
1	C_6H_5	*α*- Naphthol	**4a**	30	87	209-211	210-211 [39]
2	4-ClC_6H_4	*α*- Naphthol	**4b**	35	91	231-232	231-232 [42]
3	2-ClC_6H_4	*α*- Naphthol	**4c**	35	90	237-238	236-237 [11]
4	2,4-$Cl_2C_6H_3$	*α*- Naphthol	**4d**	35	91	220-222	222-224 [46]
5	4-$NO_2C_6H_4$	*α*- Naphthol	**4e**	37	91	230-232	231-234 [18]
6	3-$NO_2C_6H_4$	*α*- Naphthol	**4f**	40	89	208-210	208-211 [24]
7	4-FC_6H_4	*α*- Naphthol	**4g**	40	90	228-230	229-231 [42]
8	4-$MeOC_6H_4$	*α*- Naphthol	**4h**	30	85	194-195	195-196 [10]
9	3-OHC_6H_4	*α*- Naphthol	**4i**	35	83	230-232	228-232 [46]
10	4-BrC_6H_4	*α*- Naphthol	**4j**	30	90	243-244	241-243 [41]
11	4-MeC_6H_4	*α*- Naphthol	**4k**	30	84	205-206	205-206 [39]
12	4-HOC_6H_4	*α*- Naphthol	**4l**	35	85	244-246	245-247 [43]
13	C_6H_5	*β*- Naphthol	**5a**	30	86	279-280	278-280 [42]
14	4-ClC_6H_4	*β*- Naphthol	**5b**	35	90	209-210	208-210 [42]
15	2-ClC_6H_4	*β*- Naphthol	**5c**	35	91	237-238	274-276 [11]
16	2,4-$Cl_2C_6H_3$	*β*- Naphthol	**5d**	35	90	220-222	219-222 [47]
17	4-$NO_2C_6H_4$	*β*- Naphthol	**5e**	40	91	230-232	188-189 [42]
18	3-$NO_2C_6H_4$	*β*- Naphthol	**5f**	50	94	211-213	210-212 [41]
19	4-FC_6H_4	*β*- Naphthol	**5g**	55	94	231-232	232-233 [45]
20	4-$MeOC_6H_4$	*β*- Naphthol	**5h**	30	83	190-192	191-193 [42]
21	4-MeC_6H_4	*β*- Naphthol	**5i**	35	82	205-206	270-272 [40]

[a] Reaction conditions: *α*- or *β*-naphthol (1 mmol), aldehyde (1 mmol), malononitrile (1 mmol) and water (5 mL) in the presence of $FeTiO_3$ as a catalyst under reflux conditions; [b] Isolated yield; [c] All known compounds were characterized by comparing their spectral (FT-IR) and physical data with those reported.

As shown in Table 2, the presence of electron-donating groups on the aromatic aldehydes resulted in low yields of the corresponding products and the reaction was sluggish. The presence of electron-withdrawing groups, however, resulted in shorter reaction times and higher yields. Using *α*-naphthol or *β*-naphthol had no effect on the reaction time or yield of the corresponding products.

The proposed mechanism for the preparation of 2-amino-3-cyano-4-phenyl-4*H*-benzo[*h*]chromene from benzaldehyde, malononitrile, and *α*-naphthol in the presence of $FeTiO_3$ as a catalyst is depicted in Scheme 3. According to the literature [10-19], benzylidene malononitrile, containing an electron-poor C=C double bond, is formed quantitatively by Knoevenagel addition of malononitrile to the aromatic aldehyde in the presence of $FeTiO_3$ as a catalyst. Ortho C-alkylation of the electrophilic C=C double bond by *α*-naphthol gives the intermediate (I). The latter converts *via* tautomerization to intermediate (II) which is then cyclized by nucleophilic attack of an OH group on the cyano (CN) moiety to give intermediate (III). Subsequent tautomerization produced the 2-amino-3-cyano-4-phenyl-4*H*-benzo[*h*]chromene (**4a**).

Reusability is one of the important properties of this catalyst. In order to check the reusability of the catalyst, the reaction of benzaldehyde, malononitrile and *α*-naphtol was studied under similar conditions in the presence of 10 mol% of $FeTiO_3$ (Table 2, Entry 1). After the completion of the reaction, the catalyst was separated by an external magnet, and after several washings with acetone and distilled water the activated catalyst was reused to assess its efficiency in subsequent reaction cycles without any appreciable loss of its activity.

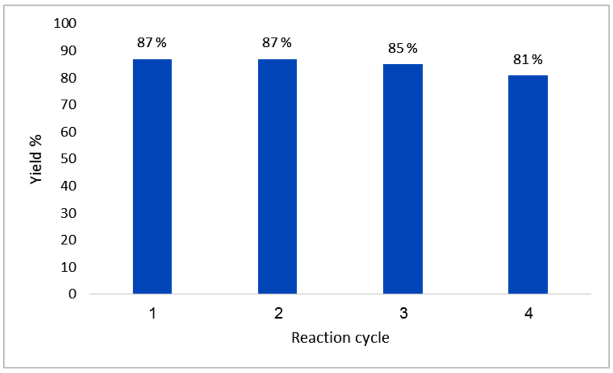


Fig. 1. Recycling of $FeTiO_3$ as a catalyst investigated for the synthesis of 2-amino-4*H*-chromene derivatives by the reaction of benzaldehyde, malononitrile and *α*-naphthol as a model reaction.

CONCLUSIONS

To summarize, herein, an efficient and eco-compatible method for the synthesis of 2-amino-4*H*-chromene derivatives *via* a one-pot three-component reaction of *α*- or *β*-naphthol with malononitrile and aromatic aldehydes in the presence of $FeTiO_3$ as a catalyst was developed. The operational simplicity, easy work-up, short reaction time, together with the use of non-toxic and commercially available catalyst are the remarkable features of the procedure.

EXPERIMENTAL

Starting materials and solvents were obtained from Merck (Germany) and Fluka (Switzerland) and were used without further purification. Melting points were measured on an Electrothermal 9100 apparatus and are uncorrected. IR spectra were measured on a Jasco FT-IR 6300 spectrometer. All obtained products are known compounds and were identified by comparing their physical and spectral data (FT-IR) with those reported in the literature. The progress of reactions was monitored by thin layer chromatography (TLC).

Synthesis of 2-amino-4H-chromene derivatives catalyzed by $FeTiO_3$. General procedure: A mixture of aldehyde (1 mmol), malononitrile (1 mmol), *α*- or *β*-naphthol (1 mmol) and $FeTiO_3$ (10 mol %) was added to a test tube and was heated in an oil bath under reflux conditions in water (5 mL) for the appropriate time (Table 2). After the completion of the reaction (monitored by TLC), the mixture was allowed to cool and the catalyst was removed by an external magnet. Then, the solvent was evaporated and the solid residue was recrystallized from ethanol (95%) to afford the pure 2-amino-4*H*-chromene derivatives.

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УДОБЕН ЗЕЛЕН ПРОТОКОЛ ЗА ЕДНО-СТАДИЙНА ТРИ-КОМПОНЕНТНА СИНТЕЗА НА 2-АМИНО-4*H*-ХРОМЕНОВИ ПРОИЗВОДНИ, КАТАЛИЗИРАНИ ОТ ИЛМЕНИТ($FeTiO_3$) КАТО ЕФЕКТИВЕН И МНОГОКРАТНО УПОТРЕБЯВАН КАТАЛИЗАТОР ВЪВ ВОДНА СРЕДА

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(Резюме)

Описана е високо-ефективна и проста процедура за синтезата на 2-амино-*4H*-хроменови производни чрез едностадийна трикомпонентна реакция от различни алдехиди, малононитрил и α- или β-нафтол в присъствие на катализатор от илменит ($FeTiO_3$). Предимствата на новия протокол включват добър добив, кратко време, просто изпълнение, ниска токсичност и екологично съобразен катализатор. Според нашите резултати $FeTiO_3$ (10 % мол.) може да се използва за тази синтеза във водна среда с рефлукс.