

FULL PAPER

Effective and convenient synthesis of 2-amino-4*H*-chromenes promoted by melamine as a recyclable organocatalyst

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This study focused on the applicability of melamine as a cheap and efficient organocatalyst in the acceleration of the synthesis of 4-aryl-4*H*-chromenes derivatives. Under the selected conditions, a wide range of aldehydes readily underwent condensation with activated phenols and malononitrile to afford the high to excellent yields. Also, organocatalyst was used for the synthesis of 2-amino-5,10-dioxo-5,10-dihydro-4*H*-benzo[*g*]chromenes by the reaction of aromatic aldehydes, malononitrile and 2-hydroxynaphthalene-1,4-dione in refluxing H₂O:EtOH. This method provides several advantages such as convenient work-up, mild reaction conditions, use of an inexpensive and readily available catalyst, short reaction times, high yields of the products and reusability of the catalyst.

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KEYWORDS

Melamine; multi-component reactions; reusable catalyst; aldehydes; chromenes.

Introduction

During recent years, substantial attention has been given to the power and potential of green chemistry as an efficacious methodology for developing modern technologies that diminish or omit the usage or production of hazardous substances [1]. In organic synthesis, green chemistry as a potent tool is employed to manufacture of various and complex molecular building blocks *via* multicomponent reactions (MCRs) with solvent-free synthesis or water as solvent. MCRs are one of the most momentous processes for the procurement desirable product in a single operation step from three or more starting materials. These types of synthetic systems have all traits that are attributed to a favorable synthesis, including efficiency, atom economy, cost and time minimizing, and easy purification. They

also enable the purposeful procurement of momentous and diverse molecular scaffolds. Therefore, by employing of novel multicomponent reactions as environmental benignant synthetic methods, the way to an extensive number of various and complex products is opened [2-5].

Today, the implementation of multicomponent reactions in green solvents, must also be considered because a solvent has a prominent role in every stage during the course of a reaction. Numerous of organic solvents are ecologically detrimental, and their utilization should be reduced as much as possible. No doubt, one of the more promising approaches from an environmental standpoint is solvent-free organic synthesis. Solvent-free reactions have plenty privileges such as reduced contamination, cost-effectiveness, and simplicity of manner and handling. These

factors are especially momentous in industry. Owing to these reasons, solvent-free synthesis not only prepares environmentally mild routes for organic synthesis, but also makes the processes economically practicable [6]. On the other hand, it is commonly believed that the best solvent from an ecological perspective is no solvent and if a solvent is required, water is better alternative compared to other solvents [7].

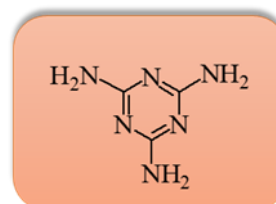
Water as a safe and green solvent renders a favorable medium for the synthesis of many of intricate molecules and compounds. Hitherto, the mixed solvent system has been extensively utilized for many years. Water and ethanol are green solvents, and the water-ethanol mixture for the reaction medium has acted extremely well in many cases [8].

One of the key aspects of green chemistry is the catalyst. Considering the growing need for energy efficiency and environmental concerns in chemical products, research attempts are being directed to the extension of green and efficacious catalysts and their usages in environmentally mild synthetic methods [9,10].

Organocatalysts are small organic molecules with low molecular weight, which can catalyze a chemical reaction in substoichiometric quantities. Organocatalysts exhibit various benefits such as facility of design or modification, cost-effectiveness,

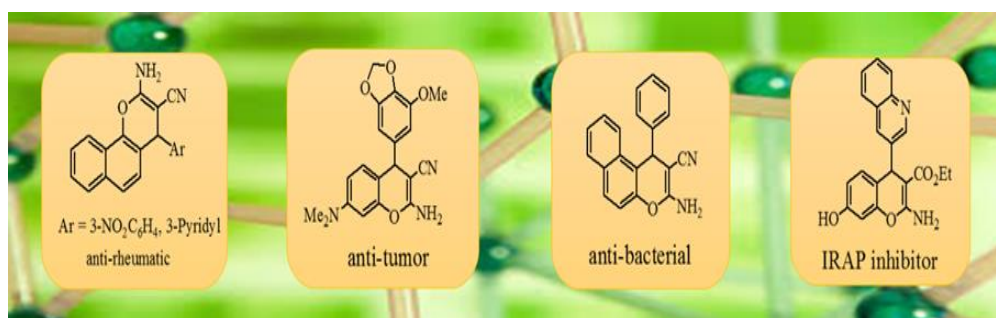
usually of less toxicity, robustness, operational simplicity, being less polluting and can be used in many different chemical reactions. These types of catalysts have an extremely important place in the pharmaceutical industry because they elude the formation of metallic waste in the ultimate product. Execution of chemical transformations in absence of transition-metal or organometallic catalysts places organocatalysis in the context of green chemistry [11,12].

Melamine (2,4,6-triamino-s-triazine) is a triamine chemical compound that possesses high nitrogen content (Scheme 1).



SCHEME 1 Chemical structure of melamine

Melamine has become an inseparable part of our daily lives and it is employed in making plastics, laminates, fire retardant, commercial filters, pigment, and in some medicines [13]. Among the heterocycles containing oxygen atoms, chromene-framed structures have a particular position owing to high abundance in nature and the immense spectrum of useful biological activities [14,15] (Scheme 2)



SCHEME 2 Some benzochromenes with various pharmacological activities

Benzo[g]chromenes as a momentous class of chromenes represent a spacious range of pharmacological properties such as

anticancer, antimalarial, anti-inflammatory, pesticide activities and occur in various natural products of medicinal significance,

such as lambertellin C, β -lapachone A, α -xiloidone B, and WS-5995A D. Moreover, some derivatives of these compounds are fluorescent heterocyclic and are valuable in a multitude of fields such as emitters for electroluminescence systems, in traditional textile and polymer fields, and fluorescent whitening agents [16].

Benzo[f]chromenes and benzo[h]chromenes are other extremely important substituted chromenes derivatives. Both classes of these chromenes derivatives are the privileged scaffold in the pharmaceutical chemistry because of their vast spectrum of fascinating biological properties including rheumatoid arthritis, dysentery, antibacterial, syphilis, remedy against gonorrhoea and cytotoxic activities [17]. Because of the above-mentioned features several strategies using different kinds of catalysts have been described for the synthesis of these types of compounds [18-24].

Looking into chromenes wide pharmacological applications and owing to advantages related to the multicomponent strategy, solvent-free reaction or water-ethanol solvent system as green media, we were interested in investigating a convenient and environmentally benign method in accordance with the principles of green chemistry to synthesize different derivatives of 2-amino-4H-chromene using melamine as an efficacious, environment-friendly and reusable catalyst. Melamine is so easily accessible and economically engrossing.

Experimental

General

All chemicals used here were purchased from Merck, Fluka and Aldrich companies and used without further purification. The yields related to the isolated products. The products were identified on the basis of their physical properties and were compared with those reported for authentic samples. The purity of

determination of the substrate and the reaction monitoring were carried out by thin-layer chromatography (TLC) on silica gel polygram SILG/UV 254 plates. Melting points were recorded on a Büchi B-545 apparatus in open capillary tubes. The ^1H NMR spectra were recorded with Bruker Avance 300 MHz instrument. The IR spectra were run on a VERTEX 70 Bruker company (Germany).

General procedure for the synthesis of 4-aryl-4H-chromenes

A mixture of malononitrile (1 mmol), aldehyde (1 mmol), activated phenols (α or β -naphthol and resorcinol) (1 mmol) and melamine (50 mg) was heated at 120 °C under solvent-free conditions for the suitable time. After the reaction was completed [monitored by TLC], water (10 mL) was added to the reaction mixture and then stirred for 5 min. The catalyst was easily removed by aqueous washing. The crude products which were insoluble in H_2O were isolated simply by filtration and recrystallization from ethanol. To continue evaporation, the solvent from the filtrate (H_2O) was accomplished to recover the catalyst.

Selected spectral data

3-Amino-1-phenyl-1H-benzo[f]chromene-2-carbonitrile (Table 2, entry 1):

IR (KBr): $\nu = 3425, 3345, 3150, 2195, 1650, 1575 \text{ cm}^{-1}$; ^1H NMR ($\text{DMSO}-d_6$), δ (ppm): 5.31 (s, 1H), 7.05 (s, 2H), 7.14-7.29 (m, 5H), 7.36-7.46 (m, 3H), 7.84-7.96 (m, 3H).

2-Amino-3-cyano-7-hydroxy-4-(4-methylphenyl)-4H-chromene (Table 2, entry 20):

IR (KBr): $\nu = 3411, 3365, 2190, 1617, 1510 \text{ cm}^{-1}$; ^1H NMR ($\text{DMSO}-d_6$), δ (ppm): 2.24 (s, 3H), 4.56 (s, 1H), 6.81 (s, 2H), 6.38-7.10 (m, 7H), 9.66 (s, 1H).

General procedure for the synthesis of 2-amino-5,10-dioxo-5,10-dihydro-4H-benzo[g]chromenes:

A mixture of malononitrile (1 mmol), aldehyde (1 mmol), 2-hydroxynaphthalene-1,4-dione (1 mmol) and melamine (30 mg) in refluxing H₂O: EtOH (2:1 mL) was stirred for the suitable time. When the reaction was completed [monitored by TLC], the mixture was filtered off and the crude product was washed with water (10 mL). The product was purified by recrystallization from ethanol. To continue, the solvent was evaporated from the filtrate to give the catalyst which can be used for the subsequent run.

Selected spectral data

2-Amino-3-cyano-4-(2-nitrophenyl)-5,10-dioxo-5,10-dihydro-4H-benzo[g]chromene (Table 4, entry 10):

IR (KBr): $\nu = 3429, 3338, 2207, 1666, 1631$ cm⁻¹; ¹H NMR (DMSO-*d*₆), δ (ppm): 5.94 (1H, s), 8.27–7.46 (10 H, m, Ar and NH₂).

Results and discussion

To investigate the deployment of effective methods for the synthesis of bioactive compounds [25-27], taking into consideration the structural features of melamine, we

decided to study its role in the progress of Knoevenagel-Michael addition reactions. This article is the report of the results of this investigation.

To provide the best conditions for performing this reaction, a set of experiments according to different factors affecting the reaction yield was studied for the reaction of β -naphthol (1 mmol), malononitrile (1 mmol) and 4-chlorobenzaldehyde (1 mmol), as the model reaction. Firstly, to choose the best reaction media, the effect of different solvents including H₂O, ethanol, *n*-hexane and acetonitrile was screened. According to the results, the best option for this reaction was the solvent-free condition which is in compliance with the goals of green chemistry. Moreover, we changed the amount of the catalyst. As it is obvious from Table 1, the reaction yield was affected crucially by the catalyst concentration. These results elucidated that 50 mg of melamine was an adequate amount to accelerate the reaction. In the subsequent stage, we investigated the effect of temperature on the completion of the desired reaction by carrying out the reaction at different temperatures under solvent-free conditions. The outcomes illustrated that when the model reaction was accomplished at 120 °C best yield in a short time was obtained (Table 1, Entry 3).

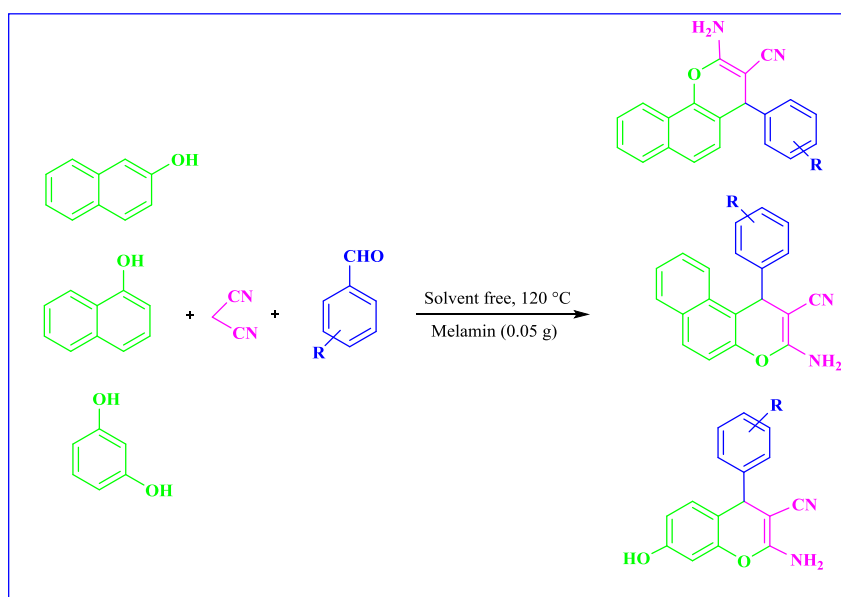
TABLE 1 Optimization of reaction conditions for the synthesis of 3-amino-1-(4-chlorophenyl)-1H-benzo[f]chromene-2-carbonitrile in the presence of melamine

Entry	Catalyst	Catalyst loading (mg)	Solvent	Temperature (°C)	Time (min)	Yielda (%)
1	None	None	None	60	240	Trace
2	Melamine	30	None	70	80	45
3	Melamine	50	None	120	20	90
4	Melamine	40	H ₂ O	80	120	35
5	Melamine	40	CH ₃ CN	reflux	90	40
6	Melamine	40	EtOH	reflux	90	60
7	Melamine	50	<i>n</i> -hexane	reflux	120	Trace

^aIsolated yield

To gain the optimized conditions, we evaluated this methodology by the one-pot three-component reaction of activated

phenols, malononitrile and diverse aldehydes, utilizing 50 mg melamine in the absence of solvent at 120 °C (Scheme 3).



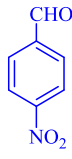
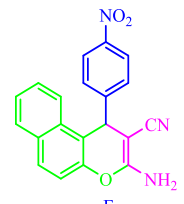
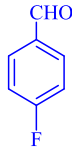
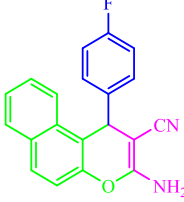
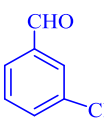
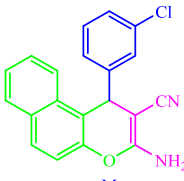
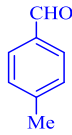
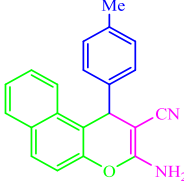
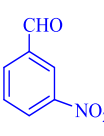
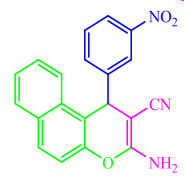
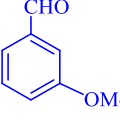
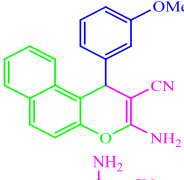
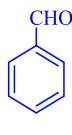
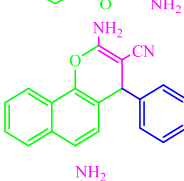
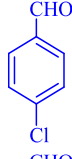
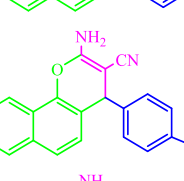
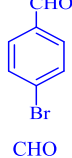
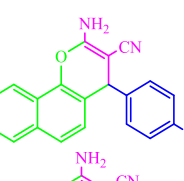
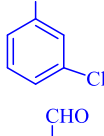
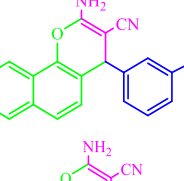
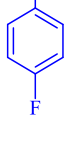
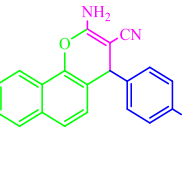
SCHEME 3 Melamine promoted one-pot synthesis of 4-aryl-4*H*-chromenes

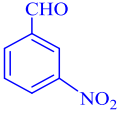
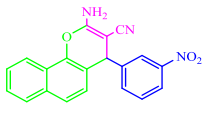
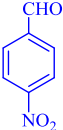
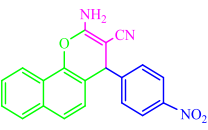
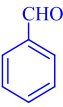
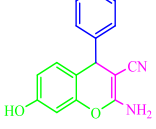
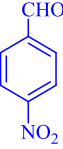
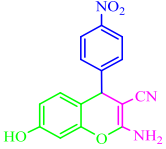
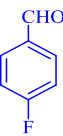
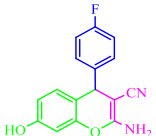
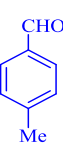
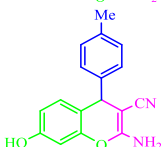
As shown in Table 2, the reaction was successfully performed with both electron-donating and electron-withdrawing groups on the aromatic aldehyde, and was not impressed by the substituents' position. The mentioned method displayed wide substrate scope and desired products were synthesized

without producing any by-products. Agreeably, we discovered that melamine as catalyst worked well for the preparation of a wide spectrum of 4-aryl-4*H*-chromenes and provided facile access to these compounds in a short period of times with good yields.

TABLE 2 Synthesis of 4-aryl-4*H*-chromenes catalyzed by melamine^a

Entry	RCHO	Product	Time (min)	Yield ^b (%)	Mp (°C)	
					Observed	Reported [Ref.]
1			30	90	286–288	286–288 [28]
2			20	90	212–213	210–212 [28]
3			25	95	236–237	238–240 [30]

4			30	87	184–185	185–186 [18]
5			25	90	234–235	233–234 [29]
6			25	90	236–238	236–238 [28]
7			30	85	270–272	268–270 [28]
8			30	85	233–235	233–235 [28]
9			25	93	256–259	256–258 [28]
10			30	90	209–210	210–211 [18]
11			20	85	232–234	235–237 [28]
12			30	90	233–234	234–236 [30]
13			25	90	220–221	220 [31]
14			25	90	233–234	234 [31]

15			30	90	211–212	212–214 [18]
16			25	84	238–240	239–241 [18]
17			15	95	230–232	230–232 [30]
18			10	93	211–212	210–211 [32]
19			10	90	192–193	190–192 [30]
20			15	90	187–189	186–188 [32]

^aReaction conditions: carbonyl compounds (1 mmol), activated phenols (1 mmol), malononitrile (1 mmol), melamine (50 mg), and solvent free conditions conducted at 120 °C.

^bIsolated yields.

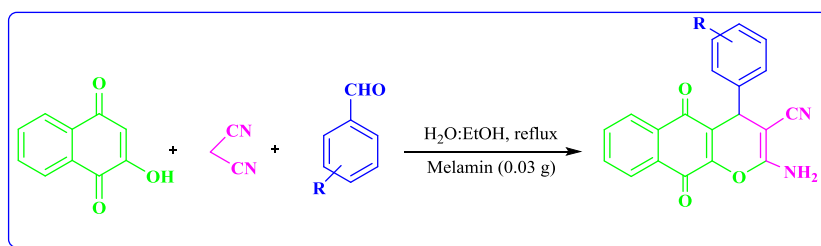
After the prosperous application of melamine for the synthesis of variant derivatives of 4-aryl-4*H*-chromenes and owing to the pharmaceutical values of 4*H*-benzopyrans, we decided to utilize this catalyst for the preparation of 2-amino-5,10-dioxo-5,10-dihydro-4*H*-benzo[*g*] chromenes. At the outset of our research, to attain the best reaction conditions, the reaction of 2-hydroxynaphthalene-1,4-dione (1 mmol), 4-chlorobenzaldehyde (1 mmol) and

malononitrile (1 mmol) was studied as a model reaction by use of melamine as catalyst. The results of the optimization reactions are summarized in Table 3. The obtained results illustrated that in this case the highest yield of the product can be acquired using 30 mg melamine as the catalyst when the reaction is carried out in refluxing H₂O: EtOH (2: 1) (Table 3, Entry 7) (Scheme 4).

TABLE 3 Optimization of reaction conditions for the synthesis of 2-amino-4- (4-chlorophenyl)-5,10-dihydro-5,10-dioxo-4*H*-benzo[*g*]chromene-3-carbonitrile in the presence of melamine

Entry	Catalyst	Catalyst loading (mg)	Solvent	Temperature (°C)	Time (min)	Yield ^a (%)
1	None	None	None	70	180	Trace
2	None	None	H ₂ O	80	120	Trace
3	Melamine	30	None	70	120	50
4	Melamine	40	H ₂ O	75	60	75
5	Melamine	40	EtOH	70	60	70
6	Melamine	40	H ₂ O:EtOH (1:1)	70	40	85
7	Melamine	30	H ₂ O:EtOH (2:1)	Reflux	20	95
8	Melamine	40	<i>n</i> -hexane	Reflux	70	Trace

^aIsolated yield

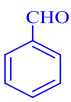
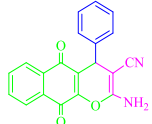
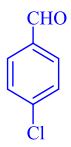
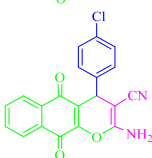
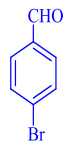
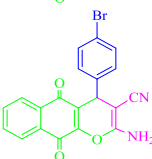
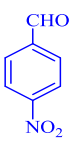
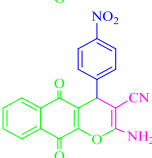
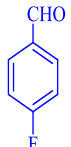
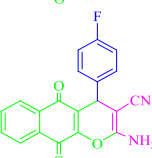
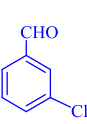
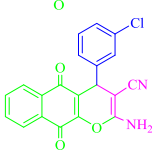



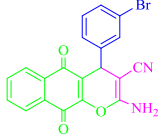

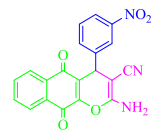

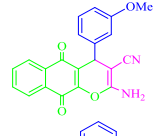

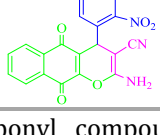
SCHEME 4 Melamine promoted one-pot synthesis of 2-amino-5,10-dioxo-5,10-dihydro-4*H*-benzo[g]chromenes

Encouraged by the notable results in hands and with the intention of exploring the generality of this reaction, various aromatic aldehydes bearing electron-donating and electron-withdrawing groups were used to react with 2-hydroxynaphthalene-1,4-dione and malononitrile under the defined

conditions. As it is clear from Table 4, this methodology proceeded well with aromatic aldehydes bearing halogen group and both electron-donating and electron-withdrawing groups under the optimized reaction conditions, and target compounds were synthesized in high yields.

TABLE 4 Synthesis of 2-amino-5,10-dioxo-5,10-dihydro-4*H*-benzo[g]chromenes catalyzed by melamine^a

Entry	RCHO	Product	Time (min)	Yield ^b (%)	Mp (°C)	
					Observed	Reported [Ref.]
1			25	95	259–260	260–261 [16]
2			20	95	250–252	250–251 [16]
3			25	90	252–253	252–253 [16]
4			25	95	234–236	232–234 [16]
5			20	90	283–285	286–288 [34]
6			20	90	253–254	252–254 [35]

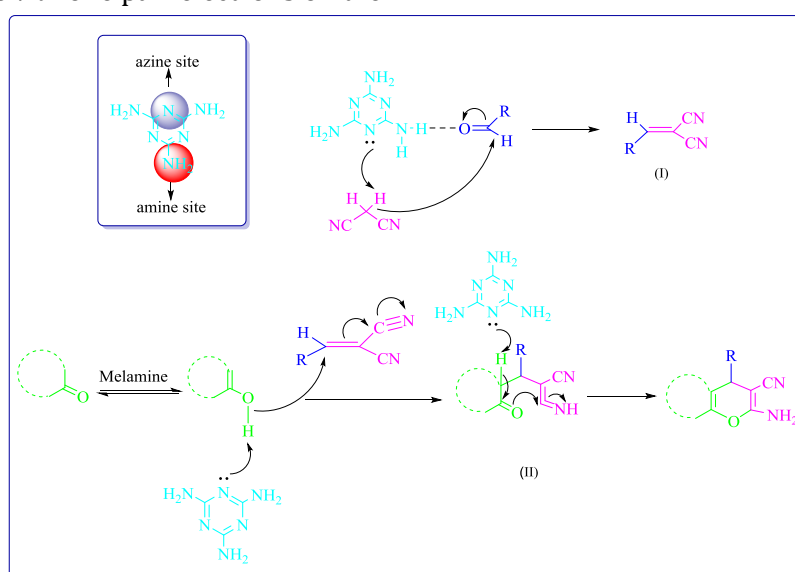
7			30	90	258–259	260–261 [16]
8			30	90	247–249	247–250 [16]
9			20	95	250–251	248–250 [16]
10			25	90	242–243	243 [16]

^aReaction conditions: carbonyl compounds (1 mmol), 2-hydroxynaphthalene-1,4-dione (1 mmol), malononitrile (1 mmol), melamine (30 mg), and H₂O: EtOH (2: 1); reactions conducted under reflux conditions.

^bIsolated yields.

Bearing in mind structural features of melamine, the following reaction pathway is suggested for the preparation of 2-amino-4*H*-chromenes (Scheme 5). In view of this mechanism, the increased reaction rate by melamine as catalyst was attributed to the double-activation of this compound. Melamine possesses three exocyclic primary amines, which can play a prominent role in activation of carbonyl substrates in the reaction by hydrogen bonding. On the other hand, melamine *via* lone pair electrons on the

N atoms of its triazine ring can take hydrogen atom away from the malononitrile. Accordingly, activated carbonyl groups and methylene anion can interact easily together to form the Knoevenagel intermediate (I). Activation of diverse phenols/2-hydroxynaphthalene-1,4-dione in reaction with (I) forms the intermediate (II). Eventually, the desired products are produced *via* a cyclization reaction which is also promoted by melamine.



SCHEME 5 The proposed pathway for the preparation of of 2-amino-4*H*-chromenes in the presence of melamine

One of the most momentous benefits from the sustainable chemistry outlook is the reusability of the catalyst owing to economic and environmental perspectives. Therefore, reusability of melamine *via* the reaction of β -naphthole /2-hydroxynaphthalene-1,4-dione with 4-chlorobenzaldehyde and malononitrile under the optimized reaction conditions was also evaluated. One of the interesting points of this catalytic system was the solubility of melamine in water, and thus catalyst could be very easily reused after filtration of the water-insoluble products.

For this purpose, after the reaction was completed, water (10 mL) was added. Then the catalyst was isolated from the reaction mixture by simple filtration and evaporation of the solvent from the filtrate, and dried prior to the next run. The recovered catalyst was submitted to a new reaction under optimized conditions. No significant change was found in catalyst activity compared with the fresh catalyst even after being reused at least four times. These results proved that melamine can be utilized as a recyclable catalyst for the condensation reactions (Figures 1 and 2).

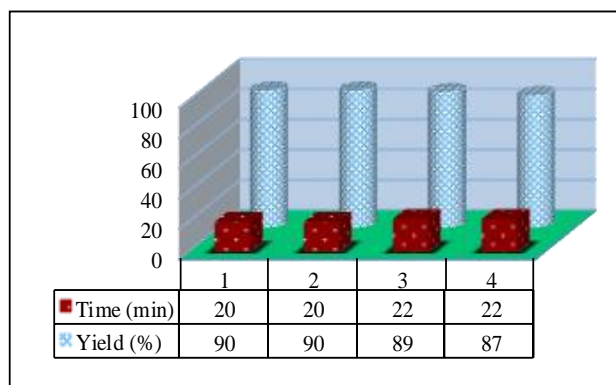


FIGURE 1 Recycling of the catalyst in the preparation of dihydropyrano[f]chromen derivatives

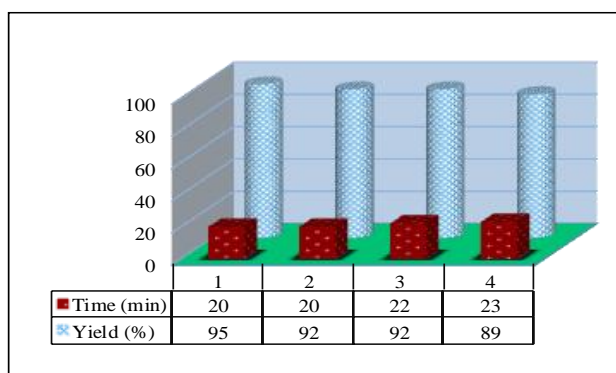


FIGURE 2 Recycling of the catalyst in the preparation of 4*H*-benzo[g]pyrans

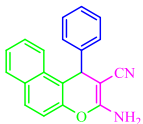
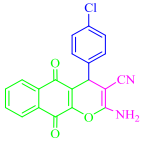
Finally, to explore the catalytic capability of melamine as an organocatalyst for the preparation of 3-amino-1-phenyl-1*H*-benzo[f]chromene-2-carbonitrile and 2-amino-4-(4-chlorophenyl)-5,10-dihydro-5,10-dioxo-4*H*-benzo[g]chromene-3-carbonitrile, we compared the results

reported in our method with some other published works in the literature. Considering the results obtained from Table 5, one can see each of reported methods has their own merits and all catalysts display acceptable catalytic activity for certain synthetic conditions, but they often suffer

from some drawbacks such as longer reaction times, non-reusable catalysts, tedious work-up procedures and organic solvent. As it is evident from Table 5, by using melamine in

the respective study, these problems are omitted which notably affirms that melamine can be brought up as an efficacious and eco-friendly benign catalyst.

TABLE 5 Comparing the performance of melamine with some of the other reported catalysts for the preparation of 3-amino-1-phenyl-1*H*-benzo[*f*]chromene-2-carbonitrile and 2-amino-4-(4-chlorophenyl)-5,10-dihydro-5,10-dioxo-4*H*-benzo[*g*]chromene-3-carbonitrile.

Entry	Product	Catalyst	Conditions	Time (min)	Yield (%) [Ref.]
1		KHP	H ₂ O, 50 °C	285	91 [32]
		Methanesulfonic acid	CH ₃ CN, reflux	180	91 [36]
		CTABr/ultrasound irradiation	H ₂ O, r.t.	150	78 [18]
		H ₁₄ [NaP ₅ W ₃₀ O ₁₁₀]	H ₂ O, reflux	165	93 [37]
		Melamine	Solvent-free, 120 °C	30	90 this work
2		Et ₃ N	CH ₃ CN, r.t.	24 h	80 [38]
		TEBA	Solvent-free, 85 °C	210	93 [34]
		SBPPSA	Solvent-free, r.t.	20	93 [16]
		[bmim]OH	EtOH, r.t.	48	92 [20]
		Melamine	H ₂ O:EtOH, reflux	20	95 this work

Conclusion

In summary, we have successfully expanded easy and efficacious method for the synthesis of various kinds of 2-amino-4*H*-chromenes via MCRs by use of melamine as an organocatalyst under solvent-free conditions and water-ethanol system which completely agrees with the principles of green chemistry. The melamine as a heterogeneous catalyst is mild, environmentally benign, easy to handle, economical, and commercially available reagent. Another notable advantage of the proposed system is the solubility of melamine in water, which facilitates its separation and recycling from the reaction medium. This green synthesis presents the desired products with good yields in a short period of times without a tedious purification procedure.

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