

### Catalytic application of a nano-molten salt catalyst in the synthesis of biological naphthoquinone-based compounds

Meysam Yarie<sup>1</sup> · Mohammad Ali Zolfigol<sup>1</sup> · Saeed Babaee<sup>1</sup> · Saeed Baghery<sup>1</sup> · Diego A. Alonso<sup>2</sup> · Abbas Khoshnood<sup>2</sup>

Received: 17 October 2017 / Accepted: 8 January 2018 © Springer Science+Business Media B.V., part of Springer Nature 2018

**Abstract** In this investigation, a new application of 1H-imidazol-3-ium tricyanomethanide catalyst was explored. The catalyst presented a robust catalytic applicability for the preparation of naphthoquinone-based compounds under mild and green reaction conditions. A wide range of aromatic aldehydes were able to react with 2-hydroxynaphthalene-1,4-dione and 3-methyl-1-phenyl-1H-pyrazol-5(4H)one or malononitrile to afford the desired naphthoquinone-based molecules in short time with high to excellent yields.

**Graphical Abstract** A good range of aromatic aldehydes were treated with 2-hydroxynaphthalene-1,4-dione (henna) and 3-methyl-1-phenyl-1*H*-pyrazol-5(4H)-one or malononitrile to afford the desired henna-based target molecules in short reaction time with good to excellent yields.

Mohammad Ali Zolfigol zolfi@basu.ac.ir; mzolfigol@yahoo.com

Diego A. Alonso diego.alonso@ua.es

Abbas Khoshnood abbas.khoshnood@ua.es

- <sup>1</sup> Department of Organic Chemistry, Faculty of Chemistry, Bu-Ali Sina University, Hamedan 6517838683, Iran
- <sup>2</sup> Instituto de Síntesis Orgánica, and Departamento de Química Orgánica, Universidad de Alicante, Apdo. 99, 03080 Alicante, Spain

**Electronic supplementary material** The online version of this article (https://doi.org/10.1007/ s11164-018-3264-9) contains supplementary material, which is available to authorized users.

Meysam Yarie myari.5266@gmail.com



**Keywords** 1*H*-imidazol-3-ium tricyanomethanide · Naphthoquinone-based compounds · Nano-molten salt catalyst · Multicomponent reactions

### Introduction

As building blocks of diverse naturally occurring products, henna-based organic compounds have found a privileged position in heterocyclic chemistry. From a historical standpoint, Lawsonia inermis (source of henna) has played a key role in religious customs and ordinary life style. Traditionally, ancient cultures widely used henna for treatment of liver and digestive disease, leprosy, diabetes, ulcer as well as protection of hands and feet against fungal pathogens [1, 2]. Also, plants containing henna-based molecules have been employed in the therapy of cancer [3, 4]. Nowadays, henna-based materials, have found a special position in different fields of science such as phytochemistry and medicinal chemistry [1, 2]. In addition to the myriad prominent in vitro and in vivo therapeutic applications of henna, their derivatives present pharmaceutical and medicinal versatility in the field of drug chemistry, such as, cytotoxic activity, anti-inflammatory, antibacterial, anti-HIV, fungicidal, molluscicidal, leishmanicidal, antimalarial, trypanocidal, and antitumoral bioactivities [5-12]. Some bioactive heterocyclic compounds bearing the naphthoquinone moiety as anthracycline drugs and naturally occurring products are depicted in Schemes 1 and 2 [1, 2, 13].

On the other hand, pyrazole-containing heterocyclic compounds have emerged as interesting bioactive materials which a broad spectrum of pharmacological and medicinal applicabilities comprising fungicidal [14], anticancer [15], vasodilatory [16], antianginal [17], and antitumor bioactivities [18]. Therefore, it is believed that the coexistence of both naphthoquinone and pyrazole segments in a given molecule



Scheme 1 Antitumour anthracycline drugs



Scheme 2 Naturally occurring products bearing a naphthoquinone fragment

may provide new bioactivities or improve referred therapeutic features of them. So a new eco-friendly approach to these versatile heterocyclic molecules is highly valuable.

Ionic liquids and molten salts, due to their special character and various applications in the recent era of modern chemistry as catalyst, reagents, and solvents, have attracted substantial consideration of researchers in the last decade [19–23]. Ionic liquids can be considered as "designer species" due to their chemical and thermal stability, solvating capability, vapor pressure, and other interesting properties which can be adjusted by using different combinations of anions and cations to form taskspecific ones [24–27].

One-pot multicomponent reactions have recently emerged as a versatile atomeconomy synthetic tool. This excellent alternative to the routine linear-type synthetic procedures has been especially used in the construction of complex heterocyclic materials. Also, multicomponent reactions can arrange facile and uncomplicated protocols to provide organic molecule libraries and diversity-oriented synthesis (DOS) [28–32].

In this study, in order to mature our interest on the use of task-specific ionic liquids, molten salts, biologically based solid acids, and nanoparticles with ionic tags as catalysts for the construction of biologically active heterocyclic materials [33–46], herein we wish to report a new application of 1*H*-imidazol-3-ium tricyanomethanide (ImITCM) as an efficient catalyst for the preparation of versatile, pharmaceutical naphthoquinone-based target molecules under green and eco-friendly reaction conditions (Scheme 3).



Scheme 3 ImITCM as a robust catalyst for the preparation of naphthoquinone-based target molecules

#### **Results and discussion**

Initially, ImITCM as a robust molten salt catalyst was prepared according to our previously reported method, as depicted in Scheme 4 [47]. The structural confirmation was fully made and all provided spectra confirmed the synthesis of ImITCM catalyst (see Electronic Supplementary Information).

After ensurance of catalyst preparation using proper technical skills, in order to obtain the optimal conditions to perform the reactions, the reaction of benzaldehyde, 2-hydroxynaphthalene-1,4-dione, and 5-methyl-2-phenyl-2,4-dihydro-3H-pyrazol-3-one was selected as the test reaction (Scheme 5). Over the model reaction, different reaction parameters such as solvent, operational temperature, and load of molten salt catalyst were screened. The obtained related data are embedded in Table 1. Firstly, using the same amount of catalyst, it is found that 90 °C is the optimal reaction temperature (Table 1, entries 1–3). Also, the model reaction was performed in refluxing common laboratory solvents and the obtained data indicated that the solvent-free condition is the best choice (Table 1, entries 4–7). Finally, the optimal amount of catalyst is demonstrated to be 6 mg (Table 1, entries 8–10). From the data in Table 1, it can be deduced that solvent-free conditions and using a catalytic

Scheme 4 Preparation route of ImITCM catalyst





Scheme 5 Screening of the optimal reaction conditions over the model reaction

Entry	Solvent	Load of catalyst (mg)	Temperature (°C)	Time (min)	Yield (%) <sup>a</sup>
1	_	4	70	45	63
2	_	4	90	22	87
3	_	4	110	15	87
4	$H_2O$	4	Reflux	30	48
5	EtOH	4	Reflux	90	Trace
6	CH <sub>3</sub> CN	4	Reflux	90	_
7	<i>n</i> -Hexane	4	Reflux	90	_
8	_	2	90	36	72
9	-	6	90	18	90
10	-	8	90	18	89

 Table 1
 Optimization of reaction conditions for the synthesis of naphthoquinone-based molecules

Reaction conditions: benzaldehyde (1 mmol, 0.106 g), 5-methyl-2-phenyl-2,4-dihydro-3*H*-pyrazol-3-one (1 mmol, 0.174 g), and 2-hydroxynaphthalene-1,4-dione (1 mmol, 0.174 g)

<sup>a</sup>Isolated yields

amount of catalyst at 90 °C are the optimal reaction conditions for the preparation of naphthoquinone-based molecules (Table 1, entry 9).

Subsequently, with optimal reaction data in hand, in order to extend the capability of the catalyst, we focused on the generality and the scope of the presented method for the synthesis of naphthoquinone-based heterocyclic molecules by using a wide range of aromatic aldehydes (possessing electron-withdrawing, electron and -donating components, and halogens) and other starting materials, as depicted in Scheme 3. The obtained data as embedded in Table 2 confirm the versatility of the presented procedure. All used starting materials interact with each other in a one-pot multicomponent reaction to afford desired target molecules in short reaction times with high to excellent yields.

Reusability of catalyst is one of the most outstanding advantages for its application in various chemical processes. Thus, the reusability of described catalyst was carried out successfully for three runs. For this purpose the reaction of benzaldehyde, 2-hydroxynaphthalene-1,4-dione, and

 Table 2
 Synthesis of the naphthoquinone-based molecules in the presence of ImITCM under mild reaction conditions

Entry	R	Product	Structure of naphthoquinone- based molecules	Time (min)	Yield (%) <sup>a</sup>	M.p. (°C) found [Lit.] [Ref.]
1	Н	1a		18	90	266–268 [267–268] [48]
2	2-Cl	1b		25	85	250–252 [272–273] [49]
3	4-C1	1c	HO HO N ME HO O	10	88	262–263 [262–264] [48]
4	4-F	1d	HO HO N Me HO O	25	74	256–258 [257–259] [48]
5	4-OMe	1e		13	92	253–254 [266–268] [49]
6	3,4- (OMe) <sub>2</sub>	1f	HO Nero HO N Me HO O	40	91	235–237 [235–237] [48]
7	4-N(Me) <sub>2</sub>	1g	HO N Me HO N Me HO O	21	83	210–212 [211–213] [48]
8	Thiophen- 2-yl	1h		17	74	238–240 [239–240] [48]

Table 2	(continued)
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9	2,4-Cl <sub>2</sub>	1i	HO CI HO CI Me HO D	23	92	256–257 [257–258] [48]
10	4-Me	1j		22	89	264–266 [265–266] [48]
11	3-OH	1k	HO N Me HO	20	81	245–246 [new]
12	3-NO <sub>2</sub>	11		22	87	248–249 [266–268] [48]
13	3-Br	1m	HO HO N Me HO	45	88	214–215 [270–271] [49]
14	4-CN	1n		15	89	226–227 [273–274] [49]
15	Н	2a		25	88	260–263 [260–263] [50]
16	4-C1	2b		17	92	248– 251[250– 251] [50]

5-methyl-2-phenyl-2,4-dihydro-3*H*-pyrazol-3-one was selected as a test reaction. After each run, distillated water was added to the reaction mixture, stirred for a few

17	2-Cl	2c	22	90	237–239 [235–237] [50]
18	4-F	2d	17	87	240–245 [244–246] [51]
19	4-NO <sub>2</sub>	2e	16	93	232–235 [232–234] [50]
20	3-NO <sub>2</sub>	2f	15	91	245–248 [247–250] [50]
21	4-Me	2g	23	89	240–243 [241–242] [50]
22	Thiophen- 2-yl	2h	20	90	274–276 [274–276] [52]
23	Н	3a	22	91	> 300 [300–302] [53]
24	4-Cl	3b	15	94	> 300 [330–332] [53]

 Table 2 (continued)

25	4-F	3с		20	84	269–272 [276–278] [53]
26	3-NO <sub>2</sub>	3d		15	93	> 300 [> 300] [54]
27	4-Me	3e	Me 0 0 0 0 0 0 0 0 0 0 0 0 0	21	86	> 300 [304–306] [53]
28	4-OMe	3f		14	91	> 300 [> 300] [54]
29	Thiophen- 2-yl	3g		22	88	> 300 [263–265] [55]

Table 2 (continued)

Reaction condition: arylaldehydes (1 mmol), 5-methyl-2-phenyl-2,4-dihydro-3*H*-pyrazol-3-one (1 mmol, 0.174 g) and 2-hydroxynaphthalene-1,4-dione (1 mmol, 0.174 g), Malononitrile (1 mmol, 0.066 g) <sup>a</sup>Isolate yields

minutes, and decanted. After evaporation of the solvent, the ImITCM catalyst was preserved for the next run. The resulted data presented in Fig. 1 illustrates that the ImITCM catalyst can be reused for three times without significantly diminishing the catalytic activity in comparison with its initial performance.

In a plausible mechanism for the synthesis of the target compound **1a** as established in Fig. 2, ImITCM as a nano-molten salt catalyst triggers the mechanistic pathway by the activation of 2-hydroxynaphthalene-1,4-dione as a nucleophilic and benzaldehyde as an electrophilic agent, respectively. The reaction proceeded by the reaction of these two activated species to generate the related intermediate **I**. On the other side, in the presence of the ImITCM catalyst, through a tautomerization process, 5-methyl-2-phenyl-2,4-dihydro-3H-pyrazol-3-one converted to its enol form **II**. In the next step of the plausible mechanism, reaction of intermediates **I** and **II** 



Fig. 1 The successful reusability test of ImITCM as a nano-catalyst

via a Michael addition reaction yields the corresponding intermediate IV. Finally, tautomerization of the intermediate IV in the presence of the catalyst generates the target molecule 1a.

### Conclusion

In this paper, a new application of ImITCM catalyst was explored. ImITCM presented a robust catalytic applicability for the preparation of naphthoquinone-based compounds in green and mild reaction conditions. A wide range of aromatic aldehydes were treated with 2-hydroxynaphthalene-1,4-dione and 3-methyl-1-phenyl-1H-pyrazol-5(4H)-one or malononitrile to afford the desired naphthoquinone-based molecules in short time with high to excellent yields. Some distinguishing features of the presented method include eco-friendly benign reaction conditions, a solventless process, easy isolation and purification profile, and short reaction times with high to excellent yields.

### General method for the synthesis of ImITCM as a molten salt catalyst

The construction method and structural confirmation of the ImITCM as molten salt catalyst were described in our previously reported study [47].



Fig. 2 Suggested plausible mechanism for the synthesis of target molecule 1a

# General procedures for the synthesis of naphthoquinone-based molecules in the presence of ImITCM catalyst

To a mixture of aromatic aldehydes (1 mmol), 2-hydroxynaphthalene-1,4-dione (1 mmol, 0.174 g) and one of the starting materials A, B, or C (1 mmol) in a roundbottom flask, ImITCM (0.038 mmol, 6 mg) was added as molten salt catalyst. The resulting mixture was exposed to the reaction in an oil bath at 90 °C for appropriate times, as illustrated in Table 2. After completion of the reactions as indicated by thin-layer chromatography TLC), in order to separate the catalyst, distillated water was added to the reaction mixture and stirred for a few minutes. Then, the watercontaining catalyst was decanted and the catalyst recovered for the next run. Finally, the desired products were recrystallized from EtOH to offer the pure products in high to excellent yields.

#### Selected spectral data

4-(2-Chlorophenyl)-3-methyl-1-phenyl-1,4-dihydrobenzo[6,7]chromeno[2,3-*c*] pyrazole-5,10-dione (1b)

Melting point = 250-252 °C.

FT-IR (KBr):  $\nu$  (cm<sup>-1</sup>) = 3165, 3063, 1655, 1611, 1574, 1401, 1370, 837, 750, 691.

<sup>1</sup>H NMR (400 MHz, DMSO)  $\delta$  (ppm) = 8.09–7.91 (m, 2H, aromatic), 7.87–7.76 (m, 2H, aromatic), 7.74–7.69 (m, 2H, aromatic), 7.52–7.42 (m, 3H, aromatic), 7.42–7.38 (m, 1H, aromatic), 7.34–7.20 (m, 3H, aromatic), 5.83 (s, 1H, CH), 2.08 (s, 3H, Me).

<sup>13</sup>C NMR (101 MHz, DMSO)  $\delta$  (ppm) = 184.0, 182.1, 158.1, 147.9, 138.3, 137.0, 134.9, 133.6, 133.1, 132.3, 130.9, 130.8, 130.7, 129.9, 129.5, 129.4, 128.5, 127.1, 126.5, 126.1, 126.0, 124.4, 121.0, 120.2, 104.1, 33.3, 11.8.

# 4-(4-Methoxyphenyl)-3-methyl-1-phenyl-1,4-dihydrobenzo[6,7]chromeno[2,3-c] pyrazole-5,10-dione (1e)

Melting point = 253-254 °C.

FT-IR (KBr):  $\nu$  (cm<sup>-1</sup>) = 3159, 3056, 2958, 1659, 1632, 1606, 1572, 1459, 1249, 1036, 757.

<sup>1</sup>H NMR (400 MHz, DMSO)  $\delta$  (ppm) = 8.03 (dd, J = 7.5, 1.0 Hz, 1H, aromatic), 7.96 (dd, J = 7.5, 1.0 Hz, 1H, aromatic), 7.85–7.75 (m, 2H, aromatic), 7.72–7.65 (m, 2H, aromatic), 7.48 (t, J = 8.0 Hz, 2H, aromatic), 7.29 (t, J = 7.5 Hz, 1H, aromatic), 7.12 (d, J = 8.5 Hz, 2H, aromatic), 6.81 (d, J = 8.5 Hz, 2H, aromatic), 5.77 (s, 1H, CH), 3.70 (s, 3H, OMe), 2.26 (s, 3H, Me).

<sup>13</sup>C NMR (101 MHz, DMSO)  $\delta$  (ppm) = 184.4, 182.5, 158.0, 147.8, 134.7, 133.6, 132.4, 131.6, 131.0, 129.6, 128.6, 126.6, 126.5, 126.0, 125.7, 120.7, 114.0, 105.4, 55.4, 32.1, 11.4.

# 4-(3-Hydroxyphenyl)-3-methyl-1-phenyl-1,4-dihydrobenzo[6,7]chromeno[2,3-*c*] pyrazole-5,10-dione (1k)

Melting point = 245-246 °C.

FT-IR (KBr):  $\nu$  (cm<sup>-1</sup>) = 3550, 3177, 3099, 1659, 1609, 1593, 1485, 1375, 974, 771, 729, 691.

<sup>1</sup>H NMR (400 MHz, DMSO)  $\delta$  (ppm) = 9.18 (br. s, 1H, OH), 8.04 (dd, J = 7.5, 1.2 Hz, 1H, aromatic), 7.98 (dd, J = 7.5, 1.4 Hz, 1H, aromatic), 7.88–7.76 (m, 2H, aromatic), 7.72–7.69 (m, 2H, aromatic), 7.50 (t, J = 8.0 Hz, 2H, aromatic), 7.31 (t, J = 7.5, 1H, aromatic), 7.05 (t, J = 8.0, 1H, aromatic), 6.69–6.60 (m, 2H, aromatic), 6.57 (d, J = 8.0 Hz, 1H, aromatic), 5.78 (s, 1H, CH), 2.28 (s, 3H, Me).

<sup>13</sup>C NMR (101 MHz, DMSO)  $\delta$  (ppm) = 184.5, 182.4, 157.7, 147.9, 141.4, 134.7, 133.6, 132.4, 131.0, 129.6, 129.5, 126.6, 126.5, 126.0, 125.5, 120.7, 118.3, 114.5, 113.4, 105.2, 32.7, 11.4.

# 4-(3-Bromophenyl)-3-methyl-1-phenyl-1,4-dihydrobenzo[6,7]chromeno[2,3-c] pyrazole-5,10-dione (1m)

Melting point = 214-215 °C.

FT-IR (KBr):  $\nu$  (cm<sup>-1</sup>) = 3156, 3074, 1660, 1634, 1595, 1501, 1416, 1286, 1054, 758, 690.

<sup>1</sup>H NMR (400 MHz, DMSO)  $\delta$  (ppm) = 8.05–7.94 (m, 1H, aromatic), 7.85–7.74 (m, 1H, aromatic), 7.69–7.67 (m, 3H, aromatic), 7.51–7.36 (m, 5H, aromatic), 7.31–7.22 (m, 3H, aromatic), 4.98 (s, 1H, CH), 2.31 (s, 3H, Me).

<sup>13</sup>C NMR (101 MHz, DMSO)  $\delta$  (ppm) = 184.1, 182.8, 160.5, 147.8, 146.8, 145.4, 143.4, 137.4, 134.7, 130.9, 130.2, 129.6, 129.5, 126.7, 126.4, 122.0, 121.2, 120.8, 104.6, 104.1, 33.2, 11.9.

# 4-(3-Methyl-5, 10-dioxo-1-phenyl-1,4,5,10-tetrahydrobenzo[6,7] chromeno[2,3-*c*]pyrazol-4-yl)benzonitrile (1n)

Melting point = 226-227 °C

FT-IR (KBr):  $\nu$  (cm<sup>-1</sup>) = 3165, 3062, 2228, 1658, 1640, 1606, 1572, 1501, 1374, 1281, 954, 727.

<sup>1</sup>H NMR (400 MHz, DMSO)  $\delta$  (ppm) = 8.03 (dd, J = 7.5 Hz, 1H, aromatic), 7.99–7.93 (m, 2H, aromatic), 7.86–7.64 (m, 6H, aromatic), 7.51–7.43 (m, 3H, aromatic), 7.32–7.27 (t, J = 7.5 Hz 1H, aromatic), 5.88 (s, 1H, CH), 2.27 (s, 3H, Me).

<sup>13</sup>C NMR (101 MHz, DMSO) δ (ppm) = 184.0, 182.7, 147.8, 146.8, 136.4, 134.7, 133.5, 132.5, 131.2, 129.6, 128.8, 126.6, 126.4, 126.0, 124.5, 120.9, 119.5, 109.1, 103.8, 33.3, 11.6.

Acknowledgements We thank Bu-Ali Sina University and the Iran National Science Foundation (INSF) for financial support (grant no. 940124), and Iran's National Elites Foundation for recognizing our research group.

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