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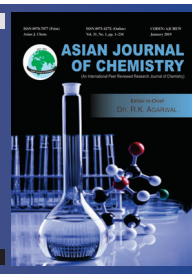

Microwave Assisted One Pot Synthesis of 3,4-Dihydropyrano[*c*]chromene Derivatives using [Emim]OH Ionic Liquid as Novel Catalyst

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One-pot efficient protocol to the synthesis of 2-amino-5-oxo-4,5-dihydropyrano(3,2-*c*)chromene-3-carbonitrile derivatives *via* condensation of various aryl aldehydes, dicyanomethane and 4-hydroxycoumarin in presence of Emim hydroxide as an excellent homogeneous liquid catalyst. The key advantages of this methodology are mild reaction conditions, novel catalyst, short reaction time, eco-friendly, easy work-up procedure and high yield of isolation of derivatives.

Keywords: Microwave assisted, [Emim]OH, Homogeneous catalyst, Green approach, 3,4-Dihydropyrano[*c*]chromene.

INTRODUCTION

The important feature of Green Chemistry is removal of harmful solvents or use of greener solvents such as to replace hazardous solvents with comparatively mild solvents like alcohol, surfactant and ionic liquids in chemical processes. Ethyl alcohol is the naturally benign, clean, economical solvent. Today major universal problem is pollution, so the most interesting challenge in organic synthesis for scientist community is to design easy and eco-friendly methodology such as multi-component reactions (MCRs). The MCRs has been attracting attention of numerous chemists because it is a powerful technique for synthesis of derivatives with biological activity and pharmaceutical properties [1]. To develop clean, easy and eco-friendly method for the synthesis of organic moiety with significant biological activity is the further most important objective in synthetic organic chemistry and is also helpful in research area of green or sustainable chemistry [2].

The natural products are rich source of active ingredients having wide range of medicinal application. The chromene is important heterocyclic compound showing great application in medicinal drug discovery. The chromene is a heterocyclic ring system containing 2*H*-chromen-2-one ring fused to pyran ring. Chromene forms the basic framework which is widely found in natural products like tocopherols, flavonoids, vitamin E, warfarin, chalcone and luteic acid. Chromene derivatives are structural moiety commonly found in most of the alkaloids with manifesting diverse biological activities [3,4]. The dihydropyrano[*c*]chromenes derivatives has fascinated attention of an important class of heterocyclic chemistry having useful pharmacological and biological activity such as analgesic [5], anti-HIV [6], anticancer [7], antituberculosis agents [8], anticoagulant [9], antibacterial [10], anti-Alzheimer [11], antimalarial [5], antimicrobials [8], antifungal [12], molluscidal [13], acetyl cholinesterase inhibitor [14] and anti-inflammatory [15]. The derivatives of dihydropyrano[2,3-*c*]chromene can also be employed as cosmetic pigment and utilized as potential biodegradable agrochemical [16].

The synthesis of various 3,4-dihydropyrano[*c*]chromene derivatives have been reported using catalysts such as K_2CO_3 [17],tetrabuthylammonium bromide (TBAB) [18], s-proline [19], nano Al_2O_3 [20], cesium carbonate [21], thiourea dioxide [22], [DBU][Ac] [23], nano-structured ZnO [24], LSMO [25], HEAA [26], SuSa [27], [Bmim]HSO_4 [28], Amberlite A21 [29], Ru(II) [30], Mo132 [31], ammonium acetate [32], DBU [33], piperidine [34], NaBr [35], ([DMAP-PEG1000-DIL][BF4]) [36] and ionic liquid [37].

EXPERIMENTAL

The solvents, starting material and catalyst were purchased from S.D. Fine Chemicals, Spectrochem and Sigma Aldrich with high purity and used without further purification. All the materials were of commercial grade. Melting points of synthesized derivatives were found out using open capillaries by visual melting point instrument. The FT-IR spectra recorded using KBr on Perkin-Elmer FT-IR spectrometer 65. The ^{1}H NMR spectras were recorded on Bruker Advance spectrometer an 400 MHz in $CDCl_3$ as a solvent at 295 K and chemical shift record in δ (ppm) using TMS (tetramethylsilane) as an internal standard. The reactions progresses was monitored by TLC. The experiments under microwave irradiation were carried out in 700 W and 2450 MHz frequency model made by RAGA's Scientific Microwave Synthesis System Pvt. Ltd., Pune, India.

General Procedure for the synthesis of 2-amino-5-oxo-4,5-dihydropyrano(3,2-*c*)chromene-3-carbonitriles (3a-p)

Conventional method: A mixture of aryl aldehyde (1.0 mmol), 4-hydroxycoumarin (1.0 mmol), malononitrile (1.1 mmol) and [Emim]OH (0.5 mL) in 50 mL round bottom flask with 5 mL ethyl alcohol as solvent, was refluxed at 50-60 ºC for 40-55 min. The reaction progress was observed using TLC in *n*-hexane:ethyl acetate (70: 30 v/v) solvent system. After completion of reaction, the reaction mixture was poured on ice-cold water, stirred and filtered. The obtained residue was purified by crystallization from aqueous ethyl alcohol (**Scheme-I**).

Non-conventional (microwave assisted) method: The mixture of aryl aldehyde (1.0 mmol), 4-hydroxycoumarin (1.0 mmol), malononitrile (1.1 mmol) and [Emim]OH (0.5 mL). The reaction mixture was refluxed in microwave oven at 700 W for 2-4 min. The reaction progress was observed using thin layer chromatography in *n*-hexane:ethyl acetate (70:30 v/v) solvent system. As soon as reaction is completed the reaction mixture was poured on ice cold water, stirred and filtered. The obtained residue was purified by crystallization from aqueous ethyl alcohol (**Scheme-I**).

Spectral data

2-Amino-4,5-dihydro-4-phenyl-4,5-oxopyrano[3,2-*c*]-chromene-3-carbonitrile (3a): Light yellow colour, m.p. 256-258 ºC; FT-IR (KBr, ν_{max}, cm^{-1}): 3350, 3250, 2955, 2180, 1690, 1570, 1080. ^{1}H NMR ($CDCl_3$, 400 MHz): δ 4.5 (*s*, 1H), 7.1-7.4 (*m*, 5H), 7.6-7.8 (*m*, 4H), 8.6 (*s*, 2H). Elemental analysis of $C_{19}H_{12}N_2O_3$ calcd. (found) (%):C, 72.15 (72.10); H, 3.82 (3.82); N, 8.86 (8.85).

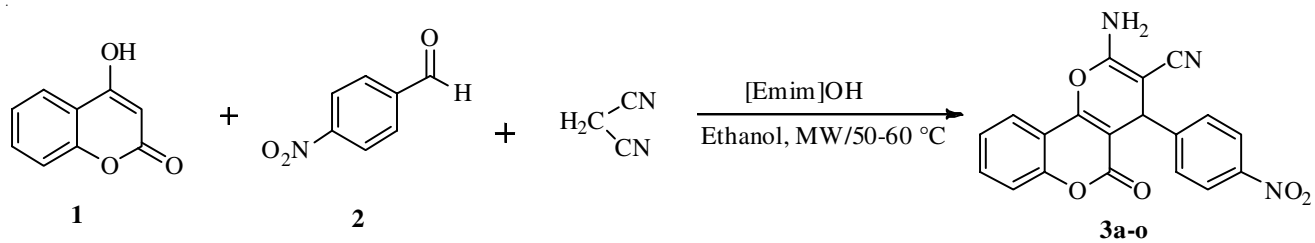
2-Amino-4,5-dihydro-4-(3-nitrophenyl)-5-oxopyrano-[3,2-*c*]chromene-3-carbonitrile (3c): Yellow colour solid; yield 92 %; melting point 257-258 ºC; Recrystallized in ethanol; FT-IR (KBr, ν_{max}, cm^{-1}): 1045, 1088, 1378, 1527, 1664, 1706, 1717, 2199, 2978, 3349, 3367 (broad); ^{1}H NMR ($CDCl_3$): δ 5.5 (*s*, 1H), 7.2 (*dd*, 1H) 7.4-7.6 (*m*, 5H), 7.6 (*s*, 2H), 7.9 (*dd*, 1H), 8.0 (*dd*, 1H); MS *m/z* 384 (M^+ + 23).

2-Amino-4,5-dihydro-4-(4-methoxyphenyl)-5-oxo-pyrano[3,2-*c*]chromene-3-carbonitrile (3d): Light yellow color, m.p. 243-244 ºC. FT-IR (KBr, ν_{max}, cm^{-1}): 3185, 2960, 2270, 1670, 1510, 1060. ^{1}H NMR ($CDCl_3$, 400 MHz): δ 3.2 (*s*, 3H), 4.6 (*s*, 1H), 7.1-7.4 (*m*, 4H), 7.5-7.8 (*m*, 4H), 8.2 (*s*, 2H). Elemental analysis of $C_{20}H_{14}N_2O_4$ calcd. (found) (%):C, 69.36 (69.36); H, 4.07 (4.08); N, 8.09 (8.10).

2-Amino-4,5-dihydro-4-(4-bromophenyl)-5-oxopyrano-[3,2-*c*]chromene-3-carbonitrile (3e): Light yellow colour solid; yield 90 %, m.p. 248-250 ºC; recrystallized in ethanol; FT-IR (KBr, ν_{max}, cm^{-1}): 1088, 1374, 1485, 1601, 1673, 1707, 2190, 2973, 3308, 3349 (broad). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 4.6(s, 1H), 7.3 (*dd*, 1H) 7.4-7.5 (*m*, 3H), 7.6 (*dd*, 2H), 7.7 (*dd*, 1H), 7.9 (*dd*, 1H), 8.1 (*s*, 2H); MS *m/z* 395 (M^+ + 1). Elemental analysis of $C_{19}H_{11}N_2O_3Br$ calcd. (found) (%):C, 57.74 (57.75); H, 2.81 (2.80); N, 7.09 (7.10).

2-Amino-4,5-dihydro-4-(4-chlorophenyl)-5-oxopyrano-[3,2-*c*]chromene-3-carbonitrile (3f): Light yellow color, mp 256-258 ºC; FT-IR (KBr, ν_{max}, cm^{-1}): 3120, 3270, 2945, 2250, 1710, 1610, 1060. ^{1}H NMR ($CDCl_3$, 400 MHz): δ 4.6 (*s*, 1H), 7.2-7.4 (*m*, 4H), 7.3-7.5 (*m*, 4H), 8.7 (*s*, 2H). Elemental analysis of $C_{19}H_{11}N_2O_3Cl$ calcd. (found) (%):C, 65.06 (65.10); H, 3.16 (3.20); N, 7.99 (7.90); O, 13.68 (13.70).

2-Amino-4,5-dihydro-4-(4-fluorophenyl)-5-oxopyrano-[3,2-*c*]chromene-3-carbonitrile (3g): Light yellow colour, m.p. 261-63 ºC; FT-IR (KBr, ν_{max}, cm^{-1}): 3150, 3280, 2955, 2250, 1720, 1620, 1070. ^{1}H NMR ($CDCl_3$, 400 MHz): δ 4.7 (*s*, 1H), 7.3-7.5 (*m*, 4H), 7.4-7.5 (*m*, 4H), 8.8 (*s*, 2H). Elemental



Scheme-I: Synthetic path of 3,4-Dihydropyrano[*c*]chromene derivatives

TABLE-1
COMPARISON OF REACTION CONDITION

Entry	Catalyst	Time (min)	Condition	Yield (%)	Ref.
1	Nano Al_2O_3	300	90 °C, Reflux	85	[20]
2	Cesium carbonate	45	Visible light	95	[21]
3	Water, thiourea	40	Reflux, 70 °C	90-95	[22]
4	[DBU][Ac]	6-30	Grading	90	[23]
5	Nano-structured ZnO	150	Reflux	78-91	[24]
6	[Emim]OH	10-20	Reflux, 50 °C	90-96	**Present work**

TABLE-2
SCREENING OF CATALYST CONCENTRATION AND SOLVENTS

Entry	Catalyst concentration (mL)	Solvent	Condition	Time (min)	Yield (%)
1		DCM	Reflux	60	Trace
2		Chloroform	Reflux	60	Trace
3		Acetonitrile	Reflux	60	Trace
4		DMF	Reflux	60	Trace
5		Ethanol	Reflux	60	25
6	0.5	–	Reflux	60	60
7	0.5	–	50 °C	60	65
8	0.5	–	60 °C	60	70
9	0.5	–	70 °C	60	72
10	0.5	–	90 °C	60	78
11	0.5	DCM	Reflux	60	45
12	0.5	Chloroform	Reflux	60	60
13	0.5	Acetonitrile	Reflux	60	55
14	0.5	DMF	Reflux	60	40
15	0.5	Ethyl alcohol	Reflux	60	61
16	0.1	Ethyl alcohol	Reflux	60	35
17	0.2	Ethyl alcohol	Reflux	60	50
18	0.3	Ethyl alcohol	Reflux	60	65
19	0.4	Ethyl alcohol	Reflux	60	78
20	0.5	Ethyl alcohol	Reflux	60	81
21	0.5	Ethyl alcohol	Reflux	60	75
22	0.5	Ethyl alcohol	30-40 °C	60	85
23	0.5	Ethyl alcohol	50-60 °C	40	96
24	0.5	Ethyl alcohol	MW	3	95

analysis of $C_{19}H_{11}N_2O_3F$ calcd. (found) (%):C, 68.26 (68.24); H, 3.32 (3.30); N, 8.38 (8.40).

2-Amino-4,5-dihydro-4-(3-chlorophenyl)-5-oxopyrano-[3,2-*c*]chromene-3-carbonitrile (3h): Light yellow colour, m.p. 245-247°C; FT-IR (KBr, ν_{max}, cm^{-1}): 3150, 3240, 2951, 2253, 1690, 1580, 1070. ^{1}H NMR ($CDCl_3$, 400 MHz): δ 4.4 (*s*, 1H), 7.1-7.2 (*m*, 4H), 7.1-7.3 (*m*, 4H), 8.5 (*s*, 2H). Elemental analysis of $C_{19}H_{11}N_2O_3Cl$ calcd. (found) (%):C, 65.06 (65.10); H, 3.20 (3.30); N, 7.99 (7.95).

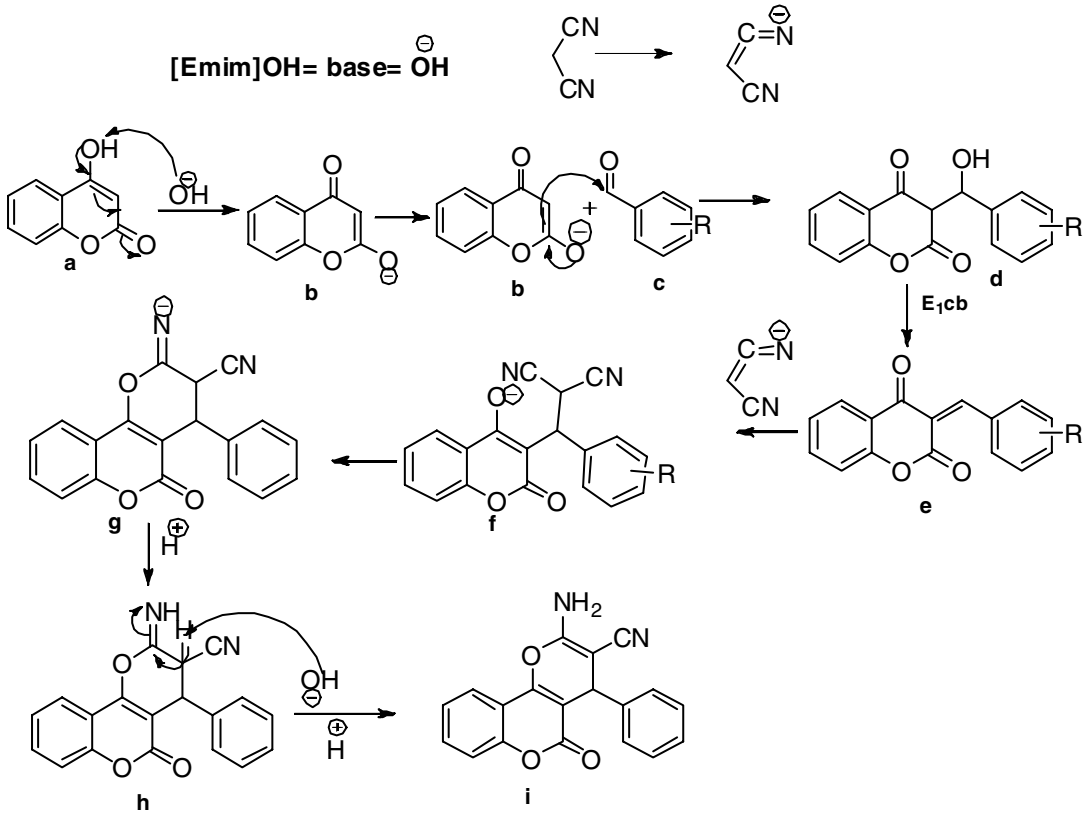
2-Amino-4,5-dihydro-4-(*p*-tolyl)-5-oxopyrano[3,2-*c*]-chromene-3-carbonitrile (3i): Light yellow colour, m.p. 280-282 ºC; FT-IR (KBr, ν_{max}, cm^{-1}): 3113, 3230, 2935, 2235, 1680, 1560, 1050. ^{1}H NMR ($CDCl_3$, 400 MHz): δ 4.2 (*s*, 1H), 6.9-7.1 (*m*, 4H), 6.4-6.6 (*m*, 4H), 8.1 (*s*, 2H). Elemental analysis of $C_{20}H_{14}N_2O_3$ calcd. (found) (%):C, 72.72 (72.70); H, 4.27 (4.30); N, 8.48 (8.50).

2-Amino-4,5-dihydro-4-(4-hydroxyphenyl)-5-oxopyrano-[3,2-*c*]chromene-3-carbonitrile (3j): Light yellow color, mp 263-265 ºC. FT-IR (KBr, ν_{max}, cm^{-1}): 3163, 3260, 2965, 2250, 1690, 1540, 1070. ^{1}H NMR ($CDCl_3$, 400 MHz): δ 4.2 (*s*, 1H), 5.2 (*s*, 1H), 6.8-6.9 (*m*, 4H), 6.3-6.5 (*m*, 4H), 8.2 (*s*, 2H). Elemental analysis of $C_{20}H_{14}N_2O_4$ calcd. (found) (%):C, 68.67 (68.70); H, 3.64 (3.62); N, 8.43 (8.42).

2-Amino-4,5-dihydro-4-(2,4-dichlorophenyl)-5-oxo-pyrano[3,2-*c*]chromene-3-carbonitrile (3k): Light yellow colour, m.p. 260-262 ºC. FT-IR (KBr, ν_{max}, cm^{-1}): 3140, 3270, 2965, 2273, 1710, 1590, 1060. ^{1}H NMR ($CDCl_3$, 400 MHz): δ 4.7 (*s*, 1H), 7.2-7.3 (*m*, 4H), 7.5-7.6 (*m*, 3H), 8.5 (*s*, 2H). Elem-

TABLE-3
[Emim]OH CATALYSED SYNTHESIS OF VARIOUS DIHYDROPYRANO[2,3-*c*]CHROMENE DERIVATIVES

Entry	Aldehyde	Time (min)		Yield (%)		m.p. (°C)	
		Conventional method	Microwave method	Conventional method	Microwave method	Found	Reported
3a	Benzaldehyde	55	3.0	92	90	256-258	255-257 [27,35]
3b	4-Nitrobenzaldehyde	40	3.0	96	95	245-247	257-262 [27,35]
3c	3-Nitrobenzaldehyde	40	2.5	92	92	257-258	260-264 [27,35]
3d	4-Methoxybenzaldehyde	55	3.0	90	88	243-244	241-243 [35,36]
3e	4-Bromobenzaldehyde	50	3.0	91	90	248-250	225-227 [27,36]
3f	4-Chlorobenzaldehyde	40	3.0	89	90	256-258	265-267 [35,36]
3g	4-Fluorobenzaldehyde	40	2.5	93	92	261-63	277-284 [35,36]
3h	3-Chlorobenzaldehyde	45	3.0	92	90	245-247	243-244 [37]
3i	4-Methylbenzaldehyde	50	3.5	90	86	280-282	262-265 [35,36]
3j	4-Hydroxybenzaldehyde	50	4.0	89	88	263-265	262-265 [35,36]
3k	2,4-Dichlorobenzaldehyde	55	3.0	90	89	260-262	258-266 [35]
3l	2-Methylbenzaldehyde	45	4.0	88	89	262-264	262-265 [35]
3m	2-Chlorobenzaldehyde	50	3.0	85	86	263-264	263-264 [37]
3n	Furan-2-carbaldehyde	45	3.0	88	90	223-224	223-224 [27]
3o	4-(Dimethylamino)benzaldehyde	50	4.0	85	88	225-226	225-226 [36]



Scheme-II: Mechanism of 3,4-dihydropyrano[*c*]chromene derivatives

ental analysis of $C_{19}H_{10}N_2O_3Cl_2$ calcd. (found) (%):C, 59.24 (59.30); H, 2.62 (2.63); N, 7.27 (7.30).

2-Amino-4,5-dihydro-4-(2-methoxyphenyl)-5-oxopyrano[3,2-*c*]chromene-3-carbonitrile (3l): Light yellow color, mp 280-282 ºC. FT-IR (KBr, ν_{max}, cm^{-1}): 3113, 3230, 2935, 2235, 1680, 1560, 1050. ^{1}H NMR ($CDCl_3$, 400 MHz): δ 4.2 (*s*, 1H), 6.9-7.1 (*m*, 4H), 6.4-6.6 (*m*, 4H), 8.1 (*s*, 2H). Elemental analysis of $C_{20}H_{14}N_2O_4$ calcd. (found) (%):C, 69.36 (69.30); H, 4.07 (4.10); N, 8.09 (8.10).

2-Amino-4,5-dihydro-4-(2-chlorophenyl)-5-oxopyrano-[3,2-*c*]chromene-3-carbonitrile (3m): Light yellow colour, m.p. 263-264 ºC. FT-IR (KBr, ν_{max}, cm^{-1}): 3140, 3250, 2951, 2253, 1710, 1570, 1055. ^{1}H NMR ($CDCl_3$, 400 MHz): δ 4.4 (*s*, 1H), 7.1-7.2 (*m*, 4H), 7.1-7.3 (*m*, 4H), 8.5 (*s*, 2H). Elemental analysis of $C_{19}H_{11}N_2O_3Cl$ calcd. (found) (%):C, 65.06 (69.30); H, 3.16 (3.15); N, 7.99 (7.95).

2-Amino-4,5-dihydro-4-(furan-2-yl)-5-oxopyrano[3,2-*c*]chromene-3-carbonitrile (3n): Light yellow colour, m.p. 223-224 ºC. FT-IR (KBr, ν_{max}, cm^{-1}): 3163, 3260, 2965, 2250, 1690, 1540, 1070. ^{1}H NMR ($CDCl_3$, 400 MHz): δ 4.2 (*s*, 1H), 6.8-6.9 (*m*, 4H), 6.3-6.5 (*m*, 4H), 8.2 (*s*, 2H). Elemental analysis of $C_{17}H_{10}N_2O_4$ calcd. (found) (%):C, 66.67 (66.70); H, 3.29 (3.25); N, 9.15 (9.25).

2-Amino-4,5-dihydro-4-(4-(dimethylamino)phenyl)-5-oxopyrano[3,2-*c*]chromene-3-carbonitrile (3o): Light yellow colour, m.p. 225-226 ºC. FT-IR (KBr, ν_{max}, cm^{-1}): 3213, 3210, 2915, 2243, 1690, 1550, 1040. ^{1}H NMR ($CDCl_3$, 400 MHz): δ 3.2 (s, 6H), 4.4 (s, 1H), 6.8-6.9 (m, 4H), 6.3-6.5 (m, 4H), 8.2 (s, 2H). Elemental analysis of $C_{21}H_{17}N_3O_3$ calcd. (found) (%):C, 70.18 (70.10); H, 4.77 (4.75); N, 11.69 (11.70); O, 13.36 (13.40).

RESULTS AND DISCUSSION

To set reaction conditions during continuation of present research work to develop new method, for synthesis of 2-amino-5-oxo-4,5-dihydropyrano(3,2-*c*)chromene-3-carbonitrile derivatives, we followed Multi-component reactions approach.

The reaction conditions were checked under various conditions as illustrated in Tables 1 and 2. The reaction tested in different solvents, such as chloroform, DCM, acetonitrile, DMF and ethyl alcohol gave poor yield in absence of catalyst (entry 1 to 5, Table-2) and we found that in presence of catalyst in solvent-free conditions and 0.5 mL [Emim]OH catalyst after refluxing at various temperature resulted into moderate yield *i.e.* upto 60 to 78 % (entry 6 to 10, Table-2). Further reaction conditions were checked using 0.5 mL of [Emim]OH and different solvents such as DCM, chloroform, acetonitrile, DMF, ethanol and found to give 45, 60, 55, 40, 61 % yield, respectively (entry 11 to 15, Table-2). To confirm catalyst concentration, reactions were performed at various catalyst concentration using ethyl alcohol as solvent from 0.1 M, 0.2 M, 0.3 M, 0.4 M and 0.5 M which resulted into 35, 50, 65, 78, 81 % yield, respectively (entry 16 to 20, Table-2).

In order to set temperature condition, temperature was increased from room temperature to 60 ºC and with increase in temperature the yield of products was increased from 81 to 96 % (entry 21 to 23, Table-2). All the reactions were monitored by thin layer chromatography. Similarly, to check the efficiency and practicality of proposed protocol, we used different aromatic aldehydes and the results are illustrated in Table-3. The reaction takes place with satisfied yield under microwave conditions in presence of polar solvents ethyl alcohol, DMF, water, aceto-

nitrile and methanol. It was observed that ethanol medium is the best reaction medium among the polar solvents used in demonstration and reaction proceeded very smoothly which gave the desired product with excellent yield within short reaction time (entry 24, Table-2). The aryl aldehydes which possess electron withdrawing substituent gave high yield as compared to aromatic aldehydes bearing electron donating substituent as illustrated in Table-3.

Reaction mechanism: In this reaction, [Emim]OH acts as the base, malononitrile and 4-hydroxycoumarine undergo enolization from a to b. The b on cross aldol condensation with c gave intermediate d. The intermediate d on E_1cB elimination gives intermediate e. The intermediate e with malononitrile on Michael addition gives intermediate f, which on cyclization gives intermediate g and after protonation forms intermediate h. Finally h on rearrangement converted into dihydropyrano[2,3-*c*]-chromene (**Scheme-II**).

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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