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Mefenamic acid as Environmentally Catalyst for Three-component Synthesis of Dihydropyrano [2,3-c]Chromene and Pyrano[2,3-d]Pyrimidine Derivatives

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Abstract

Pills of Mefenamic acid were employed as effective catalysts in the three component reaction of aromatic aldehydes, malononitrile, as well as 4-hydroxycoumarin or barbituric acid in aqueous solution, using reflux conditions in order to synthesize derivatives of dihydropyrano[2,3-c]chromene and pyrano[2,3-d]pyrimidine, correspondingly. Among the benefits of this technique, higher efficiency, shorter reaction time, along with better reaction indices could be mentioned.

Keywords: Mefenamic acid, Dihydropyrano [2,3-c]chromene, Pyrano[2,3-d]pyrimidine.

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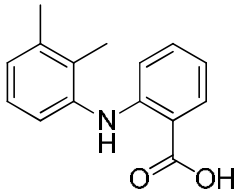
Introduction

In comparison with multistep reactions, those performed by multicomponent (MCR) procedures are considered to be more interesting and robust to synthesize organic matters, since they make it possible to form carbon – carbon as well as carbon – hetero atom bonds in a single pot [1-3]. Among the benefits of MCRs the simplicity of the processes, high efficiency in forming the bonds, saving time as well as energy, along with lower costs can be mentioned [4]. Development of MCRs has been aimed at synthesizing different small organic molecules in the fields of organic, bioorganic, as well as medicinal chemistry [5-8], while they are considered as important components of synthesis procedures in various principal heterocyclic combinations such as 4,5-dihydropyrano[2,3-c]chromene.

Derivatives of pyrano[2,3-c]chromene are considered in a category of essential heterocycles possessing a broad scope of biologic features [9] including relieving spasm, diuretic effects, anticoagulant, anti-cancer, as well as anti-anaphylactic functions [10]. Moreover, derivatives of aminochromene show a broad range of biologic functions consisting of anti-hypertension along with anti-ischemic characteristics [11-13]. Several procedures have been proposed in a variety of conditions to synthesize the mentioned heterocyclic compounds. Preparation of several of the compounds has been done using piperidine [14], diammonium hydrogen phosphate (DAHP) [15], K_2CO_3 with microwave irradiation [16], $H_6P_2W_{18}O_{62\_18}H_{2O}$ [17], MgO [18], tetra-butylammonium bromide (TBAB) [19], DBU [20],bisferrocene-containing ionic liquid supported on silica coated Fe_3O_4 [21], Nano SiO_2 [22] as well as 3-hydroxypropanaminium acetate (HPAA) [23].

Recently, significant attention has been paid to pyrano[2,3-d]pyrimidines, since they show a broad scope of different pharmacological functions and act for example as antitumor agents, cardio-tonics, or show features such as hepato-protection, anti-hypertension, as well as anti-bronchitis [24-28]. Synthesis of the above-mentioned compounds is usually done through a single-pot three-component cyclocondensation of 1,3-dimethylbarbituric acid, aryl aldehydes as well as malononitrile, while different catalysts including 1,8-diazabicyclo[5.4.0]undec-7-ene [29], MgO [30], PEG-stabilized Ni nanoparticles [31], $ZnFe_2O_4$ nanoparticles [32], KF [33], Mn-ZIF8@$ZnTiO_3$ nanocatalyst [34], copperated Fe_3O_4@polyvinyl alcohol magnetic nanoparticles [35] and Mn/ZrO_2 [36] are present. Moreover, these compounds have been synthesized applying microwave irradiation [37] along with electro-catalytic processes, while sodium bromide has been present as the electrolyte [38].Nevertheless, longer reaction time, multi-stage reactions, along with complicated synthetic paths will be needed in these procedures, leading to an average efficiency level. As a result, it is essential to introduce more moderate, quicker, and harmless techniques which will also lead to more significant efficiency. Mefenamicacid (MA) (Scheme 1) which is a non-steroid medicine used for

anti-inflammation, has been widely employed in clinical conditions [39].



Scheme 1. Structure of mefenamic acid.

In this primarily mefenamic acid, coplanarity can be observed between carboxylic group and nitrogen atom in the aromatic ring [40]. Investigation of the catalyst role of mefenamic acid and its derivatives has not been prevalent by the application of mefenamic acid to synthesize the novel derivatives and enhance its performance. The present study can be mentioned as the first one employing mefenamic acid as the catalyst. Further to our previous works [41, 42], we would like to present a novel green reaction through application of mefenamic acid tab as an effective catalyst to synthesize derivatives of dihydropyrano[2,3-c] chromene as well as pyrano[2,3-d]pyrimidine in single-pot conditions and through adding 4-hydroxycoumarin or barbituric acid, malononitrile and different aldehydes directly into the green media.

Experimental

General

Commercial sources supplied the chemicals and solvents required for the study; therefore, there was no need to further purify them, except in the conditions otherwise had been mentioned. Melt-Tem II melting point apparatus was used to determine the melting points which have not been corrected. Moreover, Matson-1000 FT-IR spectrometer was applied to determine the IR spectra. Wave numbers (cm^{-1}) have been used to report the peaks. A Bruker model DRX-300 AVANCE (^1H: 400, ^{13}C: 100 MHz) NMR spectrometer was also employed to record the NMR spectra. Chemical changes of ^1H as well as ^{13}C-NMR have been recorded in parts in million (ppm) from tetramethylsilane (TMS) as the inner criterion in DMSO-d_6 as a solvent.

General experimental process to synthesize derivatives of dihydropyrano[2,3-c]chromene as well as pyrano[2,3-d]pyrimidine

A combination of aldehyde (2 mmol), malononitrile (2.2 mmol), 4-hydroxycoumarin (2 mmol) or barbituric acid (2 mmol) along with mefenamic acid tab (0.5 g for dihydropyrano[2,3-c]chromenes or 0.3 g for pyrano[2,3-d]pyrimidines) in water (10 mL, for dihydropyrano[2,3-c]chromenes) or EtOH (10 mL, for pyrano[2,3-d]pyrimidines) was heated using reflux according to the time

intervals represented in tables 2&4. Control and supervision of the reaction could be performed by TLC. When the reaction was completed, filtration of the combination was carried out and then the mixture was washed with hot methanol, after which it was dried through decreasing the pressure.

2-amino-4-(2-hydroxynaphthalen-1-yl)-5-oxo-4,5-dihydropyrano[3,2-c]chromene-3-carbonitrile (**4h**)

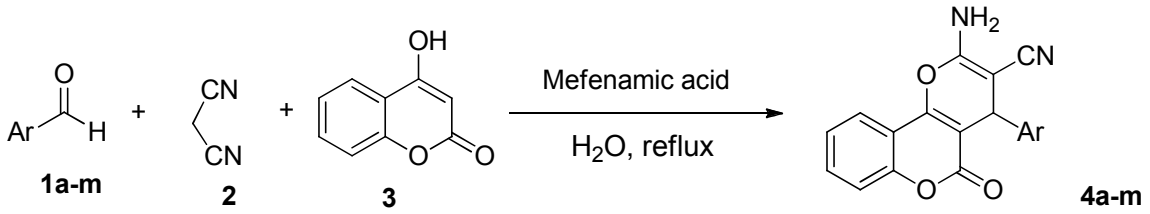
Yield: 86%. M.p. =258-260 °C. IR (KBr, cm^{-1}), 3436, 225, 1728, 1623. ^1H NMR (400 MHz, DMSO): δ = 3.37 (s, 1H), 6.95 (m, 1H), 7.49 (m, 1H), 7.66 (m, 4H), 7.75 (m, 1H), 7.80 (m, 1H), 8.11 (d, J = 8.4 Hz, 1H), 8.30 (d, J = 8.8 Hz, 1H), 8.39 (d, J = 8.8 Hz, 1H), 8.64 (d, J = 8.4 Hz, 1H), 10.78 (s, 1H).^{13}C NMR (100 MHz, DMSO): δ = 29.00, 100.76, 112.29, 115.02, 116.57, 116.73, 117.03, 119.34, 122.52, 126.37, 126.83, 128.74, 129.04, 129.35, 129.93, 131.16, 134.71, 135.47, 137.13, 138.86, 149.56, 154.98, 158.90.

7-amino-5-(3-nitrophenyl)-2,4-dioxo-2,3,4,5-tetrahydro-1H-pyrano[2,3-d]pyrimidine-6-carbonitrile (**6a**)

Yield: 97%. M.p. =268-270 °C. IR (KBr, cm^{-1}), 3417, 3203, 2192, 1711, 1659. ^1H NMR (400 MHz, DMSO): δ = 4.47 (s, 1H, CH), 7.30 (br s, 2H, NH2), 7.61 (t, J = 8.0 Hz, 1H), 8.06-8.12 (m, 2H, H-Ar), 11.12 (s, 2H, NH), 12.18 (s, 1H, NH).

Results and Discussion

A reaction was primarily carried out through exposing 4-hydoxycoumarine, malononitrile as well as 3-nitrobenzaldehyde by applying mefenamic acid tab as a catalyst in reflux conditions in water or different solvents such as EtOH, MeOH, $CHCl_3$, CH_2Cl_2, acetone and acetonirile. (Scheme 2, Table 1).



Scheme 2. Synthesis of dihydropyrano[2,3-c]chromene derivatives

Table 1. Optimization reaction condition in three component reaction of 3-nitro benzaldehyde, malononitrile and 4-hydroxycoumarin.

Entry	Solvent	Catalyst (Mefenamic acid 0.5 g)	Time	Isolated yield (%)
1	MeOH	0.5	290 min	64
2	EtOH	0.5	72 h	36
3	MeOH:H_2O (1:1)	0.5	30 min	70
4	EtOH:H_2O (1:1)	0.5	15 min	79
5	H_2O	0.5	8 min	95
6	$CHCl_3$	0.5	48 h	40
7	CH_2Cl_2	0.5	18 h	68
8	acetone	0.5	20 h	73
9	acetonitrile	0.5	20 h	45
10	H_2O	0.1	270 min	74
11	H_2O	0.2	120 min	73
12	H_2O	0.3	30 min	84
13	H_2O	0.4	10 min	90
14	H_2O	0.5	8 min	95
15	H_2O	0.6	8 min	95

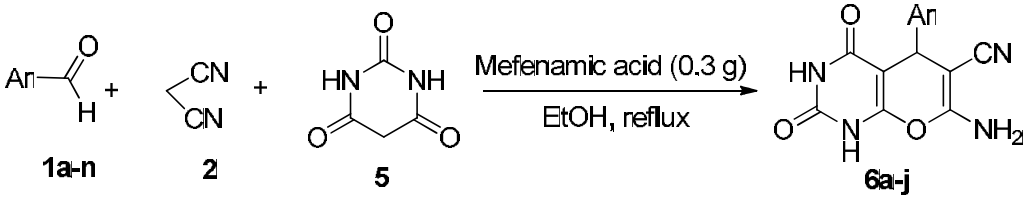
According to Table1, using water led to 95 percent of chemical output at reflux condition while mefenamic acid tabs were present (Table 1, Entry 5). Several organic solvents consisting of EtOH, MeOH, $CHCl_3$, CH_2Cl_2, acetone as well as acetonitrile led to 36 to 79 percent of output in a similar temperature, while 0.5 g of mefenamic acid tab had been used as the promoting factor. Consequently, water has been considered as the solvent, since it indicates environmental friendly features. For better optimization catalyst quantity, a similar reaction was performed using various catalysts at a range of 0.1-0.6 g.

According to Table 1, the product 4a output showed an improvement, while a shorter reaction time was needed with an increase in the catalyst quantity from 0.1g to 0.4 g (Entry 10-13). Subsequently, no sensible progress could be observed at the time of increasing the catalyst dose from 0.4 g to 0.6 g; however, an insignificant reduction of the reaction time was reported. In conclusion, it was reported that 0.5 g of mefenamic acid tab could be an appropriate parameter for obtaining derivatives of dihydropyrano [2,3-c] chromene, while water was used as the solvent at a temperature of 100°C.Afterward, the substrate domain of the procedure was examined through extension to different aldehyde groups which donate or withdraw electron in optimum conditions. According to table 2, different easily accessible aromatic aldehydes which bear functional groups that withdraw or donate electron, including nitro, chloro, bromo, or methyl were considered appropriate to be used in the reaction (Table 2, entries 1 to 13). In addition, performing exact analyses, subtle electronic behaviour could be observed: aromatic aldehydes with strong withdrew electron groups (entry 1, 2) had a faster reaction compared to the electron-rich groups (entry 8, 12) which reduced the ability to react and needed more reaction time.

Table 2. Synthesized dihydropyrano[2,3-c]chromene derivatives using mefenamic acid as catalyst.

Entry	Ar	product	Time (min)	Yield (%)	Melting points (°C) Found	Reported
1	3-$NO_2C_6H_4$	4a	8	95	260-262	266-268[24]
2	4-$NO_2C_6H_4$	4b	7	86	262-264	256-258[24]
3	C_6H_5	4c	16	89	256-258	255-257[24]
4	4-ClC_6H_4	4d	13	89	253-255	259-261[24]
5	4-$MeOC_6H_4$	4e	16	93	238-240	240-242[24]
6	4-MeC_6H_4	4f	10	91	253-255	250-252[24]
7	2,4-$(MeO)_2C_6H_3$	4g	14	83	240-242	236-238[44]
8	2-hydroxy naphthaldehyde	4h	35	86	258-260	-
9	2,4-$Cl_2C_6H_3$	4i	25	82	255-257	257-259[46]
10	2-$MeOC_6H_4$	4j	10	92	256-260	250-253[42]
11	2-MeC_6H_4	4k	13	91	273-275	279-282[46]
12	4-OHC_6H_4	4l	50	95	262-265	262-266[46]
13	3-Cl-C_6H_4	4m	10	96	234-235	236-237[49]

Investigation of the generalize ability of the procedure was performed at the optimum reaction conditions. The domain of the reaction was examined by selection of barbituric acid (Scheme 3). However, it was interesting to note that after applying barbituric acid it was impossible to detect the expected products from the combination reaction. According to these observations and to find an optimized condition for the synthesis of pyrano [2,3-d] pyrimidine derivatives, various conditions were examined, and the best results were obtained when 0.3 g of mefenamic acid tab in ethanol was used (Table3).



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

Table 3. Optimization of reaction conditions in three component reaction of 3-nitro benzaldehyde, malononitrile and barbituric acid.

Entry	solvent	Catalyst (Mefenamic acid, gr)	Time	Isolated yield (%)
1	H_2O	0.5	130 min	-
2	MeOH:H_2O (1:1)	0.5	150 min	-
3	EtOH:H_2O (1:1)	0.5	30 min	64
4	MeOH	0.5	140 min	40
5	EtOH	0.5	25 min	94
6	EtOH	0.4	25 min	96
7	EtOH	0.3	25 min	97
8	EtOH	0.2	30 min	92
9	EtOH	0.1	35 min	90

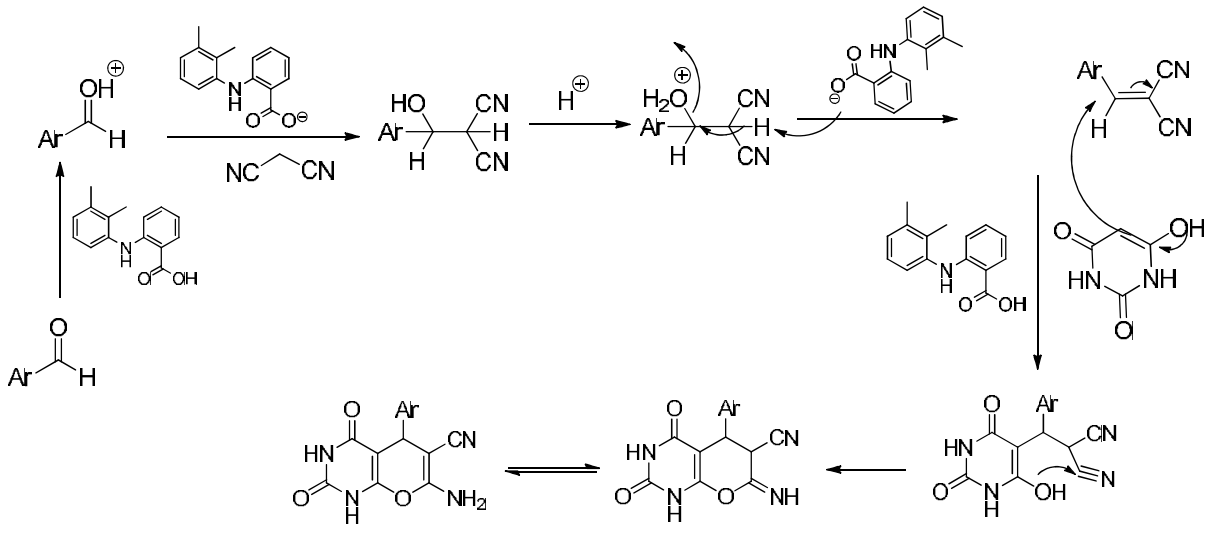
Different aldehydes were used (scheme 2) in order to examine the generalize ability of the protocol and investigate the employment of this procedure to synthesize pyrano[2,3-d]pyrimidinederivatives (Table 4).

Table 4. Synthesized of pyrano[2,3-d]pyrimidine derivatives using mefenamic acid tablet as catalyst.

Entry	Ar	Product	Time (min)	Yield (%)	Melting points (°C)	
					Found	Reported
1	3-$NO_2C_6H_4$	6a	25	97	268-270	269-270[42]
2	4-$NO_2C_6H_4$	6b	20	92	242-244	230-234[42]
3	2-$NO_2C_6H_4$	6c	35	95	253-255	257-258[54]
4	C_6H_5	6d	35	91	206-208	206-209[42]
5	4-ClC_6H_4	6e	30	98	228-230	234-237[54]
6	2,4-$Cl_2C_6H_3$	6f	25	94	240-242	239-240[42]
7	3-MeC_6H_4	6g	30	90	224-225	228-229[42]
8	4-MeC_6H_4	6h	45	97	223-225	226-227[52]
9	2-$MeOC_6H_4$	6i	40	94	203-205	198-201[52]
10	3,4,5-$(OCH_3)_3C_6H_2$	6j	15	95	247-249	247-249[52]

It was obviously observed that after using of the aldehyde with groups donating or withdrawing electrons, excellent yields were obtained and as a result higher efficiency could be achieved.

Proposed mechanism for the formation of pyrano[2,3-d]pyrimidine derivatives has been illustrated in scheme 4. Initially, Knoevenagel reaction took place when mefenamic acid was present and benzylidene malononitrile intermediate was primarily formed through condensing protonated aromatic aldehyde with malononitrile. In the next step, it proceeds through Michael addition of enolic barbituric acid to the benzylidene malononitrile intermediate, after which cyclization as well as tauomerism was performed.



Scheme 4. Proposed mechanism.

The results of the present study were compared to those found in the previous studies in relation to the reaction of aldehyde, malononitrile and 4-hydroxycoumarin or barbituric acid in order to ensure

the efficiency of the proposed method (Tables 4&5). As it can be observed, higher yields, green media, convenient work-up, and shorter time required for reaction can be mentioned as the benefits of the proposed method.

Conclusion

Overall, the proposed method which was green and highly efficient was developed to synthesize derivatives of dihydropyrano[2,3-c]chromene as well as pyrano[2,3-d]pyrimidine correspondingly. This was performed through the one-pot reaction of aldehydes, malononitrile, and 4-hydroxycoumarin or barbituric acid in water or EtOH using reflux conditions while mefenamic acid tab was employed as the catalyst. The reactions indicated extensive substrates of employing easily accessible as well as cheap initial substances. The green synthesis, especially indicates some interesting features including employment of water for the reaction media, simple techniques used to yield the products, shorter time required for the reaction, convenient work-up, considerable product yield, along with a plain and straightforward process.

Table 5. Comparison of our results to some of those reported in the literature for the reaction of aldehyde, malononitrile and 4-hydroxycoumarin.

Entry	Reaction condition	Yield (%)	Time	Ref.
1	DAHP (10 mol%), Ethanol-Water, r.t.	85	6 h	15
2	S-proline (10 mol%), Ethanol-Water, reflux	78	3 h	15
3	DBSA (25 mol%), water, reflux	90	4 h	24
4	SDS (20 mol%), water, reflux	88	2.5 h	43
5	K_2CO_3, MW, water	87	3 min	16
6	$RuBr_2$(PPh3)4, Ethanol, reflux	45	45 min	44
7	$H_6P_2W_{18}O_{62}$ $18H_2O$, H_2O:EtOH, reflux	87	60 min	17
8	Silicagel, Ethanol, r.t.	95	4 h	45
9	Iron ore pellet, Water, reflux	71	30 min	47
10	Nano aluminum oxide (Al_2O_3), ethanol, reflux	71	2 h	48
11	mefenamic acid, H_2O, reflux	95	8 min	This work

Table 6. Comparison of our results to some of those reported in the literature for the reaction of aldehyde, malononitrile and barbituric acid.

Entry	Reaction condition	Yield (%)	Time	Ref.
1	DBU Water/reflux	90	5 min	29
2	TBAB, H_2O, reflux	80-90	25-35 min	50
3	PEG-Ni nanoparticle, ethylene glycol, rt	87	5 min	31
4	DHP, EtOH, r.t.	71-81	2 h	51
5	DMF, MW	5-10 min	60-70	37
6	L-proline, H_2O/EtOH, r.t.	68-86	30-90 min	53
7	SBA-Pr-SO3H, H_2O/EtOH, r.t.	28	6 h	46
8	Iron ore pellet, H_2O/EtOH, reflux	73-93	8-31 min	42
9	mefenamic acid, EtOH, reflux	97	25 min	This work

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